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# THE JOURNAL
OF THE
# Society of Chemical Industry.

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## A MONTHLY RECORD
FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

No. 1.—Vol. XVII.] (1898) JANUARY 31, 1898. [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

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Mr. J. W. Lovibond. "The Curing of Malt in relation to Colour and Value."

Messrs. A. R. Ling and J. Baker. "Clerget's Method of Estimating Sugar, and a New Modification of it, specially applicable to After-Products and Molasses."

Dr. L. T. Thorne and Mr. E. H. Jeffers. "Note on the Estimation of Water in Invert Sugars."

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Friday, Feb. 4th, 1898.—Dr. J. Grossmann. "The Treatment and Disposal of Town Sewage."

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Feb. 22nd, 1898 (Edinburgh).—Dr. Wm. Frew. "The Education of the Brewer."

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In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

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The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

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At a meeting of the Committee of University College, Nottingham, held on Tuesday, January 18th, it was resolved:—"That in recognition of the valuable services rendered to the College by Prof. Clowes, first in organising the Chemical Department, and afterwards for 16 years discharging the duties of Chemical Professor, and for three years the duties of Principal, an honorary position of Emeritus Professor of this College be conferred upon him."

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*Meeting held on Monday, January 3rd, 1898.*

DR. R. MESSEL IN THE CHAIR.

### NEATSFOOT OIL.

BY J. H. COSTE, F.I.C., AND E. J. PARRY, B.SC., F.I.C.

Very little information of a reliable character has been published concerning this oil; one reason of this, probably, is the difficulty of obtaining satisfactory samples, as most of those met with in commerce are, even when supposed to be genuine, mixed with other animal oils; grosser adulteration is not at all uncommon. Neatsfoot oil is obtained by boiling the feet of oxen in water until all the oil has risen to the surface; and very often, doubtless, the feet of sheep pigs, and other animals are boiled together with those of the ox, and so a mixed animal oil is obtained, sold as neatsfoot oil, but of somewhat variable composition. Bone oil, lard oil, and fish oils are also said to be added to this oil, which, commanding as it does a fairly high price, offers incentives to adulteration.

As we are from time to time called upon to examine samples of this oil, we have obtained specimens of known purity and examined them. One of these samples (I.) came from one of the most reliable sources in the trade; the other two (II. and III.) were obtained by boiling, in each case, two neatsfeet in a large saucepan for some hours with water, after they had been scalded to remove the hair and chopped at the joints. The clear oil which separated was skimmed off the top of the liquor when cool. In each case about half a pint of oil was obtained; this was filtered through a dry filter paper whilst warm to remove any traces of connective tissue, &c., and then kept in a water oven for an hour to expel traces of water. In each of these cases the oil was of a pale golden-yellow colour, with a not unpleasant odour of beef fat, and slowly deposited solid stearin.

The following are the results of the chemical and physical examination of these samples :—

| —— | I. | II. | III. |
|---|---|---|---|
| Specific gravity $D\frac{15 \cdot 5^\circ}{15 \cdot 5^\circ}$ C. ........ | 0·9163 | 0·9169 | 0·9174 |
| Temperature rise with sulphuric acid (equal vols). | 51° C. | 58° C. | 56° C. |
| Viscosity at 140° F. (Redwood's viscosimeter) | 74 sec. | 70 sec. | .. |
| " " 200° F. | .. | 43 sec. | .. |
| Iodine absorption by Hübl's method | 65·2 % | 71·1 % | 72·4 % |
| Percentage of potash (KOH) for saponification. .................. | 18·9 | 19·55<br>19·66 | 19·74 |
| *Free acid (calculated as oleic acid) | .. | Trace | 0·37 |
| " (saturates KOH) ...... | .. | " | 0·07 |
| Insoluble fatty acids (by Hehner's method). | .. | 95·3 | 95·5 |

* Redetermined after keeping ten months: II., 0·14; III., 0·38.

On looking at the above figures it will be seen that the two samples obtained from known neatsfeet agreed much more closely in properties with one another than with that obtained through the trade source.

The fatty acids of these three samples were prepared by saponifying large quantities of oil, and, after washing with hot water and drying at 100°, gave the following results:—

| —— | I. | II. | III. |
|---|---|---|---|
| Specific gravity $D\frac{100^\circ}{100^\circ}$ C. ......... | .. | 0·8742 | 0·8800 |
| Iodine absorption................... | 68·4 | 74·5 | 75·8 |
| Percentage of potash (KOH) for neutralisation. | 19·17 | 20·12 | 20·06 |
| Mean combining weight........... | 292 | 279 | 280 |
| Solidifying point.................. | .. | .. | 26·1 |
| Melting point...................... | 30° C. | 29·2° C. | 28·5° C. |

In each case the fatty acids were nearly white, crystalline, almost odourless.

Other samples of neatsfoot oil of more or less—apparently in most cases less—genuine character have been examined by us. We give some of the analytical figures of a few of these :—

| — | Specific Gravity. | Temperature Rise with $H_2SO_4$. | Percentage of KOH to Saponification. | Iodine Absorption. | Free Oleic Acid. |
|---|---|---|---|---|---|
| | | ° C. | | | |
| 1 | 0·9197 | 54·0 | .. | .. | .. |
| 2 | 0·9050 | 27·0 | 16·82 | .. | .. |
| 3 | 0·9220 | 68·5 | .. | 93·5 | .. |
| 4 | 0·9220 | 65·0 | 17·58 | 130·4 | .. |
| 5 | 0·9179 | 55·0 | 17·58 | 84·5 | .. |
| 6 | 0·9174 | 56·0 | .. | 69·0 | .. |
| 7 | 0·9224 | 54·0 | 20·40 | 87·6 | 5·0 |
| 8 | 0·9201 | 37·0 | 18·45 | 82·9 | 1·7 |
| 9 | 0·9186 | .. | 19·64 | 67·6 | .. |
| 10 | 0·9197 | 54·0 | 20·00 | .. | .. |

Of these samples, No. 2 contained a considerable proportion of paraffin, as the small amount of potash required for its saponification shows. The only two of these samples which agree reasonably well with the pure samples obtained by us are Nos. 6 and 9. The rest appear to contain other animal oils with cotton-seed and perhaps linseed oils.

The fatty acids of Nos. 8 and 9 gave the following figures :—

| —— | $D\frac{100^\circ}{100^\circ}$ C. | Percentage of KOH to neutralise. | Iodine Absorption. |
|---|---|---|---|
| 8 | 0·8752 | 20·60 | 89·2 |
| 9 | 0·8717 | .. | 70·0 |

Whereas the fatty acids from the pure samples were almost white, both these preparations were decidedly yellow.

## DISCUSSION.

Mr. E. **GRANT HOOPER** agreed with the author of the paper that neatsfoot oil was very largely adulterated. No doubt as a fairly high-priced oil there was considerable temptation in this direction, and his experience had been that sophistication was not merely very common, but was also in many cases of a very gross character. Thus, in a batch of samples that recently came before him, numbering eight, tendered in connection with a single contract, the following variations were observed, *viz.* :—Densities from 0·9048 to 0·9196; saponification values (per cent. of potash), 5·64—19·9; iodine absorbed, 46·5—86·7; acid (as oleic), 1·20—6·20; refractions, −3·6 to +8·2, and one exceptional sample +43·7. Glancing over the results of about 80 samples examined during the past year and a half, a large number of similar cases were found, and to the figures he had already quoted he need only add that, as regards acidity, whilst a percentage as low as 0·39 had been observed, the range had extended to as high as 12, 15, 19, and 34 per cent. as oleic acid; and with reference to the refractions, readings of −11, −13, −14, and +7 and +57 had been obtained. Thus there could be no doubt, not merely that neatsfoot oil was frequently adulterated with animal and vegetable oils, but that mineral oil was also not infrequently employed. In view of these circumstances, and of the few results of the examination of undoubtedly genuine neatsfoot oil that were available, he thought the figures obtained by Mr. Coste were very valuable, and especially those yielded by the sample which the author of the paper had himself prepared. It was interesting to note that these results closely agreed with those given by Allen, Lewkowitsch, and others. He noticed that Mr. Coste had given no refractions. He would like to inquire if these had been determined, for there could be no doubt that the refractometer was a most valuable instrument in connection with the examination of oils.

Mr. **OTTO HEHNER** said that Mr. Hooper's figures were interesting, but did not furnish any contribution to the question of the composition of genuine neatsfoot oil, as many of his samples were obviously grossly adulterated. He would like to warn chemists against pinning their faith upon any particular number of so-called constants worked out from a small number of samples. Experience invariably showed that the composition of all oils and fats varied considerably. Lard, for instance, showed wide differences, depending upon the feeding of the animals, the position from which the fat was taken, &c.; and no doubt such differences would also be found in neatsfoot oils. He would have been interested if the authors had somewhat more closely examined the unsaturated portion of the oil, with a view to ascertain whether less saturated glycerides were contained in it than oleine.

Dr. S. **RIDEAL** pointed out that the progressive nature of the figures in the three samples made it seem that the ratios between the different determinations were fairly constant. Mr. Hooper's figures would have been more interesting if he had given the iodine value of those samples which required so little potash for saponification.

Dr. **PARKER** said that the figures were important as far as he was concerned, as no oil was more used in the leather trade than neatsfoot oil, and none more generally adulterated. The exact action of oils upon leather was as yet but little understood, and there was room for much work thereon.

Mr. **WM. CROWDER** remarked that probably the oil from the feet of the bull, the cow, and the calf would vary in composition, so that any great regularity would not be expected in the figures obtained from various samples.

Mr. **J. H. COSTE**, in reply, said that, of course, in such substances as natural oils and fats, consisting of mixtures of the glycerides of several allied acids, considerable variation in composition, and, as a result, in the various empirical figures obtained in their examination, must be expected, as Mr. Hehner had pointed out, and any attempt to define hard-and-fast limits would be injudicious. It would be a matter of considerable scientific interest to examine the oil from the feet of not only the bull, the ox, the cow, and calf, but of cattle of various breeds; but at the same time the oils found in commerce, where genuine, would probably be mixtures of those obtained from animals of different sexes, ages, and breeds. The relations pointed out by Dr. Rideal were interesting, and showed how desirable it was, in pronouncing judgment on samples, to consider the general aspect of the case rather than particular "values."

Mr. Grant Hooper's figures, though interesting as evidence of adulteration, could hardly be quoted as having much bearing on the nature of pure neatsfoot oil, as oils requiring 6 per cent., or thereabouts, of potash, must be very grossly adulterated with unsaponifiable oils. It was their intention to examine more fully the nature of the fatty acids of this oil.

## STANDARD METHODS FOR THE SAMPLING AND ANALYSIS OF TANNING MATERIALS.

WITH REMARKS BY PROF. HENRY R. PROCTER AND DR. J. GORDON PARKER.

The introduction of the Löwenthal volumetric method for the estimation of tannin, marked a certain advancement on the processes formerly known for the estimation of tannin in tanning materials. This method was, however, found to be unsuitable for some materials, and latterly has been almost completely dropped in favour of a gravimetric hide-powder method, suggested some years ago by Simond, which consisted in digesting a tanning solution with successive quantities of hide powder. Since that date, however, many improvements in procedure have been suggested, notably those of Prof. Procter, of the Yorkshire College, Leeds, who first invented the hide-powder filter process, the detail of which was further improved by Director Eitner, of the Vienna Leather Industries Research Station. In this method a solution of tanning matter was passed through a siphon fitted with a tube containing purified hide powder, and the amount of tannin contained in the solution was calculated by weighing the dried residue from a portion of the liquor before passing through the hide powder, and from a second portion detannised by passing through. The results from this method were found to be nearer to those obtained by the practical manufacturer in his tannery than those of the volumetric process. Those engaged in the chemistry of the leather trade have now used this method for several years; but it has been found that even by it concordant results are not always obtainable, the process, like many other analytical methods, being more or less empirical, and only giving comparable results under identical conditions. Each individual chemist appears to have introduced his own modification or improvement of the process, the outcome being that when buyers sent a sample of a delivery to one chemist, and sellers a part of the same sample to another, the results of analysis were seldom in exact agreement, the difference being sometimes as high as 5 per cent. This fact, which was acknowledged on all sides, did much harm in the eyes of the practical tanner, many tanners losing all faith in the use or application of science to their trade.

A commission on tannin analysis was held in Berlin in 1885, which dealt with the older volumetric process. They endeavoured to fix some standard methods by which all should work, and altered the Löwenthal process, laying down fixed rules as to how it should be worked. The introduction of the hide-powder filter process practically superseded the Löwenthal method; but no subsequent meeting of the commission has been held.

In the early part of the present year, Prof. H. R. Procter, Dr. J. Gordon Parker, Mr. A. Seymour Jones, and Mr. C. E. Parker took the initiative in calling a conference of chemists engaged in the leather trade. Their efforts in this direction were first of all confined to Great Britain, and considerable interest was manifested by the trade chemists in the movement. It was thought, however, that the resolutions of a conference would be of much greater value if the conference were international; and in this idea Prof. Procter and Dr. Parker opened up correspondence with all the best-known leather-trades chemists in the world, inviting them to a conference which should be held in London; and it was laid down, as a condition of having voice and vote in the proceedings of the conference, that each chemist, before it was held, should send in to Prof. Procter a full and detailed statement of the methods adopted by him in—

I. Drawing samples: (*a*) liquid extracts; (*b*) solid extracts, including gambier; (*c*) valonia; (*d*) algarobilla and divi-divi; (*e*) general tanning materials.
II. Preparation of sample for analysis.
III. Preparation of infusion.
IV. Determination of tanning matter.
V. Colour measurement.

A number of such statements having been received in reply to the invitations sent out, these papers were summarised and tabulated by Prof. Procter in the form of a Report, which was printed and circulated. The Conference was then called to meet at the Leather Industries' Laboratory, Herold's Institute, Bermondsey, London, on September 28th, 29th, and 30th. The number of chemists attending was 24; the countries represented being Great Britain, Austria, Belgium, Denmark, France, Germany, Hungary, and the United States of America; the Directors from all the leather industries research stations being either present or represented. The first day's proceedings were presided over by Dr. W. H. Perkin, who summarised the difficulties which were met with in the analysis of tanning materials, and emphasised the necessity of finding out which were the best and most reliable methods of analysis at present known, and the importance of each chemist abiding by the methods which should be decided on by the Conference. As it was found to be the general wish that the Conference should be an annual one, it was resolved to then and there form an Association, to be called the International Association of Leather-Trades Chemists, which should have for its objects the fixation of standard methods of analysis and the inter-communication of research work.

It was further decided that any qualified leather-trades chemist could become a member of the Association if proposed by one member and supported by two others, those members recommending a candidate to be assured of his eligibility.

The newly-formed Association then went on to the consideration of Prof. Procter's report; their resolutions, with some words of comment now follow:—

In respect of—

### I.—*Drawing Samples.*

(*a.*) *Liquid Extracts.*—"In drawing samples, at least 5 per cent. of the casks must be taken; the numbers being selected as far apart as possible. The heads must be removed and the contents mixed thoroughly by means of a suitable plunger, care being taken that any sediment adhering to sides or bottom should be thoroughly stirred. All samples must be drawn in the presence of a responsible person."

This first resolution was of paramount importance to the object in view, as it had frequently been proved that the differences obtained by chemists were due to unmethodical drawing of samples. Tanning extracts are usually thick, viscous liquids, frequently having a density of 1·3 or upwards. When casks containing such liquids are allowed to stand for some time, they are very liable to deposit the less easily soluble tanning matters in the form of sediment, and unless this be thoroughly stirred up, it is impossible to get a representative sample. This resolution was carried *nem. con.*

The second resolution refers to the sampling from bulk of solid or pasty extracts, and is as follows:—

(*b.*) "Gambier and pasty extracts should be sampled from not less than 5 per cent. of the blocks, by a tubular sampling tool, which shall be passed completely through the block in *seven* places. Solid extracts shall be broken, and a sufficient number of portions drawn both from the 'inner' and 'outer' parts of the blocks to fairly represent the bulk. In both cases, samples shall be rapidly mixed and enclosed at once in an air-tight bottle or box, sealed and labelled."

We might point out, in reference to the above resolution, that the discussion on it showed that gambier was one of the materials which caused the greatest trouble to the chemist. Gambier is an extract of a pasty or solid nature, made by boiling the leaves and twigs of the gambier shrub, which grows in the neighbourhood of Singapore and the islands of the Malay Archipelago. It is mostly prepared for the market by natives, who, instead of paying great attention to the improvement of its manufacture, have rather concentrated their attention on the best and most suitable methods of adulteration. The product is put on the market either in the forms of blocks weighing from 1½ cwt. upwards, or in the form of small cubes of about 1½ ins. Analyses have shown that the blocks frequently contain as much as 40 per cent. of adulterants. In the large blocks, therefore, as the adulterating material was sometimes put in after the liquid had been poured out to set, and therefore imperfectly mixed, it was necessary that tubular samples should be drawn, and from at least seven different portions of the block or bale.

Solid extracts, including quebracho and canaigre, are generally made up into small bales, weighing, perhaps, 14 lb. The outer or drier surface, containing less moisture, would naturally show a higher percentage of tannin than the interior.

The above resolution, which meets the difficulties, was passed unanimously.

(*c*, *d*, *e*.) "Valonia, algarobilla, and all other tanning materials containing dust or fibre shall be sampled, if possible, by spreading at least 5 per cent. of the bags in layers, one upon another, on a smooth floor, and taking several samples vertically to the floor. Where this cannot be done, the sample must be drawn from the centres of a sufficient number of bags. While valonia and most tanning materials may be sent to the chemist ground, it is preferable that divi-divi and algarobilla shall be unground. Bark in long rind, and other materials in bundles, shall be sampled by cutting a short section from the middle of 3 per cent. of the bundles with a saw or other sharp tool."

The reason for the decision that algarobilla and divi-divi should be sent to the chemist whole was that, in the grinding, the fibre, which contains comparatively little tannin, separates completely from the dust or fine material, which contains over 90 per cent. of the tannin; and it was thought to be better that the chemist should receive the material whole, as he could then thoroughly gauge the correct proportions of fibre and dust.

"Samples to be submitted to more than one chemist must be drawn as one sample, and well mixed, and then divided into the requisite number of portions, not less than *three*, and at once enclosed in suitable packages, sealed and labelled."

This resolution is of great importance, as frequently a sample was drawn and sent off for analysis. A report having been obtained, it was, perhaps, not up to the guarantee of the seller. Litigation ensued, and the seller probably came down and drew a fresh sample. The material having, however, stood for some time, had possibly undergone change. The second sample might not have been drawn from the same casks or bags, and it naturally could not represent the same material as was drawn in the first instance.

The resolutions above have particular reference to the tanner, or tanning material merchant, but were passed as rules by the Association for the guidance of tanners and dealers, as the members of the Association would be prepared to give evidence in any law case only when a guarantee was given that the samples were drawn according to the methods prescribed by them.

We now pass on to the more particularly chemical portion of the proceedings. The first was the preparation of sample of analysis.

"Liquid extracts shall be thoroughly stirred and mixed immediately before weighing, which shall be rapidly done to avoid loss of moisture. Thick extracts, which cannot be otherwise mixed, may be heated to 50° C., then stirred and rapidly cooled before weighing; but the fact that this has been done must then be noted in the report."

Some extracts, especially quebracho, or samples which may have been kept for a considerable period, sometimes deposit a very difficultly soluble sediment in the bottom of the bottle in which they are kept. It was found in practice very difficult to get this thicker portion, which was of a sticky nature, to mix properly with the more liquid portion; and in some cases this was found absolutely impossible. In order to meet this difficulty, permission is given to heat the extract to a temperature at which it was found that most of these extracts would mix.

"Solid extracts shall be coarsely powdered and well mixed. Pasty extracts shall be rapidly mixed in a mortar, the requisite quantity weighed out with as little exposure as possible, to avoid loss of moisture. Where extracts are partly dry and partly pasty, so that neither of these methods is applicable, the entire sample shall be weighed and allowed to dry at the ordinary temperature sufficiently to be pulverised, and shall then be re-weighed, and the loss of weight taken into calculation as moisture.

"In cases such as gambier, in which it is not possible to grind or by other mechanical means to thoroughly mix the constituents of the sample, it is permissible to dissolve the whole or a large portion of the sample in a small quantity of hot water, and immediately after thorough mixing to weigh out a portion of the strong solution for analysis."

This resolution gave considerable difficulty, as it was found that many different methods were in use by the various Continental and British chemists bearing on this subject. After considerable discussion, the above resolution was carried as meeting best the various opinions held. One point worthy of note is the stress laid upon the necessity of weighing as rapidly as possible, in order to avoid loss of moisture.

(*c*.) Barks and other solid tanning materials. "The whole sample, or not less than 250 gr., shall be ground in a mill until it will pass through a sieve of 5 wires per centimetre. When materials, such as barks and divi-divi contain fibrous material which cannot be ground to powder, the ground sample shall be sieved and the respective parts which do and do not pass through the sieve shall be weighed separately, and the sample for analysis shall be weighed so as to contain like proportions."

### III.—*Preparation of Infusion.*

"The strength of tanning solution employed shall be such that from evaporation of 100 c.c., the residue shall amount to between 0·6 and 0·8 gr."

On the Continent, the favoured strength for solutions varied very much, and the strength adopted, *viz.*, 0·6—0·8, is a fair mean of those favoured by different Continental and English chemists. As tanning infusions are liable to oxidation from the evaporation on the water-bath, and the subsequent drying in the air oven, it was deemed advisable that the smallest quantity should be taken, with which definite and accurate results could be obtained, without allowing the chance of large manipulative errors, and research showed that the above strength appeared to combine speed and accuracy.

*Solution of Liquid Extracts.*—"A sufficient quantity shall be weighed into a covered basin or beaker, from which it shall be washed into a litre flask with circa 500 c.c. of boiling water, well shaken and the flask filled nearly to the mark with cold water. The neck being covered with a small beaker, it shall be placed under a cold-water tap, or otherwise rapidly cooled to a temperature between 15° and 20° C., and made up to the mark, after which it shall be thoroughly mixed, and the filtration at once proceeded with."

"The filtration shall take place through Schleicher and Schuell's filter paper, No. 602. The first 150 or 200 c.c. of the filtrate shall be rejected, or employed for the determination of the non-tannins. Where a clear filtrate cannot be obtained, the use of a little kaolin, previously washed with a portion of the tanning solution, is permissible."

Some solutions of tanning material, especially canaigre, gambier, and quebracho, are not obtainable perfectly clear by ordinary filtration. A very obstinate solution was sent to the above well-known filter paper manufacturers, with the result that they made this special paper to meet the case in question. It is found most satisfactory, but still cases are met with where even this special paper does not filter perfectly clear. Kaolin, having no action upon tannin, may therefore be used in small quantities, but should be avoided as far as possible, as it appears to fix some colouring matters which would otherwise be estimated as tannins.

"Solid extracts shall be dissolved by stirring in a beaker with boiling water, the undissolved portions being allowed to settle, and treated with further quantities of boiling water, and the solutions poured into a litre flask. After the whole of the soluble matter is dissolved the solution is treated similarly to that of a liquid extract."

*Extraction of Solid Materials.*—"Such quantities shall be weighed as will give an infusion of the strength already prescribed (Resolution III.*a*). Not less than 500 c.c. of the infusion shall be extracted at a temperature not exceeding 50° C., after which the temperature shall be raised to 100 C., and the extraction continued till the percolate is free from tannin and the whole made to one litre, the weaker portions of the solution being concentrated if necessary by evaporation in a flask, in the neck of which a funnel is placed."

Although no specified extraction apparatus was decided upon, it was recommended that the form prescribed by Procter, and published in this Journal, should be used, because of its simplicity and the ease with which the temperature of extraction could be regulated.

### IV.—*Total Soluble Matter.*

(*a.*) "100 c.c. of the clear filtered tanning solution, or a smaller quantity if the balance employed is of sufficient delicacy, shall be evaporated in an open weighed basin of platinum, hard glass, porcelain, or nickel on the water-bath, and the basin shall afterwards be dried in an air oven, at a temperature of 100° to 105° C., or *in vacuó* at a temperature not exceeding 100° C. till constant, care being taken that no loss occurs by splintering of the residue."

The above temperatures were decided upon as the highest at which tannins may be dried without danger of driving off any material other than water. Some chemists formerly used a higher temperature, but doubts were expressed whether some of the tanning materials were not partially decomposed above 105°. The necessity of going above 100 is accounted for by some of the tannins forming compounds with water which were only decomposed at a temperature exceeding 100°.

(*b.*) Determination of non-tannins. "That the filter method be adopted until next Conference."

It was found that a considerable number of the members of the Association were strongly in favour of the American shake method as proposed by Fiebing and Yocum, and almost exclusively used by the American leather-trades' chemists. Many of those on this side of the Atlantic were in favour of retaining the older "bell form" of filter. After considerable discussion upon the merits of the one process over the other, a vote was taken, and it was decided that for the present, until more experience had been obtained of the shake method, the filter method should be retained.

"That the 'bell form' of filter, as described by Prof. Procter, be adopted, and that not less than 5 grms. of hide powder be used, and that the filtrate be rejected so long as it gives a turbidity with a clear tanning solution. The filtrate may be used for the determination of non-tannins so long as it gives no reaction with hide-powder water. That 50 c.c. or less quantity of detannised solution be evaporated in a weighed basin on a water-bath, and then dried till constant in an air oven, at a temperature of 100 to 105 C., or *in vacuó* not exceeding 100 C."

It should be noted that though it is a matter of indifference whether 25, 50, or 100 c.c. are evaporated, it is a point of considerable importance, which has not yet been decided, whether 50 (or 100) c.c. should be filtered and the whole or a portion of this taken, or whether the *first* 25 c.c. after the filtrate is free from soluble hide substance should be evaporated. The filter absorbs acids and certain other substances not strictly tannins, and this absorption diminishes rapidly with the volume passed through the filter. Until this point is authoritatively decided, it seems advisable that the portion evaporated should at least represent an average of the first 50 c.c.

The tanning solution mentioned above may consist of any clear solution containing tannin. The hide-powder water may be made by shaking up a few grms. of hide powder in distilled water, and filtering. This has been found to give a more definite and accurate result than any of the gelatinous or iron solutions which are used for the detection of tannin by some chemists.

(*c.*) "That the hide powder must be sufficiently absorbent for use in the filter, and that in a blank experiment conducted with distilled water in the same way as an analysis, the residue from the evaporation of 50 c.c. should not exceed 5 mgrms."

Since the Conference, the London Leather Industries' Laboratory has undertaken to supply a first-class hide powder guaranteed up to the standard approved of in the above resolution. The hide powder is made in Vienna under the careful supervision of the Director of the Leather Industries' Laboratory in that city, and is sent off in large deliveries to London. By using the same hide powder, all chemists in England should be able to get accordant results.

The remaining agenda were decided as follows:—

"(*d.*) That a determination of moisture in the sample be determined by drying a small portion at the temperature adopted in the determination of the 'total soluble.'

"(*e.*) *Statement of Results.*—That the statement of results, when a full analysis is given, should be made in the following manner:—

"(1.) *Tanning Matters Absorbed by Hide.*—Obtained by deducting the 'soluble non-tanning matters' found by evaporating the hide-powder filtrate, from the 'total soluble.'

"(2.) *Soluble Non-tanning Matters.*—Found by evaporation of filtrate from hide-powder filter.

"(3.) *Insoluble.*—By deducting 'total soluble matter' from 'total dry matter.'

"(4.) *Moisture.*—Determined by drying a portion at the temperature adopted in the determination of 'total soluble.'

"If other determinations are given, they shall form a separate additional statement."

The above is of more moment to the tanner than to the chemist. Frequently chemists estimated mineral ash, and sometimes added it in the analysis, deducting its percentage from the non-tannins, and it was not explained that this was done, thus leading to misunderstandings.

### *Measurement and Valuation of Colour.*

"The fixing of a standard method of measuring and valuing colour should be left for consideration at next year's meeting in Vienna. It was recommended that the method used by English chemists, *viz.*, measuring with Lovibond's tintometer (as described by Dr. Parker and Prof. Procter), should be used, and the results stated as degrees of red, yellow, and black."

We might point out here that one of the reasons why this method was not actually adopted by the Conference was because Lovibond's tintometer was not known to the Continental chemists. A large amount of work has been done in this country on the subject, and three papers bearing on the same have been read before this Association. Still further work has more recently been done with this instrument, and it has been found to be a most valuable and accurate instrument for the measurement of the colour contained in various tanning materials. It has also been found to be perfectly accurate in the comparison of the degrees of colour contained in different samples of the same material, and as a result of this many extract manufacturers are now selling their extracts with a guarantee of the percentage of tannin, and that the extract shall not contain above a certain number of colour units, as measured by the Lovibond tintometer. We are of the opinion that at the next year's Conference the Lovibond tintometer will be adopted as the standard appliance for measuring the colour contained in tanning materials for all countries.

The above resolutions are to come into force on January 1st, 1898, and all leather trades' chemists who are members of the Association have agreed to work by the Conference methods, until they are altered or supplanted at a future conference of the Association.

The next meeting of the Association will be held in Vienna, during the month of July, when it is believed that a still larger number of leather trades' chemists will attend.

In reporting to this Society the foregoing resolutions of the International Association of Leather Trades' Chemists, we trust that we may have done something to assist those members of the Society who have to deal at times with materials containing tanning matters, and to enable them to arrive at satisfactory results. The resolutions remain nominally in force until the Vienna Conference this year, but there is very little likelihood of their being materially altered until more definite knowledge has been arrived at of the constitution of the various tannins. A fertile field for research is here presented; but little work has so far been done in it, and there is, perhaps, no other branch of applied chemistry so promising for investigation, or in which there are ready at hand so many subjects that demand experiment and consideration. Had there existed an absolute gravimetric method of estimating or precipitating tannins from solution there would have been no need of a conference. Until such method is found there is no lack whatever of work for the new association.

### Discussion.

Mr. Otto Hehner asked for a definition of the term "Leather-Trades Chemist."

Dr. F. Clowes considered it a matter for congratulation that the application of scientific chemistry to the tanning industry had been taken up by responsible scientific men. He thought it would not be too much to expect that, before many years had passed, the industry would become as dependent on the application of chemistry as the brewing trade was at present. The Pasteur of tanning had not yet arisen, but a great many competent gentlemen were combining their forces to produce the same result that Pasteur had produced in the brewing industry. He had long had to do with the methods explained in the paper before them, and had also had the advantage of frequent information from Mr. Procter as to the method of applying his process; and he considered that even if the hide-powder process could not be accepted as scientifically accurate, it was, nevertheless, a great advance on the older method of Löwenthal. The processes of tanning involved the use of such complicated mixtures as to make it difficult to fix on any method of analysis which would yield uniform and trustworthy results. It might have appeared to many that the authors had gone unnecessarily into analytical details in their paper. If so, he begged to assure members that this was not the case, and that the successful estimation of tanning materials did depend to a great extent on the most careful attention to these minute details of working. It was therefore with the greatest satisfaction that he had heard of this promising attempt to place the methods of sampling and determination on a satisfactory basis, and he cordially wished the new Association success.

Mr. Seymour Jones, in answer to Mr. Hehner's question, defined a Leather-Trades Chemist as one who devoted himself exclusively to, and had practical experience, in the leather trade; and he would instance as examples Dr. Parker, Prof. Procter, Dr. Eitner, of Vienna, and Franz Kathreiner, of Worms. The Association was in no sense a trades union, for anyone might become a member by following the procedure which had been laid down. The conference had done much practical work in the three days of its meeting last year; this year they would probably meet in Vienna. He was glad to be able to say that the Association had its origin in England, which was the birthplace of most of the kindred associations; but what was more gratifying still, was that they had the unanimous support of all their brethren in Europe and other parts of the world in working out definite and concordant methods. Their great object was to place the leather-trades industry in a higher and better position scientifically than it had hitherto occupied.

Mr. J. T. Wood had taken part in the Leather-Trades Conference, and was in thorough agreement with the views of the authors. He would like to know whether hide powder was to be made in London. He hoped that was to be the case, for the Vienna powder was not always satisfactory. Indeed, since the Conference met, he had received hide powder mixed with paper—purposely mixed, in order to assist the analyst. Such a mixture might work well with the Vienna filter, but it did not suit the filter used by English analysts, and the results got were not always concordant.

Mr. J. Stewart wished to be informed as to the composition of the precipitate resulting from the tanning extract.

Mr. Otto Hehner wished to know whether any experiments had been made with a view to determine whether hide powder took up tannin and nothing else. It seemed to him that it must necessarily absorb a portion of whatever salts might be in the solution, and therefore a portion of the loss estimated in the latter as tannin would represent salts. As regards the action of the new Association, he could not help thinking that the preferable course would have been for them to have come to this Society and invoke a convention on this subject, instead of first arranging the methods, and then asking the Society to endorse the same. He was much impressed by Dr. Parker's statements as to the necessity for observing the very minute conditions laid down by the Association for obtaining correct analyses; but the word scientific could hardly be applied to a method which was only accurate for very narrow conditions of working.

Dr. H. E. Armstrong agreed with Mr. Hehner's views, and thought it a matter for congratulation that the paper had been brought before the Society, as it would enable them to correct what he regarded as a mistake. It was pleasant to find that gentlemen engaged in an industry like tanning—in which empirical methods admittedly obtained at present—should have agreed to work together in the remarkably unanimous manner set forth by Dr. Parker. It was unfortunate, however, that these gentlemen had, as it were, gone off in a swarm by themselves. If other industries followed their example the result would be disastrous, because every industry would be working in its own narrow sphere instead of collaborating for the common good and advancement. It would be far better that an association should be formed of scientific men in general, and that such an association should include sections for those doing special work. Within recent times several international meetings of analytical chemists had been held with a fair amount of success, and in the United States they were very hopeful that these meetings would become more and more important. He thought it very undesirable, therefore, that those engaged in the leather trade should withdraw themselves from the general body, and that they could do more good to themselves and to others by uniting to form a section of a general international conference. He had himself felt somewhat disappointed that the Society of Chemical Industry had not succeeded in uniting the various smaller societies dealing with special subjects in which chemistry played a part. His view was that all such bodies ought to work together, and as far as possible publish together, so that all the members might have information from a variety of sources put under their eyes and be forced to acquire it. One remark made by Dr. Parker had greatly interested him—that Messrs. Schleicher and Schüll had actually undertaken the manufacture of a special filter paper for the use of one trade. It was hardly conceivable that any British manufacturer would have done such a thing.

Dr. S. Rideal regarded the proceedings of the Conference which had been laid before them as specially interesting, and of much greater range and importance than appeared to have been recognised by the 24 gentlemen whose resolutions had been put before them. Some four years ago, he had brought before the Society a paper dealing with tannin determinations, and had suggested some modifications in the hide-powder method, which remove or overcome many of the difficulties which had been referred to. He agreed with Mr. Hehner that the filtrate from hide powder must necessarily contain some of the salts present in the hide powder, and so vitiate the result. They had heard from Mr. Wood about the Vienna standard hide powder, and the views of the Conference as to the great proneness of solutions to oxidation and consequent increase of weight. Those two facts made it extremely difficult to form any valuation upon the Procter method. He felt certain that if the matter were taken up by analytical chemists of general knowledge and experience, they would be able to arrive at some method which would have a greater chance of being universally accepted than had the processes laid before them by the authors.

Mr. Forbes Carpenter thought the paper divided itself into two parts: the question of sampling, and the question of submitting to analysis the samples taken. Gentlemen engaged in the particular industry under consideration were, after all, the best judges of how to sample the materials which they used. The agreement arrived at by the Conference would have a very important bearing on any future consideration of the methods of analysis. That was a question which had engaged the attention of the Society of Chemical Industry some years ago. The various Sections, however, had been in entire accordance on the subject, and consequently no definite agreement had been arrived at. He thought, therefore, that the leather-trades' chemists had done good service to their own industry by the proceedings at their Conference, and he hoped that other industries would follow their example.

Dr. L. T. Thorne thought that the matters referred to in the paper would be better dealt with by influential bodies containing members having a wider range of chemical work and experience than by the members of the particular industry concerned. There did not seem to be any authoritative and representative chemical association prepared and suitable to take up these questions and give the desired help. That being the case, it was difficult to see how the narrowing of conferences was to be avoided. If it were understood that some influential body was willing to interest itself in such work, it would be of immense value to technical chemists generally.

Mr. W. P. Dreaper desired to point out that if a set of hard-and-fast rules such as had been laid before them were adopted in the leather trade, that might inflict an injustice on other industries without their having an opportunity of saying a word in their own interests. For example, tannic acid was a body largely used in the trade, and yet the dyer was allowed no voice on the methods to be adopted for estimating it. As to the hide-powder process, his opinion was that the best course would be to do away with it altogether. At present they had not the slighest idea how much gallic acid was precipitated by the hide powder. He himself had given examples showing how different were the results obtained by gelatin and by hide powder respectively. It was far more important from the dyers' point of view that they should know the "mordant value" of the extract employed, rather than the total amount of mixed acids extracted by hide powder. He believed that this could be estimated by the process he had previously brought before the Society.

Dr. F. Clowes stated that he saw some difficulty in making either the Institute of Chemistry or the Society of Chemical Industry serve as a court of reference or appeal on questions of analytical procedure. He would like to see the Association of Leather-Trades' Chemists co-operating with the Society of Chemical Industry in the way referred to by some of the speakers; at the same time he considered the course which this Association had already taken was by no means an unwise one. These gentlemen had been spoken of as if they were simply analysts, who ought to call in the aid of scientific chemists. If such were the feeling, he begged to point to the position held by Dr. Parker, Mr. Procter, and other members, who had received the highest chemical training, as evidence that the Association was competent to settle these questions amongst themselves; but if these gentlemen would discuss their own special business and would afterwards bring their decisions before that Society, he thought that was the very best course to be taken. No one regretted more than himself that the Dyers and Colourists Society had been started independently of the Society of Chemical Industry, in spite of efforts to persuade them to make common cause with the older Society.

Dr. Rideal desired to know whether at the Conference a vote was taken on the use of glucol as a substitute for hide powder, and on the use of the polarimeter.

Dr. J. G. Parker, in reply, said that he quite agreed with the answer to Mr. Hehner's question given by Mr. Seymour Jones. He could not say that all the members of their Association combined practical with scientific knowledge, but he thought that the time would come when that would be the case. Certainly of those who had attended the Conference, not more than two or three were without a degree, either English or German. He quite agreed with Mr. Wood that the question of the quality of hide powder was one of great importance. He had bought some recently in London, and had found it so impure as to be absolutely unusable. There were various brands of it at various prices. The Association had tested them all, and had found that the best obtainable at present was that made under the careful supervision of Prof. Eitner, of the Vienna Research Station. Parcels sent by him were carefully tested by the Association's method, and if they gave more than 5 mm. residue by a blank test, were returned. The addition of paper to hide powder was not an unusual occurrence. Cerych suggested the use of filter paper admixed with the hide powder in order to prevent the tannin solution forming channels, and to make the percolation of the solution through the hide powder easier; and for some materials it was satisfactory. With regard to the nature of the sediment formed, if Mr. Stewart would read the published researches on that subject he would find that, according to Mr. Procter, most of them were difficultly soluble tannins, and partly anhydrides of tannic acid. The sphere taken up by the Association had been described as a narrow one. He ventured to think, however, that if they had taken the other course suggested, and asked the Society of Chemical Industry to call a conference together to discuss these details of analysis, their request would hardly have been acceded to. What more or better could they have done than call together gentlemen whose whole lives were spent in the investigation of matters connected with their trade, let them fix the definite outlines of the best methods to be followed, and report their proceedings to that Society? They had done this, and now invited candid criticism of members on their results; but up to now their invitation had met with very little response from that point of view. In reply to Dr. Rideal, he might mention that the Report of the Association would be issued in full in the course of a few weeks, and a copy would be sent for the library of the Society of Chemical Industry. In reference to Dr. Clowes's remarks, he believed he might say, that out of the 24 gentlemen who attended the Conference, at least 20 were members of the Society of Chemical Industry. And, inasmuch as the Society did now give some attention to matters connected with the leather trades, no doubt leather-trades chemists would feel that the Society of Chemical Industry was the Society to which they should communicate the results of their researches. Unfortunately, they had no reliable method for estimating tannic acids in tanning solutions, and he had spoken all through of "tanning matters" absorbed by the hide, because that was all the practical tanner wanted to know. They had confined their attention to that aspect of the question; and, as it was too expensive to experiment on hides themselves, they had used the next best thing, *viz.*, purified hide powder. There was nothing more needed in the tanyard than an accurate method of estimating tannin, but they had not arrived at it yet. Mr. Hehner had asked whether something else was not absorbed besides the tannin. Undoubtedly colouring matter was also absorbed. But the same thing took place in the tanyard, and therefore they had made their process resemble practical conditions as far as possible.

The Chairman heartily welcomed the two papers, and hoped the authors would favour the Society with further contributions. Chemists practically connected with the leather industry had met together with a view of raising its empirical practices to more scientific methods. This could not fail to benefit the said industry and to increase our knowledge.

# Manchester Section.

*Meeting held on Friday, January 7th, 1898.*

MR. GEORGE E. DAVIS IN THE CHAIR.

## ON THE OXYGEN TEST FOR SEWAGE AND EFFLUENTS, AS CARRIED OUT IN THE LABORATORY OF THE MERSEY AND IRWELL JOINT COMMITTEE.

BY J. CARTER BELL, A.R.S.M., ETC.

The estimation of the polluting matter in effluents from sewage works, both as regards the amount, and its liability to putrescence, has been a subject much discussed in this Section. There has been a great divergence of opinion, and up to the present time no uniform method of estimating the impurities in an effluent has been adopted.

Each chemist has his own pet way of working the oxygen test, for all work at different temperatures, one even as high as 212° F. It is impossible to obtain concordant results under these conditions. Dr. Tidy, when he introduced his modified oxygen test nearly 20 years ago to the Chemical Society, said: "I am afraid the public have taken note, and are taking notes of chemists' differences, and distrust our work accordingly. Nor, indeed, is their distrust to be wondered at, deeply as it is to be lamented. To clear up these differences chemists must be prepared not merely to criticise, but to study other processes besides their own, and accurately to compare results."

Some years ago (this Journal, 1883, 109), in a paper before this Section on Standard Methods of Analysis, I pointed out the necessity for certain standards, since chemists, when analysing portions of the same material, and each working according to his own particular method, obtained very varying percentages.

I have now to bring before you a further illustration of the urgent need for what I then pleaded. The Mersey and Irwell Joint Committee lay great stress on the permanganate test for the estimation of the impurity of the effluents taken by them from the various works in their jurisdiction, and they look with disfavour upon any effluent which absorbs more than one grain of oxygen per gallon "from an acid solution of potassium permanganate, and further contains more than one-tenth of a grain of albuminoid ammonia."

I have not found an effluent by simple precipitation, and settling in tanks, that would come up to that standard, except when the sewage itself has been so weak as to be below that standard.

During the last few months many samples of effluents have been taken by the Joint Committee from the Salford Corporation Sewage Works, and have been examined by this permanganate test. When the results were reported to the Corporation they were almost invariably higher than those I reported, sometimes as much as 40 per cent.

In view of these discrepancies, the Corporation thought I must be in error, but before making any complaint samples were sent to other chemists from time to time, all of whom confirmed my results. The question then resolved itself into this, that the Joint Committee must work the permanganate test by some method different from mine. The Deputy Chairman of the River Committee applied for information as to their particular method, and the answer received was "that their process of testing was fully explained at the Salford Corporation trial before Judge Parry."

The following is the verbatim report of this particular part of the evidence:—

Mr. Bidder, Q.C., cross-examined the witness.

With regard to the figures in your analysis, Table A. of the permanganate test, I am going to put in results from the same samples that differ from yours. Ours are in most cases some 15 or 16 per cent. We get a result some 15 or 20 per cent. less than you, and it is only fair that I should put that matter in cross-examination to you to see if you can explain it.

Answer.—I used the process as described by Dr. Tidy and approved by the Society of Public Analysts. The description of the process used is found in Sutton's "Volumetric Analysis."

I maintain that the above is no answer to the question. The description to be found in Sutton is for drinking waters, and not so much for effluents; but granting that it does apply to effluents, then I say that the Mersey and Irwell do not work according to what is stated there, and approved by the Society of Public Analysts, for one of the most important recommendations given there they leave out. I speak as to the question of temperature. The Society of Public Analysts, after long deliberation, stated that the process was to be worked at 80° F. The Joint Committee work at 60° F. or lower. Dr. Tidy recommended that 250 c.c. of potable waters should be taken and 10 c.c. of permanganate. The Joint Committee take 70 c.c. of effluent and 50 c.c. of permanganate.

The Society of Public Analysts recommended that 15 minutes, and four hours respectively should be the time during which the permanganate should act upon the water at 80° F. The Joint Committee allow three minutes, and four hours at 60° F.

Our deputy chairman, through the courtesy of Sir John Hibbert, at last succeeded in obtaining a full description of their method of working, and the following is their process, in detail:—

### *Permanganate Test.*

"Process for the estimation of oxidisable organic matter as measured by the action of an acid solution of permanganate of potash on the sewage effluent.

"Two determinations are made, *viz.*, the amount of oxygen absorbed during three minutes, and that absorbed during four hours, both determinations being made at a temperature of about 60° F.

"Standard solutions required:—

1. $H_2SO_4$ solution.—1 part by volume of pure $H_2SO_4$ + 3 parts pure distilled water. Permanganate solution is added until a faint red tinge remains for some hours.

2. $KMnO_4$ solution.—0·395 grm. per litre of pure crystallised $KMnO_4$.

3. KI solution.—1 part KI to 10 parts of distilled water.

4. $Na_2S_2O_3$ solution.—4 parts $Na_2S_2O_3$ by weight dissolved in a litre of water distilled.

5. Starch water.—Process.—Take 70 c.c. of the sewage effluent in a 5 to 8 ounce stoppered bottle, add 10 c.c. $H_2SO_4$ solution + 50 c.c. $KMnO_4$ solution. Allow to stand four hours. If during this period the permanganate colour is much faded another 50 c.c. $KMnO_4$ solution is added, so as to always have an excess of permanganate present. At the end of four hours a few c.c.'s. of potassium iodide solution are added, and the iodine liberated titrated with the $Na_2S_2O_3$ solution, using starch for the end reaction.

"Each day a blank experiment is made with 70 c.c. distilled water + 10 c.c. $H_2SO_4$ + 50 c.c. $KMnO_4$, so as to standardise the hyposulphite.

"The three minutes' test is determined by directly adding $KMnO_4$ solution from a burette to 70 c.c. sewage effluent + 10 c.c. $H_2SO_4$, the colour of the permanganate forming a sufficiently delicate indicator."

Anyone reading the above will see that it differs very considerably from that described in Sutton.

The committee requested the corporation to send my method of working the permanganate test for effluents, which is as follows:—

Nos. 1, 2, and 3 solutions are exactly the same as the Joint Committee's.

No. 4. $Na_2S_2O_3$ solution. 1 grm. to the litre.

No. 5. Starch. Sutton recommends an alkaline solution. I always use the starch called "Tous les mois," 1 grm. to

300 c.c. of distilled water, brought nearly to the boiling point, then poured into a long test tube, allowed to settle during the night, and the clear liquor poured off. I find that this starch, prepared in the manner I have described, is much more delicate and sharper in its reaction than any other I have tried.

My process of working is different. I take 20 c.c. of the effluent, 50 c.c. of distilled water, 10 c.c. of sulphuric acid, and 10 c.c. of the permanganate solution in a wide-mouth, corked, dwarf flask, holding about 200 c.c. This is allowed to stand at the ordinary temperature for four hours.

The following figures will show what a considerable difference there is between the analyses of the effluents taken from the Salford Sewage Works.

*Oxygen required in Grains to Oxidise the Organic Matter in One Gallon.*

| Mersey and Irwell. | | J. Carter Bell. | |
|---|---|---|---|
| 3·9 | 3·0 | 2·3 | 2·4 |
| 2·3 | 1·9 | 1·8 | 1·4 |
| 4·4 | 2·8 | 3·4 | 1·8 |
| 3·1 | | 2·3 | |

The above are just a few instances picked out of many samples. These discrepancies may arise from two or three causes. In the first place temperature will make a considerable amount of difference, as the following table will show. A series of effluents was treated with permanganate at different temperatures, with the following results:—

*Lime Effluents.*

| 60° F. | 80° F. | 100° F. | 212° F. |
|---|---|---|---|
| 2·1 | 2·5 | 3·2 | 5·8 |
| 2·5 | 2·8 | 3·6 | 5·2 |
| 1·8 | 2·3 | 2·8 | 6·1 |

The following effluents were prepared in the laboratory from the same sample of sewage.

*Chemicals used for One Million Gallons of Sewage. Oxygen required for Four Hours at Different Temperatures.*

| —— | 60° F. | 80° F. | 212° F. |
|---|---|---|---|
| No. 1. Shaken sewage........ | 8·7 | 9·2 | 12·9 |
| 2. Settled „ ....... | 7·0 | 7·5 | 9·5 |
| 3. 25 cwt. of lime........ | 3·8 | 4·2 | 7·8 |
| 4. 15 cwt. of alumina sulphate. | 5·8 | 6·1 | 7·9 |
| 5. 30 cwt. of alumina sulphate. | 5·0 | 5·5 | 7·5 |
| 6. 7½ cwt. of perchloride of iron, and 15 cwt. of lime. | 5·8 | 6·3 | 7·6 |

The time between taking the samples and beginning the analysis has a very important bearing upon the results, and it is impossible to obtain concordant results when the interval of time varies considerably. The following will illustrate my meaning:—A sample of settled sewage was analysed just after the time of collection and was found to be 6·5 of oxygen, but after being kept in a closed bottle for a few hours in a moderately warm place the oxygen number rose to 6·9, and after about 4 days it had risen to 7·5. This was due to the generation of $H_2S$ from the decomposing organic matter. There is sometimes an increase and sometimes a decrease in the amount of oxygen required.

The following are samples taken by the Mersey and Irwell Board on the dates given below. In the first column is the oxygen required a few hours after taking the sample. The second and third columns give the oxygen required on December 29th, 1897, the absorbed oxygen being estimated in two ways. The samples between the two dates have been kept in closed bottles.

| 1897. | By J. Carter Bell's Method. | By Mersey and Irwell Method. | By J. Carter Bell's Method. |
|---|---|---|---|
| February 18th ........ | 4·2 | 0·80 | 0·50 |
| March 11th ........... | 2·6 | 1·20 | 1·15 |
| April 20th ............ | 1·8 | 1·00 | 1·12 |
| May 10th .............. | 2·4 | 0·60 | 0·84 |
| „ 18th .............. | 3·8 | 1·50 | 1·08 |
| June 24th ............. | 1·3 | 0·80 | 0·70 |
| July 8th............... | 2·3 | 1·10 | 0·63 |
| August 23rd .......... | 1·4 | 0·81 | 0·48 |
| September 9th........ | 1·8 | 1·00 | 0·73 |
| November 4th ........ | 3·8 | 1·70 | 0·90 |
| „ 9th ........ | 1·9 | 1·04 | 0·81 |

Another and third cause of variation may be due to the end reaction not being sharp enough, and this may occur when too much sewage or effluent is present. Instead of the violet colour vanishing with one drop of the hyposulphite, as it should do, a faint colour remains, which causes the operator to be in some slight doubt as to whether the reaction is really finished. That is one of the reasons why I prefer to dilute. Another source of error may arise from the hyposulphite being run in slowly or quickly. Some chemists run it in a little at a time; I prefer to run it in quickly, agitating the whole time. Another source of error is when the effluent contains suspended organic matter; this will settle and decolorise the permanganate at the bottom of the flask. Hence the flask should be constantly shaken during the four hours. Whenever anyone says that he works with the permanganate test the manner in which it is worked should be most clearly defined.

Thus, Mr. Barnes, in a paper which he read before this Section (this Journal, 1896, 82), gives the following directions for working the permanganate process:—

100 c.c. of the liquid to be tested, or a smaller measured quantity made up to this volume with pure water, are placed in a stoppered bottle, 10 c.c. of dilute sulphuric acid are then added, and finally 10 c.c. of permanganate (4 grms. per litre). The bottle is placed in a tank of water at 70° F., and allowed to remain 2½ hours, maintaining the temperature at 70° F., if necessary, by the occasional addition of hot water. Now add 10 c.c. of ferrous sulphate solution, and, after the liquid has become quite clear, titrate it with the standard permanganate (1 c.c. = 0·0005 mgrm. of O). A blank experiment is made at the same time with 100 c.c.

I have not had time to compare the above method with the Mersey and Irwell one or my own, but I am inclined to think a very serious difference would be found, as the question of temperature and time comes in very forcibly.

The colour of the permanganate may disappear, and yet there may be no organic matter in the water. The water may contain sulphate of manganese, which the permanganate would precipitate as a manganite of manganese; but on adding hyposulphite the precipitate would not interfere, as when iodide of potassium is added the precipitate is decomposed and an equivalent amount of iodine liberated. This precipitate of manganese always comes down when a large quantity of the effluent is taken, but by my method of dilution the solution remains perfectly clear.

An effluent was brought to me which required more than 6 grains of oxygen to the gallon; this had been condemned as a bad effluent, but I pronounced it good, as it contained phenol and cresol, and these acids were the cause of the large absorption of oxygen. When one is dealing with the effluents from a large manufacturing district, too much stress should not be laid upon the amount of oxygen absorbed, for the organic matter may be very stable, and take many hours before the whole of it is oxidised; in that case I look upon it as harmless, but, if the organic matter is in a rapidly putrescent state, dilution or concentration will make very little difference upon the permanganate. On this point Dr. Frankland observes: "The fallacy of the permanganate test has often been suspected; but it was imagined that, although not to be relied on for quantitative estimations, yet its rapid decoloration afforded positive evidence of the presence of organic matter in actual putrescence, and consequently in its most dangerous condition. We fear, however, that, even for this subsidiary purpose, the permanganate is not sufficiently trustworthy."

This is very true for the Manchester district, because, if the waters contained antiseptics, they would be most beneficial to the Ship Canal, but the permanganate test would condemn such effluents.

Waters were prepared containing various organic bodies, such as phenol, sulphate of quinine, &c. Permanganate was allowed to act upon these for three minutes and four hours.

| — | Oxygen required for | |
|---|---|---|
| | Three Minutes. | Four Hours. |
| Phenol, 1 grain to gallon......... | 0·87 | 1·3 |

The three minutes' test by the Mersey and Irwell method is 0·00004. This means a difference of many thousands per cent.

Sulphate of quinine, 5 grains to gall., four hours; oxygen, 0·84. Many similar bodies could be brought forward.

Seeing that carbolic acid is so largely used as a disinfectant, it is not unreasonable to think that effluents may contain one grain of tar acids to the gallon. This theory is borne out by three samples of effluent taken by the Mersey and Irwell inspector on the dates given below:—

| — | Oxygen absorbed during Four Hours. | |
|---|---|---|
| | Mersey and Irwell. | J. Carter Bell. |
| September 16, 1895 .. | 2·94 tarry smell. | 2·2 |
| " 26, " .. | 3·70 tarry smell. | 2·9 |
| November 6, " .. | 4·57 tarry smell. | 2·8 |

You will notice that to each of these the chemist to the Joint Committee has appended the words "tarry smell." These effluents must have contained tar acids to produce this odour, and thus these bodies are branded by the oxygen test as harmful.

During these particular months the whole of the Salford sewage was being purified by the Bacillite Company, and they were using lime, carbonic acid, and phenol, and therefore it is not very wonderful that the effluents at this particular time had a tarry smell.

It will be interesting to observe the oxygen test at this particular period:—

| 3 Minutes. | 4 Hours. | 3 Minutes. | 4 Hours. |
|---|---|---|---|
| 1·4 | 2·7 | 1·1 | 1·7 |
| 2·0 | 3·8 | 0·5 | 2·0 |
| 1·2 | 3·8 | 1·8 | 2·1 |
| 1·0 | 3·5 | 1·2 | 1·7 |
| 0·5 | 2·1 | 1·7 | 2·9 |

These effluents remained bright and clear for weeks, showing no signs of secondary decomposition. Yet this oxygen test classified them all as bad effluents.

Nothing was said about the phenol and cresol that were being blown into the effluent by the company, which would be equal to 1 to 1·5 grains of oxygen; and then there were the other deoxidising bodies of an innocent nature, such as are found in the effluents of all large towns. Even Tidy, when he brought this oxygen process before the Chemical Society, was disposed to register the reduction which occurs in three minutes in the absence of FeO and $H_2S$ as due to nitrites. Neither nitrites nor even salts of iron are harmful in an effluent; therefore it would be fairer, in reporting upon an effluent, to make some reduction for the three minutes' test. This test must be carried out in the orthodox way, for the difference is so great between the two methods of adding the permanganate till a pink colour is produced, or allowing it to act in excess for three minutes, the difference being, as I have already shown, some thousands per cent. The way in which the three minutes' test is conducted by the Mersey and Irwell Joint Committee is simply going back to the original process of Forchhammer, which he suggested in 1850. His method was to add a standard solution of $KMnO_4$ till a pink tinge was produced.

Dr. Tidy says: "I am free to admit that, conducted in this way, the permanganate process is irregular, inaccurate, and wanting in delicacy."

My method is to add the 10 c.c. of permanganate at once, and allow it to act upon the water for three minutes, then drop in KI, and work back with hyposulphite. The following few from many experiments will show what a considerable difference there is between the two methods:—

| Mersey and Irwell. | J. Carter Bell. | Mersey and Irwell. | J. Carter Bell. |
|---|---|---|---|
| 0·10 | 1·10 | 0·56 | 1·20 |
| 0·24 | 0·56 | 0·34 | 0·82 |
| 0·12 | 0·34 | 0·12 | 0·74 |

I think the few remarks which I have made, and the experiments which I have performed, plainly show that some reform is needed in using this test upon effluents; and all chemists will agree with me when I say that it is not pleasant to have one's analyses constantly in dispute.

The Discussion was postponed until a future meeting.

## A NEW HYDROSTATIC BALANCE.

BY DR. B. W. GERLAND.

The beam of this balance has two knife-edges of steel working on agate planes; the end one is placed parallel with the central knife-edge by an arrangement similar to that of Kuhlmann's No. 4 balances. The divisions are sharply cut by machine, and the riders have also a V-shaped edge, so that they must necessarily fall into their true position at once. In addition to a divided arc, the horizontal arm of the stand carries a circular spirit level, and the position is adjusted by two levelling screws in the feet. The end knife-edge supports a stirrup suspender with an agate plane, to which the platinum wire of the sinker is hooked. The instrument exhibited was provided with a Reimann's sinker, displacing 5 grms. of water at 15° C.; it works accurately to the fourth decimal. If desired, sinkers of solid glass for 2 or 10 grms. displacement, with convenient thermometer, are supplied.

For every balance the value of each rider in each of the ten niches is controlled by direct weighing in air before the instrument is sent out, and every care is taken to secure its utmost reliability and sensitiveness.

# New York Section.

*Meeting held at the College of Pharmacy on Friday, November 19th, 1897.*

MR. THOS. J. PARKER IN THE CHAIR.

## ON THE NATURE AND ORIGIN OF ASPHALT.

BY CLIFFORD RICHARDSON.

In 1892 I showed (J. Anal. and Appl. Chem. 6, 696), in a report to the Engineer Commissioner of the District of Columbia, on the Pitch Lake of the Island of Trinidad, that this remarkable natural phenomenon was distinguished by certain characteristics which offer some evidence as to its particular origin and that of asphalt in general.

I determined, what had hitherto not been noticed, although the view has since been adopted by others, that the lake itself is the crater of an old mud volcano or geyser which has filled up with pitch. I showed, for the first time, that the crude pitch, extending over an area of 114 acres, is of wonderfully uniform composition, and that it consists of an emulsion of water, gas, bitumen, mineral matter, and some organic matter not soluble in the ordinary solvents for bitumen. The water was proved to be a mineral water, and probably of thermal origin, as it contains borates and iodides, which also seemed to point to the thermal origin of the asphalt itself, since it is improbable that such an intimate and uniform emulsion of water, bitumen, and mineral matter could have originated in any other way than at one and the same time and place. I also called attention to the large evolution of gas, not only at the centre of the lake, but all over its surface, and to the fact that this gas is a mixture of hydrogen sulphide and carbonic acid. I showed that this evolution of gas goes on at ordinary temperatures at such a rapid rate in the soft pitch that tin cans, in which it was tightly sealed, exploded in a few days; that this was accompanied by a hardening of the material to the ordinary consistency of the large mass of the asphalt of the lake; and that with increasing age, especially where exposed to the action of the soil and weathering outside of the lake, there was a gradual deterioration and further hardening of the bitumen, as evidenced by changes in its physical properties and chemical composition, the latter being marked by the smaller proportion of the entire bitumen that is soluble in light naphtha. These, together with other similar details of interest, constituted the results of a few months' investigation of the subject after a visit to the lake in November 1891.

Since that time I have had a much more extended opportunity of studying this and other asphalts, both as they occur in nature and in the laboratory, and am enabled to present an abstract of the results of my further investigations in the following pages. I am induced to do so, not because my results are at all complete, but because the subject is now under discussion, and some of the information I have to offer may prove of interest in connection therewith.

### THE TRINIDAD ASPHALT DEPOSITS.

For a general description of the Trinidad asphalt deposits, reference must be made to my report of 1892, and to the numerous writings of others on the same subject from the time of Alexander Anderson in 1789 to the present day.*

### FORM OF THE LAKE DEPOSIT.

In 1891 all that was known of the pitch-lake deposit was purely superficial, and showed merely that it covered an area of about 114 acres; that the surface was in constant motion, as was proved by the appearance on its surface of stumps of trees brought up from below, which, after being carried to a certain height, would topple over, to be again engulfed and disappear; that there were several groups of trees or islands sustained on the pitch; that there was a pool of soft pitch of ordinary temperature at the centre where gas was evolved; and that wherever a hole was dug in the pitch, whether deep or shallow, it filled up, and the surface resumed its level after a short time. Nothing was known as to the size or depth of the deposit, the shape of the enclosing walls, or of the amount of new material which flowed into the lake each year.

In 1893 and 1894, however, when the Trinidad Asphalt Company began to establish a pier and cableway for conveying the crude pitch from the lake to vessels for shipment, a series of borings was made upon the lake by Mr. P. W. Henry, now General Manager of the Barber Asphalt Paving Company, as well as on the land between the lake and the gulf, and in the gulf to the north and west of the lake; and a line of levels was run from the shore to the lake, and across it through the centre in several directions, with stations at intervals of 100 ft., all secured by bench marks on firm ground.

The boring at the centre of the lake was carried to a depth of 135 ft., the entire distance being through pitch, which, as far as ocular evidence goes, has the same character as that at the surface. It was impossible to carry the boring deeper, as the movement of the pitch had so inclined the tube—one foot in six—which formed the lining, that it had to be abandoned. It then gradually toppled over and was engulfed. Nothing has been seen of it since. The result was sufficient, however, to show the great depth of the crater and the uniformity of the pitch. The depth attained was within a few feet—not more than three and a half—of sea level, and yet we do not know how much deeper the pitch may extend. The boring on the north side of the lake, about 1,000 ft. from the centre, and 100 feet from the edge, was in pitch of the usual character for 75 ft., showing a very steep slope to the sides of the crater. At 80 ft. a layer of fine white sand was met for a few feet, and then asphalt was again encountered. At 90 ft., sand mixed with asphalt was struck, and this continued to a depth of 150 ft.

Further borings, made at some distance from the lake, gave results near the surface which were similar to those found at the deeper levels at the edge of the lake. Sand, mixed with asphalt here and there, was the common material, while at a depth of 80 ft. on the southern side of the lake, and about 80 feet south of the road, and between 1,200 and 1,300 ft. from the centre of the lake, a very hard asphaltic sandstone was found.

All the evidence thus goes to show that the sides of the crater are of sand or sandstone, more or less impregnated with bitumen, the sandstone being no doubt the rock of the hillside toward the south against which the crater has been built up.

From the borings it was thus learned for the first time how enormous the deposit was, and the idea that the mound was really a crater seemed to be confirmed. It is, nevertheless, hard to realize that there is at this point, 138 ft. above the sea, a bowl-like depression over 2,300 ft. across, and over 135 ft. deep, reaching below the sea level, and filled with a uniform mass of pitch, which must amount to over 9,000,000 tons. Nothing less remarkable are the conclusions reached from the results of the lines of levels run across the lake.

In February 1893, four lines of levels crossing at the centre were run across the lake and secured by plugs on the shore. Bench marks of concrete were also put in on hard clay some distance back from the lake, which in 1894 were found not to have changed their level. The results of these levels of 1893 showed that the centre of the lake was about a foot higher than that portion a thousand feet outward toward the edge, and that from the latter point to the edge was a rise of six inches. The elevation of the centre above sea level was 138·5 ft.; of the station, 1,000 ft. N. 27° W., 137·5 ft.; of the edge, 1,100 ft.; from the centre, 138·0 ft.; while the top of the crater wall itself, 100 ft. further on, was 140·1 ft., and at the diametrically opposite side of the lake, 141·4 ft. There is considerable irregularity in the height of the crater wall, due, no doubt, as suggested by Peckham, to its breaking down in part under the pressure of the pitch. The highest part, as shown in my 1892 report, is to the south, with an elevation of 141·4 ft. above sea level, and the lowest toward the west and toward the north-east, where it is not less than 137·8 or 138 ft. above sea level, the highest level probably being that of the original rim, as confirmed later.

The surface of the lake, it is very evident from even a casual examination, is lower to-day than some years ago, and the deposit now seems to occupy a shallow depression. This fall is due to the removal of the crude pitch for shipment, which has reached over a million tons. From the difference in level of the surface of the lake between any two years, and from the amount of pitch removed, it is easy to calculate how many tons of it correspond to a fall of an inch or a foot. In a similar way, from the area of the surface and the density of the pitch, it is possible to calculate on another basis how much pitch should correspond to an inch or a foot in depth. From the latter figures, it appears that the lake should have fallen many more feet than it has in the last 30 years, and from a comparison of the results of the first and second calculations, extending over three periods and three sets of levels, there is no doubt that there must be an influx of fresh pitch at the soft spot, which, in the periods between 1893 to 1896, amounted to an average of from 20,000 to 18,000 tons per year. There is, therefore, no doubt that there is a vast influx of pitch into the lake at the present time, amounting to at least 18,000 long tons per year, and adding to a supply which must, from the area and depth of the crater, reach 9,000,000 tons. The enormous size and activity of this deposit are equally striking.

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* Anderson, Alex., 1789; Phil. Trans. **79**, 65. Nugent, Nicholas, 1811; Trans. Geol. Soc. of London, **1**, 63. Alexander, J. E., 1833; New Edin. Phil. Mag.; J. Franklin Inst. **15**, 337. Manross, N. S., 1855; Amer. J. Sci. and Arts, (2), **20**, 153. Wall and Sawkins, 1860; Rep. on Geol. of Trinidad. Peckham, S. F., 1895; Amer. J. Sci. (3), **50**, 30.

### Movement of the Lake Surface.

The constant movement of the surface of the lake, as shown by the course which sticks and logs take, which rise in the pitch, was well brought out in the work of running the levels which have been mentioned. Stakes driven for stations in levelling in a right line across the lake were, near the more actively moving central portion, much out of line in 24 hours, and within three weeks those at intervals of 100 ft. for 600 ft. from the centre had moved as follows :—

| | | |
|---|---|---|
| Centre 0 .... | 20·6 ft. to right .... | 12·2 ft. ahead of line. |
| „ 1 | 13·5 „ | 1·1 „ „ |
| „ 2 | 6·5 „ | 11·0 „ „ |
| „ 3 | 1·7 „ | 0·0 „ „ |
| „ 4 | 0·2 „ | 2·4 „ „ |
| „ 5 | 0·9 „ | 3·8 „ „ |
| „ 6 | 3·2 „ | 1·4 „ „ |

On one of the lines there was an island or mass of floating vegetation which was marked by a hub in 1893. In 1894 this island, 600 ft. from the centre of the lake, had moved 5·5 ft. to the left of the line, and 23 ft. in the direction of the line toward the edge of the lake. The position of the islands intersecting the lines of levels were also determined in 1896, and all of them found to have shifted their position.

By these determinations, therefore, the activity of the surface movement of the lake is definitely settled.

### Proximate Composition of Trinidad Asphalt.

When in Trinidad in 1891 I made a collection of specimens of the crude pitch, or asphalt, at intervals of 200 ft., on lines laid out on two diameters of the lake. These were examined according to the methods in use at that time, the water having been previously removed by drying at 100° C. and the solvents being applied at ordinary temperatures. The results obtained showed great uniformity in the composition of the crude asphalt, but were not entirely satisfactory, for several reasons. The specimens were taken too near the surface of the lake ; they were liable to have suffered an alteration and loss of light oils by being dried at too high a temperature ; and the methods of analysis, with the use of light naphtha as a solvent, had not been elaborated. I found soon after, that the emulsified water in the pitch could be removed very readily, without the aid of heat, by grinding the pitch to a fine powder and exposing it to the air. The rapidity with which the water evaporates and the ease with which the pitch dries is illustrated by the following determinations. A piece of crude pitch was weighed, ground, and passed through an 80-mesh sieve, and then exposed in a thin layer to the air of the laboratory. The loss of water was as follows :—

| Loss in | Per Cent. | Per Cent. of Entire Water. |
|---|---|---|
| 5 minutes................ | 2·2 | 7·0 |
| 12 „ ................ | 3·5 | 12·1 |
| 20 „ ................ | 6·5 | 22·4 |
| 30 „ ................ | 9·5 | 32·7 |
| 1 hour .................. | 16·5 | 56·9 |
| 2 hours .................. | 20·0 | 69·0 |
| 3 „ .................. | 21·0 | 72·4 |
| and after regrinding, | | |
| 4 hours .................. | 22·5 | 77·6 |
| 24 „ .................. | 29·0 | 100·0 |
| and *in vacuo* over $H_2SO_4$ | 0·6 | .. |

On exposure to air saturated with moisture, the powder of crude pitch after final drying *in vacuo*, gained in 12 hours 2·8 per cent., an amount of hygroscopic moisture which is not large, which is quite different from the emulsified water originally present, and which evaporates as soon as the bituminous cells which enclose it are broken down. In fact, the manner in which the water exists in Trinidad asphalt is quite different from that in which it is found in most other substances occurring in nature, since it cannot be removed by diffusion or osmosis, as in the case of drying a lump of clay or a vegetable structure, but only by breaking down the enclosing cell wall of bitumen. The novelty of this method of drying the crude asphalt was acknowledged by the Patent Office and a patent granted therefor. The peculiarity has since been noticed by other investigators. Owing to the ease with which the water can be removed from Trinidad lake asphalt in this way, a means was afforded of collecting the pitch and drying it at the lake, so that no change could take place in transit.

A final series of samples was collected for me in this way by Mr. P. W. Henry, during his work at the lake in February 1894. In order also to obtain true representative samples of the material forming the mass of the deposit, the surface samples were taken about 2 ft. below it, and others from the borings which have been mentioned as extending as deep as 135 ft. at the centre and 150 ft. on the edge. I was thus supplied with a set of samples which fairly represent the true character of the crude pitch as it exisits in the lake. These samples were examined as follows :—

### Method of Analysis.

Separate weighed portions of the material dried *in vacuo* over sulphuric acid were extracted in beakers with successive portions of hot carbon bisulphide and naphtha of 88° B., the decanted solvents being passed through a Gooch crucible with heavy asbestos felt. The filtrate was allowed to settle, in the case of the carbon bisulphide extract, for 24 hours, again decanted, and any fine sediment which had passed the filter brought upon it. The losses represented the bitumen soluble in the two solvents. The mineral matter was determined by direct ignition, and the organic matter not soluble, by difference. This determination by difference is too large, owing to too great loss on ignition in the determination of the mineral matter ; and too small, owing to the calculation as bitumen of some substance that is left in the bisulphide solution which is not bitumen. The two errors nearly neutralise each other, although in more recent analyses the carbon bisulphide solution is finally burned and ignited and the amount of mineral matter found subtrated from the loss taken as the amount of bitumen.

Great care is essential in these determinations, especially that the solvents be perfectly dry. On this account bisulphide is much more suitable for use than oil of turpentine or chloroform, as it is not nearly so hygroscopic, although it is not quite as complete a solvent unless used hot.

Examined in this way, the following results were obtained for the 1893 collection (see next page).

In these analyses of the surface samples an even more striking uniformity in composition is found than in those collected in 1891, owing to the care in their collection and preparation and to the method of analysis. The amount of bitumen is somewhat higher, on account of the more thorough extraction (to-day, with still further improvements in our mode of treatment, it would be even more so), but relatively we find the same evidence of a fixed proportion of bitumen, mineral matter, and organic matter not bituminous.

In the samples from the boring at the centre of the lake, which extended to a depth of 135 ft. and was still in asphalt, we find not as great uniformity, because of the way in which it was necessary to collect them by washing the particles detached by the boring machine up through the bore with a current of water and catching the material in a pail, where it was allowed to settle and the sample taken. Nevertheless the results are sufficiently close to show that the material at all depths is the same, and when they are averaged the agreement between the composition at the surface and for an average depth are remarkable.

AVERAGE COMPOSITION OF TRINIDAD LAKE PITCH IN CIRCLES.

| — | Bitumen by $CS_2$. | Mineral Matter. | Organic, not soluble. | Soluble in Naphtha. | Total Bitumen thus soluble. |
|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Circle 2, 200 ft. from centre | 55·02 | 35·41 | 9·57 | 31·83 | 57·85 |
| Circle 4, 400 ft. from centre | 54·99 | 35·40 | 9·61 | 31·63 | 57·55 |
| Circle 6, 600 ft. from centre | 54·84 | 35·49 | 9·67 | 31·85 | 58·28 |
| Circle 8, 800 ft. from centre | 54·66 | 35·56 | 9·78 | 31·67 | 57·97 |
| Circle 10, 1,000 ft. from centre | 54·78 | 35·44 | 9·78 | 31·58 | 57·64 |
| Circle 12, 1,100 ft. from centre | 54·62 | 35·45 | 9·93 | 31·77 | 57·51 |
| General average | 54·92 | 35·46 | 9·72 | 31·72 | 57·79 |
| Circle 14, 1,100 ft. from centre | 53·86 | 36·38 | 9·76 | 30·52 | 56·66 |

AVERAGE COMPOSITION OF TRINIDAD LAKE PITCH FROM THE BORING.

| — | | | | | |
|---|---|---|---|---|---|
| — | 54·66 | 35·90 | 9·44 | 31·53 | 57·67 |

The average composition of the two lots of samples from the surface and from the boring is as follows:—

| — | Bitumen. | Mineral. | Organic. | Bitumen Soluble in Naphtha. | Total Bitumen. |
|---|---|---|---|---|---|
| | | | | | Per Cent. |
| Surface... | 54·92 | 35·46 | 9·72 | 31·72 | 57·79 |
| Boring.... | 54·66 | 35·90 | 9·44 | 31·53 | 57·67 |

At the side of the lake, the boring, about 100 feet from the edge of the crater, showed similar pitch to that on the surface for 75 ft., after which there was, as has been mentioned, a change, and the composition of the borings proves to be that of sand and soil mixed with asphalt, showing that the side of the crater had been reached.

The uniformity of the proximate composition of all the pitch that exists in the lake and is newly forming there, leads me to believe also that the name Parianite, suggested for it by Prof. Peckham, as a mineral species, is entirely justified.

### BITUMEN SOLUBLE IN NAPHTHA.

In my report of 1892 I showed that between the refined products of that pitch from the Trinidad lake and that from deposits outside of the lake, known as "land asphalt," a decided difference could be detected in the relative amount of their total bitumen soluble in 88° naphtha.

Determinations, in the same way, of the amount soluble in the lake samples, collected by Mr. Henry, have proved of interest in connection with the comparison of those from the surface and from the borings. The average percentage of bitumen soluble in naphtha, in both the surface samples and those from the boring at all depths, is the same, or nearly so—57·79 and 57·67 per cent.; but on the surface itself the amount is found to diminish somewhat toward the edge of the lake, and in the boring the average for the first 70 ft. is 56·83 per cent., while for the second it is 58·50 per cent. The effect of age is seen in these cases much in the same way as, to a much greater extent, is found to be the case with the land asphalt.

### THE DEPOSITS OF LAND ASPHALT.

Beyond the boundaries of the Trinidad pitch lake, and between it and the sea, and even in the sea itself, are found deposits of crude pitch very similar to that in the lake, or in some stage of alteration which permits of their recognition as being originally derived from such material. The character of the pitch from these deposits, which is technically known as land pitch, was discussed in my earlier reports. The subject has been one of great technical interest ever since.

There is no doubt that a large area to the north and east of the lake contains large quantities, and it has been an open question as to how it got there. Manross describes an overflow from the lake, and to this is undoubtedly due the presence of pitch on the lands adjoining it and for some distance toward the sea to the east, but a large amount of the pitch must has been ejected independently of the lake source of supply, but so long ago that it has now been buried with soil. There has been no overflow for years, nor any evolution of fresh pitch at any point in the neighbourhood of the lake, except one or two small cones, so that there is no activity to-day which will reveal what happened in past centuries. The pitch has reached the sea shore in many instances, and even forms reefs beyond it. It is spread out on the Point d'Or estate to the east of the lake, evidently in a large sheet, which seems more like an evolution of pitch independent of the lake supply than any other. This has been overgrown for some time, and its age, which can only be determined by the changes which analysis would show it to have undergone, is doubtful.

Where the pitch comes in contact with salt water, it is hardened and does not rot, and to this cause are due the asphalt pebbles of all sizes and shapes found upon the beach. Where exposed to the alternate action of the sea water and air, the pitch is converted into alteration products, which will be noticed later in this paper.

It is evident that the pitch forming the land deposits is, as a whole, of very varied character, and to be found in all stages of alteration. The results of a chemical and physical examination of some of these products were given in my 1892 report. They have been considerably extended since that time.

### PROXIMATE COMPOSITION AND PROPERTIES OF TRINIDAD ASPHALT FROM THE LAND DEPOSITS.

From the results of the examinations of the specimens of pitch collected by me in 1891 from the land deposits, the conclusion was drawn that the relative proportions of bitumen, organic matter not soluble, and inorganic or mineral matter in this form of pitch were not far different, in carefully selected material from which all oxidised and and altered pitch has been excluded, from those found in the lake substance. The amount of water was, however, proved to vary somewhat in the land pitch, and in the dry material the proportion of the entire bitumen which was soluble in naphtha of low specific gravity —0·63 to 0·64—was shown to be decidedly smaller in the land than in the lake samples, while the physical properties of the two kinds of asphalt also proved different. As these results and conclusions were of great commercial and technical importance, and as the latter had been questioned by certain persons, it seemed advisable to repeat the determinations with a new collection of samples from all the land deposits which were available, drying out the water *in vacuo* after pulverising, so that the bitumen might not be in any way altered by heat.

Examinations of a collection of representative land pitch specimens, made for me by Mr. Kearney in 1893, at the same time that he gathered a series of lake samples, resulted as follows, the naphtha used having a considerably greater solvent power than that in use in examining the samples collected by Mr. Henry.

### Analyses of Land Pitch, Dried *in Vacuo*, Kearney Collection.

*No. 1.—Specimens from Lot C., nearest the Lake.*

| — | Bitumen-soluble $CS_2$. | Mineral Matter. | Organic, not soluble. | Bitumen-soluble Petroleum Naphtha. | Total Bitumen-soluble. |
|---|---|---|---|---|---|
|  | Per Cent | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Average...... | 54·03 | 36·49 | 9·48 | 33·02 | 61·11 |

*No. 2.—Specimens from Crown Lands Lots adjoining C.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 53·81 | 36·62 | 9·57 | 32·29 | 60·01 |

*No. 3.—Specimens from East of Road, Middle Ground.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 53·31 | 37·80 | 9·89 | 31·25 | 59·74 |

*No 4.—Specimens from Village Lots, nearest the Gulf.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 52·27 | 37·73 | 10·01 | 31·42 | 60·12 |

### Average Composition of Lake Pitch, Dried *in Vacuo*, Kearney Collection.

| — | Bitumen-soluble $CS_2$. | Mineral Matter. | Organic, not Soluble. | Bitumen-soluble Petroleum Naphtha. | Total Bitumen-soluble. |
|---|---|---|---|---|---|
|  | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Average...... | 54·25 | 36·51 | 9·24 | 35·41 | 65·27 |

### Average Composition of Land Pitch, Dried *in Vacuo*, Kearney Collection.

*Eight Specimens from Lot C., near the Lake.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 54·03 | 36·49 | 9·48 | 33·02 | 61·11 |

*Four Specimens from Crown Land Lots adjoining C.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 53·81 | 36·62 | 9·57 | 32·29 | 60·01 |

*Five Specimens from East of Road, Middle Ground.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 52·31 | 37·80 | 9·89 | 31·25 | 59·74 |

*Seven Specimens from Village Lots, near the Gulf.*

|  |  |  |  |  |  |
|---|---|---|---|---|---|
| Average...... | 52·27 | 37·73 | 10·01 | 31·42 | 60·12 |
| General average. | 53·10 | 37·16 | 9·74 | 31·09 | 60·14 |

These averages enable us to compare the composition of the pitch from various parts of the deposits outside the lake among themselves, as well as with that of the lake pitch analysed under similar conditions. It appears in the most striking way that the further from the lake the pitch is found, the more it shows signs of age, as evidenced by the increase of the percentage of organic matter not soluble, that is to say, of altered bitumen and of mineral matter, and, generally, a decrease in the per cent. of the total bitumen which is soluble in the naphtha used. The lake pitch has, with the naphtha used as a solvent, 65·3 per cent. of its bitumen soluble in naphtha, while just outside the lake the land pitch has only 61·1, and further on only 59·7 per cent. soluble. This may seem a small difference, but it is evidence of a large change. In a glance pitch, examined in the same way, 49 per cent. only of the entire bitumen was found to be soluble in naphtha, in lake pitch 65·3. Land pitch may, therefore, be inferred to be about a quarter converted from lake to glance pitch.

The relations between the composition of the two kinds of pitch has also been shown in comparing the results of analyses of some land samples collected by Mr. Henry at the same time that he gathered the lake specimens. Analysed with them, the three sets of material were found to have the following average composition:—

*Comparative Composition of* 1894 *Samples.*

| — | Bitumen. | Organic insoluble. | Mineral inorganic. |
|---|---|---|---|
| Lake, 2ft. below surface | 54·92 | 9·72 | 35·46 |
| „ boring, 135 ft. deep at centre. | 54·66 | 9·44 | 35·90 |
| Land.................... | 52·36 | 11·24 | 36·40 |

| — | Bitumen soluble in Naphtha. | Total Bitumen soluble in Naphtha. |
|---|---|---|
|  |  | Per Cent. |
| Lake, 2ft. below surface | 31·72 | 57·79 |
| „ boring, 135 ft. deep at centre. | 31·53 | 57·67 |
| Land.................... | 29·02 | 55·43 |

The same relative differences are seen as in the previously mentioned series.

Again, we have the evidence of another investigator, Prof. S. F. Peckham, who gives, in the American Journal of Science for March, 1896, a series of analyses of specimens of crude lake and land pitch which he collected personally in 1895. I have rearranged these analyses, classifying them according to their lake or land origin, and leaving out one analysis of lake pitch, No. 21, which is plainly in error. Averages derived from these analyses, which are as follows, although carried out on somewhat different lines from mine, permit of the same conclusions in regard to the changes which have taken place in the land samples:—

Crude Lake Pitch.

|  | Percentage soluble in |  |  |  | Percentage of Total Bitumen only soluble in |  |  |
|---|---|---|---|---|---|---|---|
| — | Petroleum Ether. | Boiling Spirits Turpentine. | Chloroform. | Total Bitumen. | Petroleum Ether. | Boiling Spirits Turpentine. | Chloroform. |
| Average | 35·2 | 12·4 | 5·2 | 52·8 | 66·8 | 23·3 | 9·9 |

Crude Land Pitch.

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| Average | 33·3 | 11·9 | 6·5 | 51·7 | 64·7 | 23·0 | 12·3 |

*Iron Pitch.*

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| No. 3... | 33·6 | 13·8 | 9·9 | 57·2 | 58·7 | 24·1 | 17·2 |

The additional determinations of bitumen made by Prof. Peckham with chloroform also furnish some additional and conclusive evidence of the differences between the two kinds of material. These show that the average land specimen contains 2·4 per cent. more of its bitumen in this difficultly soluble form, while iron pitch, which is acknowledged to be of no value for paving purposes and is always rejected in digging land pitch, has 7·3 per cent. more of its bitumen in this form. From this we can draw the inference that the land pitch collected by Prof. Peckham is one-third converted into iron pitch, and of so much less value than lake pitch for paving purposes. In fact, his results are as conclusive evidence of the differences between lake and land pitch as any that have been offered, and confirm the results of experience with the asphalt from the land deposits in the laying of street pavements.

### Composition of Soft Pitch and Pitch from Blow Holes.

As has been shown, the soft pitch is in an active state of change, so that it might be reasonably supposed that it would vary somewhat in composition from the general run of pitch in the lake. A specimen of this material, after standing until it had ceased to give off gas, was examined, according to the improved methods, with the following results:—

| | |
|---|---|
| Loss at 100° C. | 28·89 |
| Loss on melting | 31·36 |

The melted material had the following composition:—

| | |
|---|---|
| Bitumen | 63·4 |
| Organic insoluble | 2·8 |
| Mineral | 33·8 |
| | 100·0 |

The amount of mineral matter and water is the same as in the ordinary pitch, but the percentage of bitumen is higher, and not so much has been converted, with evolution gas, into the insoluble form. On heating to the melting point, there is also a loss of gas, which corresponds to that which would take place slowly under ordinary circumstances in the lake with the gradual change and hardening of the material.

Samples of comparatively soft pitch from a blow hole, a foot or two in diameter, near the power plant, a few feet beyond the edge of the crater, were examined to determine if this was from the same source as the rest of the deposit, and with the following results.—

| | Per Cent. |
|---|---|
| Bitumen | 54·0 |
| Organic | 9·1 |
| Mineral | 36·9 |

These blow holes, of which two were seen near the lake, therefore, must be connected with the main supply; but they are not accompanied in their evolution by any water, as at the centre of the lake.

### Water in the Pitch and in the Lake.

The analyses of the crude pitch in 1891 showed that there was a loss, on heating the samples that had been brought from the lake without drying to 100 C., of from 25·8 to 30·6 per cent. This was, of course, largely water; but in some cases, at the comparatively high temperature employed, some gas and light oil was driven off, while in others there had been a loss of water in transit. In the dried samples of 1891 it was, of course, impossible to determine the water, but this has been done with carefully taken samples of the crude pitch from large and fresh cargoes as soon as they were discharged in New York. In this way it has been found that the amount varies but little from the limits, 28·5 to 29 per cent. On this basis the composition of the crude pitch, as taken from the lake by Mr. Henry, although analysed in a dried condition, was-

| | |
|---|---|
| Water | 28·5 |
| Mineral matter | 25·4 |
| Organic, not soluble in hot carbon disulphide | 6·9 |
| Bitumen | 39·1 |

Of the character of the water in the crude pitch, it was shown in 1892 that it was strongly mineral. Attempts to collect it in any other way than by melting the crude pitch and allowing it to rise to the surface have been unsuccessful, although it would be more satisfactory to collect it at ordinary temperatures and without condensation or change. As thus secured from the still in which the fire-refined asphalt is produced, and in a somewhat concentrated condition, it had the following characteristics:—

| | Grms. per Litre. |
|---|---|
| On evaporation the residue was equivalent to | 19·872 |
| Dried at 100 C. | 17·484 |
| " 130 C. | 17·093 |
| Ignited gently | 15·011 |

An analysis of a more concentrated specimen was given in my 1892 report.

These specimens of water from the stills must, of course, have become very much concentrated beyond their normal condition, but they illustrate its nature as well. It contains, as most striking constituents, iodine and borates (characteristic of thermal water), and ferrous sulphate, which gives it a strong acid reaction. There is also a remarkably large percentage of ammonia, the presence of which is of great interest. The principal salts are chlorides and sulphates, and its acidity is sufficiently great to attack and corrode deeply the steel buckets in which the crude pitch is brought from the lake to the shore. In fact, the ground and surface water, wherever it comes in contact with the crude pitch, acquires an acid reaction, and has been found unsuited for use in boilers.

### Mineral Spring in the Lake.

In a further investigation of the soft pitch, and the manner in which it appears in the lake, it was observed that in addition to the evolution of gas, there was also a large volume of water brought up with it, which deposited sulphur on standing. Some of this was collected for me by Mr. Carleton Greene, and on analysis gave the following results. It was obtained by making a depression in the soft pitch, where no rain and surface water could flow in, and collecting what gathered there. Unfortunately it had suffered considerable alteration before reaching this country:—

| | Grms. per Kilo. |
|---|---|
| Specific gravity | 1·0599 |
| Solids at 110° C. | 82·100 |
| Sodium, Na | 27·193 |
| Potassium, K | 0·528 |
| Chlorine, Cl | 38·210 |
| Sulphuric acid, $SO_3$ | 3·207 |
| Calcium oxide, CaO | Trace |
| Magnesium oxide | 0·506 |
| Carbonic acid, $CO_2$ | 3·701 |
| Silica, $SiO_2$ | 0·222 |
| Organic matter | ? |
| | 73·563 |

The composition of this water proved very surprising. It was natural to suppose that it would be the same as, or similar to, that in emulsion in the crude pitch, but instead it appears to be alkaline in reaction, to effervesce with acids, to be enormously rich in salts, and quite a different water. In the amount of salts it contains, it is ten times as strong as sea water, and equal in strength to the Hunyadi Janos spring, although the salts are almost entirely chlorides and not sulphates. The water, on standing for some time, deposits a large amount of sulphur from decomposition of hydrogen sulphide, and this, with the presence of carbonates, may explain why, on the removal of pressure as the water approaches the surface, there is such an evolution of carbonic acid and hydrogen sulphide. The blowing off of puffs of the gas from time to time in the soft pitch is due to its collection in masses sufficiently large to force their way through it. An explanation of why an alkaline water is found free, and only mixed with the soft pitch while an acid water holding ferrous sulphate in solution is emulsified with it, remains to be found.

It is evident, however, that this mineral spring at the lake's centre is the means of bringing the fresh asphalt to the surface, and is, perhaps, as much responsible for the evolution of gas as is the pitch itself.

### Gas Evolved by the Pitch.

It was shown in 1892 that the crude pitch in the lake continues to evolve gas after it has turned quite hard, and also when it is heated and melted. This gas proved to be principally hydrogen sulphide and carbonic acid at the soft spot, and it seemed of interest to learn whether the same or other gases are evolved on heating. For this purpose some fresh pitch from the lake was placed in a flask and heated in a bath, very gradually at first, and afterwards approaching the fusing point. The evolved gas was at first all carbonic acid, and at higher temperatures all hydrogen sulphide. No other gases were found, and especially no marsh gas.

The same gases are, therefore, evolved on heating the crude pitch as are found at the lake's centre, but hydrogen sulphide only at a comparatively high temperature. How much of each gas is derived from the water in the pitch, and how much, if any, from the reducing action of hydrocarbons on sulphates, or from splitting off of hydrogen sulphide by hydrocarbons in the act of condensation, has not yet been determined.

### MINERAL MATTER IN THE PITCH.

The mineral matter in Trinidad asphalt consists of silica principally, accompanied by some clay, oxide of iron, and the substances soluble in the water of the crude pitch.

An analysis of the entire ash left on burning the crude pitch resulted as follows :—

| —— | Soluble in Acid. | Insoluble. | Total. |
|---|---|---|---|
| Silica, $SiO_2$ | .. | 70·64 | 70·64 |
| Alumina, $Al_2O_3$ | 7·38 | 9·66 | 17·01 |
| Ferric oxide, $Fe_2O_3$ | 6·30 | 1·32 | 7·62 |
| Lime, CaO | 0·46 | 0·24 | 0·70 |
| Magnesia, MgO | 0·11 | 0·79 | 0·90 |
| Soda, $Na_2O$ | 1·56 | .. | 1·56 |
| Potassium, K | 0·35 | .. | 0·35 |
| Sulphuric oxide, $SO_3$ | 0·97 | .. | 0·97 |
| Chlorine, Cl | 0·22 | .. | 0·22 |
| | 17·35 | 82·65 | 100·00 |

The silica under the microscope appears in the form of flakes with sharp fracture. It seems probable, as suggested by Dr. Carl Barus and Prof. Peckham, that it must have been deposited from solution in the water found in the pitch. The particles are naturally much larger than those of the rest of the mineral matter, although in themselves very small. By treatment of the ash with strong acid to remove the soluble portion, the silica can be obtained in a white condition, but mixed with some impalpably fine white clay, which can be separated by decantation. It will be seen that the insoluble portion of the mineral matter of Trinidad pitch contains 85·4 per cent. silica.

Some of the mineral matter is so impalpably fine that it will not separate from a solution of melted or dried Trinidad patch in any of the usual solvents even after days of standing and many hours' treatment in a centrifugal. It will pass also through the finest filters. It has been thought by Peckham and others, on this account, to be chemically combined with the organic compounds of the asphalt, but I have found that by continued swinging in a centrifugal it can be so far reduced that it amounts to but 2 per cent., and is plainly only in a state of mixture with the bitumen, since an analysis of the very finest portion recovered by burning the pure bitumen thus swung out, shows that it is a ferruginous clay, and could not possibly be combined with organic matter, since it consists of silicate of alumina, and a very considerable amount of sulphuric acid, as well as oxide of iron.

*Analysis of Finest Mineral Matter.*

| —— | Insoluble in HCl. | Soluble. | Total. |
|---|---|---|---|
| $SiO_2$ | 32·36 | .. | 32·36 |
| $Al_2O_3$ | 6·74 | 33·64 | 40·38 |
| $Fe_2O_3$ | 1·40 | 11·74 | 13·14 |
| CaO | 0·45 | 3·20 | 3·65 |
| MgO | 0·34 | 1·40 | 1·83 |
| $K_2O$ | .. | 1·18 | 1·18 |
| $Na_2O$ | .. | 0·53 | 0·53 |
| $O_3$ | .. | 7·16 | 7·16 |
| | 41·29 | 58·94 | 100·23 |

### RESIDUAL MINERAL MATTER FROM WEATHERING OF PITCH.

On the shore at Brighton near the pier, and in several places along it toward Guapo are found brilliant red cliffs or ledges, some of which have been termed porcellanite by Wall and Sawkins and others. A careful study of these in 1895 convinced me that at least that one at Brighton owed its origin to the weathering out of a mass of pitch which had run down to the shore, hardened and then lost its organic matter. The bright red colour is characteristic of the mineral matter of Trinidad pitch and the material of the ledge reminded one, who is accustomed to see the ash of pitch constantly in the laboratory, of this substance. A careful examination enabled me to collect specimens of the material in all stages of conversion. As at first oxidised it becomes a rotten friable mass of black material, which with the further disappearance of the organic matter, cements itself together into the hard red mineral. I have no doubt that some of the other similar red cliffs along the shore may have originated in the same way with, perhaps, modifications of the conditions under which the change has taken place. The red mineral from Brighton is porous, rather easily reduced to a powder, and has the following composition :—

| | |
|---|---|
| Water at 110° C. | 0·31 |
| Loss on ignition | 2·69 |
| Silica, $SiO_2$ | 69·34 |
| Alumina, $Al_2O_3$ | 12·40 |
| Ferric oxide, $Fe_2O_3$ | 12·54 |
| Lime, CaO | Trace |
| Magnesia, MgO | 0·79 |
| Soda, $Na_2O$ | 1·30 |
| Potash, $K_2O$ | 0·69 |
| Sulphuric oxide, $SO_3$ | Trace |
| Total | 100·06 |
| Soluble in HCl | 12·70 |

Peckham (Amer. Phil. Soc. 1897, Feb. 5) has described a similar alteration product of asphalt in Ventura County, California, and attributes the result to the action of heat. The "expulsion of carbon" is as complete in Trinidad as in California, and yet the transition products show no signs of heat as an element in the process. It has been purely one of slow oxidation, aided by the hardening action of salt water, which, perhaps, is necessary in this case, as the red product is only found along the shore.

### CLAY.

Underlying these masses of altered pitch on the beach at Brighton, is a bed of grey clay with streaks of oxide of iron, and underneath this, yellow sand of which the beach is formed. This clay is very greasy and has the following composition :—

| | |
|---|---|
| Water at 110° C. | 2·40 |
| „ on ignition | 7·67 |
| Silica, $SiO_2$ | 63·56 |
| Alumina, $Al_2O_3$ | 17·42 |
| Ferric oxide, $Fe_2O_3$ | 6·00 |
| Lime, CaO | Trace |
| Magnesia, MgO | 0·89 |
| Soda, $Na_2O$ | 0·41 |
| Potash, $K_2O$ | 1·84 |
| Sulphuric oxide, $SO_3$ | Trace |
| Total | 100·19 |
| Soluble in HCl | 14·69 |

The coincidence in composition between this clay and that found in the asphalt is striking. When mixed thoroughly with water, dried, and reduced to a powder, it has a colour which is quite like that of the mineral residue of the pitch and it would seem at first glance as if this clay might be the source of that found in the lake asphalt, especially as the bed dips under the lake. It is, however, impossible to explain how it could be in any way mixed with the bitumen at such a high level in the regular and fixed proportion in which it occurs in the pitch.

It is of interest in this connection to note that under the land pitch deposits where they have been carried to a sufficient depth in the lots in the village of La Brea, a clay is encountered which is described as black and oily and that no asphalt is found below this, while in five borings extending at intervals from the shore, 1,700 ft. out into the Gulf, along the line of the iron pier, there have been found strata of clay below the silt forming the bottom, and belonging probably to that formation described by the geologists who have named them in Trinidad as the Moruga clays. Sections of these borings are as follows :—

| | Distance from Shore Line of High Tide. | | | | |
|---|---|---|---|---|---|
| | 539 ft. | 895 ft. | 1,038 ft. | 2,426 ft. | 1,697 ft. |
| Brown silt | 15' | 12' | .. | 11 | 15' |
| Pocket of asphalt | .. | .. | 11' | .. | .. |
| Clean sand and shells | .. | .. | 25' | .. | .. |
| Grey clay, mixed with shells and asphalt. | 20' | 17' | .. | 8' | 0 |
| Brown asphaltic clay | 20' | 13' | .. | 12' | 0 |
| Greasy grey clay | + | + | 4' + | 6' + | 7' + |

These sections are interesting as they are in part oily, and must also from their situation, run under the lake. The asphalt may be in part in place, although some is adventitious. The pocket of sand and asphalt is evidently a spring hole in which bitumen brought up by the spring has collected, thus seeming to prove that the source of the bitumen is still far below the level of the clays, and to illustrate how water alone may be the means of moving bitumen without recourse to that more powerful agent, steam, suggested by Peckham as an important agent in the transfer.

### ORGANIC MATTER NOT SOLUBLE.

This cannot be separated from the mineral matter in Trinidad asphalt after the removal of the bitumen with solvents, but its ultimate composition can be obtained by a combustion of the mixture of mineral and organic matter and the calculation of the results to an inorganic free basis. In this way the following results were obtained :—

| — | As obtained. | Organic Matter. |
|---|---|---|
| Carbon | 10·60 | 52·65 |
| Hydrogen | 1·55 | 7·69 |
| Sulphur | 2·08 | 10·32 |
| Nitrogen | 0·12 | 2·05 |
| Mineral | 79·85 | |
| Oxygen by difference | 5·50 | 27·29 |

It appears that this substance is one that is very much oxidised and one rich in sulphur and very likely an oxidation product of the original bitumen of the pitch. Its composition and properties, as well as that of the bitumen of the pitch, show that the products of oxidation of the Trinidad bitumen are not soluble oxygen derivatives of the hydrocarbons but insoluble matter.

This is also shown by the eventual conversion of the entire bitumen where it has been exposed to weathering into either the chocolate powder or soil, mentioned in my previous report, or into a black resonant and insoluble mass where it has been protected by the action of salt water as in the asphaltic ledges and reefs along the shore.

The protective action of salt water on Trinidad asphalt is well illustrated by the examination of a sample of a lot of the refined material which lay in salt water by accident for several years, which resulted as follows :—

| | Per Cent. |
|---|---|
| Bitumen | 55·7 |
| Organic insoluble | 9·0 |
| Mineral | 36·3 |
| Total | 100·0 |

The nitrogen in the insoluble organic matter may be largely due to the ammonia salts found in the water of the pitch.

### THE BITUMEN OF TRINIDAD ASPHALT.

The bitumen of Trinidad asphalt can be separated from the mineral matter and organic matter not bituminous by solvents and, as has been seen, amounts to about 39 to 40 per cent. of the crude pitch, or 54 to 57 per cent. of the dried substance. For purposes of investigation it is more safely extracted with chloroform, at a boiling temperature. It is freed from suspended mineral matter by subsidation and long treatment in a centrifugal machine. As thus prepared, it contains about 2·3 per cent. of clay and iron oxide which, thus far, it has been impossible to remove.

The total bitumen of Trinidad asphalt is a brilliant glossy pitch like substance which has a semi-conchoidal fracture when struck a sharp blow, but which yields to gentle pressure and slowly flows at summer temperatures. It softens rapidly at 76° C., and flows quickly at 83° C., but is not liquid until above 100° C. It has a specific gravity, as extracted, of 1·071 at 25° and, after correction for the 2·6 per cent. of mineral matter present, of 1·032.

The ultimate composition of several preparations which involved complete extraction, was determined with the precautions necessary in burning such organic substances, and gave the following results :—

*Total Bitumen in Trinidad Lake Asphalt.*

| Preparation. | I. | IV. | V. | Average. |
|---|---|---|---|---|
| Carbon | 82·59 | 81·95 | 82·44 | 82·33 |
| Hydrogen | 10·74 | 10·51 | 10·81 | 10·69 |
| Sulphur | 6·04 | 6·54 | 5·90 | 6·16 |
| Nitrogen | 0·51 | 0·92 | 1·00 | 0·81 |
| | 99·88 | 99·92 | 100·15 | 99·99 |

This bitumen is characterised by the large percentage of sulphur which it contains, and the presence of nitrogen. There are apparently no oxygen derivatives present in the bitumen or in very minute amounts.

On treatment with bromine the following absorption and liberation of hydrobromic acid took place :—

| — | I. | II. |
|---|---|---|
| Br absorbed | 30·42 | 29·99 |
| HBr liberated | 12·19 | 12·58 |

The comparatively large absorption of bromine might be thought to be due, at least in part, to the sulphur compounds, but it is no larger than with many soft asphalts with a small amount of sulphur, so that it would seem that the absorption is due rather to the presence of a large amount of unsaturated hydrocarbons of which the bitumen is composed.

Determinations of the bromine absorption of a number of other asphalts and malthas show that there are no distinguishing or marked differences between them, based upon their bromine absorption, but that the amount is large with all. Following are some results :—

BROMINE ABSORPTION AND SULPHUR IN ASPHALTS, &c.

| — | Sulphur. | Bromine Absorption. | HBr. |
|---|---|---|---|
| Trinidad oil spring | .. | 29·21<br>21·09 | 18·20<br>10·87 |
| „ pure bitumen | .. | 30·42<br>29·99 | 12·19<br>12·58 |
| "Petrolene" Trinidad | 3·25 | 24·16 | 9·54 |
| "Petrolene" Bermudez | 3·01 | 19·75 | 8·49 |
| "Asphaltene" Trinidad | 10·07 | 32·89 | 7·43 |
| "Asphaltene" Bermudez | 4·78 | 25·38 | 7·35 |
| Bermudez soft asphalt, middle of lake | 5·23 | 29·71<br>21·22 | 19·25<br>15·31 |
| Bermudez softer asphalt, middle of lake | 5·87 | 21·80<br>32·69 | 13·74<br>18·89 |
| Bermudez softest asphalt, middle of lake | .. | 20·83<br>26·78 | 12·19<br>15·74 |
| Bermudez oil volatilised from the asphalt | 3·31 | 23·21<br>23·11 | 12·21<br>14·07 |
| Asphalto maltha, California | 1·40 | 23·22<br>23·73 | ..<br>15·31 |
| Pedernarles refined | 2·29 | 39·16<br>38·54 | 16·09<br>16·83 |

### BROMINE ABSORPTION AND SULPHUR IN ASPHALTS, &c. —*continued.*

| —— | Sulphur. | Bromine Absorption. | HBr. |
|---|---|---|---|
| Alcatraz liquid asphalt ......... | 1·32 | 25·40<br>29·31 | 16·95<br>19·85 |
| Asphalt cement from Berlin surface........................ | .. | 28·86<br>25·89 | ..<br>.. |
| Pittsburg flux..................... | 4·22 | .. | .. |
| Kerosene ........................... | .. | 3·18<br>3·62 | ..<br>.. |
| Petroleum residuum, Penn...... | 0·60 | 20·80 | 16·40 |
| " " " ...... | 0·46 | 22·15 | 15·82 |
| " " Ohio...... | 2·26 | 34·07 | 19·72 |
| " " " ...... | 2·07 | 33·41 | 11·23 |
| Asphalt oil distilled *in vacuo*, 210°—220°. | 1·15 | 23·07 | 9·16 |
| Asphalt oil distilled *in vacuo*, 260°. | 0·84 | 23·80 | 7·11 |
| Asphalt oil distilled *in vacuo*, 270°. | 0·55 | 21·42 | 8·82 |

### BITUMEN OF LAND ASPHALT.

With a view of comparing the bitumen of lake and land asphalt for the purpose of seeing whether the changes, which the latter has been shown to have suffered by age and weathering, would affect its ultimate composition, carefully made preparations of the bitumen from authenticated samples of land asphalt were analysed with the following results:—

| —— | II. | III. | Average. | Average Lake. |
|---|---|---|---|---|
| Carbon .................. | 85·71 | 83·65 | 83·68 | 82·33 |
| Hydrogen ............... | 10·92 | 10·76 | 10·84 | 10·69 |
| Sulphur ................. | 5·21 | 4·98 | 5·10 | 6·16 |
| Nitrogen ................ | .. | 0·45 | 0·45 | 0·81 |
| | .. | .. | 100·07 | 99·99 |

It appears that the changes which the bitumen has undergone since its original formation are as well marked by the difference which they have produced in its ultimate composition as in its solubility in naphtha. There has been a loss of sulphur, due, perhaps, to its evolution as hydrogen sulphide, as well as to oxidation, and a relative increase in the proportion of carbon which would continue with further hardening.

### NAPHTHA-SOLUBLE BITUMEN.

As has been explained, I showed in my first report that by means of light naphtha of 88° B. the bitumens of asphalts could be separated into portions soluble in this medium and insoluble in it although soluble in carbon disulphide, chloroform, and oil of turpentine. That this separation is exact and quantitative, appears from our examination of the various samples from the pitch lake where the amount removed by the same kind of naphtha from all of them varied within very narrow limits. I have also shown that the relative proportions of bitumen soluble in naphtha and insoluble in it is a fair index, technically at least, of its constitution. Other solvents of a more definite character have been suggested, such as acetone and ethyl ether, but none has served as well practically as naphtha, because the latter is cheap, works readily, and has medium solvent powers. The only objection to it is that different naphthas of the same gravity may have different solvent powers. This is, however, put aside by making the determination a relative one and always comparing the results obtained with any new material with the action of the same solvent on a standard asphalt, such as Trinidad lake pitch.

The bitumen of Trinidad lake asphalt separated in this way by naphtha yields two substances of entirely different character. The bitumen obtained by the complete extraction of dried crude Trinidad asphalt with light naphtha is a soft and extremely sticky mass which flows very slowly at ordinary temperatures, is a deep amber brown colour in thin layers, and has a specific gravity of 0·9935 at 20°C./20° C. As thus prepared it has the following ultimate composition:—

| | Per Cent. |
|---|---|
| Carbon.................................... | 84·59 |
| Hydrogen ................................ | 11·30 |
| Sulphur.................................... | 2·91 |
| Nitrogen ................................. | 0·59 |
| | 99·39 |

It is marked by having a much smaller per cent. of sulphur than the entire bitumen, 2·91 as compared to 6·16 per cent., the greater portion of the sulphur compounds being insoluble in naphtha.

Its bromine absorption is, however, much the same as that of the original bitumen, while the nitrogenous substances do not seem to be all found in the naphtha solution.

### NATURE OF THE SULPHUR DERIVATIVES.

If the bitumen soluble in naphtha is treated while in solution in the naphtha with an alcoholic solution of mercuric chloride and acetate of soda, a plentiful precipitate of the mercury compound of the sulphur derivatives of the hydrocarbons is obtained showing that they are probably mercaptans and sulphides, but it has not been further examined. All the sulphur compounds are, however, not of this kind, as they are not all removed in this way, and in the tar from the treatment of the oil with sulphuric acid other forms exist which can be recovered with steam as will be described later.

### NAPHTHA-SOLUBLE BITUMEN PURIFIED WITH $H_2SO_4$.

If the naphtha-soluble bitumen, while still in naphtha solution, is treated several times with common concentrated sulphuric acid, a heavy tar-like sludge is formed, and a large part of the sulphur compounds, the aromatic substances, some unsaturated hydrocarbons and the nitrogenous bases are removed from the oil. It then has the following composition:—

| | Per Cent. |
|---|---|
| Specific gravity........................ | 0·9758 |
| Carbon................................... | 86·44 |
| Hydrogen ............................... | 12·66 |
| Sulphur ................................. | 0·45 |
| Nitrogen ................................ | 0·07 |
| | 99·62 |

These figures show that the nitrogenous bases and sulphur compounds are left in but very small amounts, and that we are dealing with a mixture of unsaturated hydrocarbons. It is a deep brown amber colour, transparent in layers of an inch in depth, of the consistency of heavy cylinder oil and without fluorescence. It has usually an odour which is aromatic, and which shows that the low hydrogen is due to the presence of hydrocarbons of the aromatic or allied series.

### PARTIAL EXTRACTION WITH NAPHTHA.

If the extraction with naphtha is not carried to completion the bitumen obtained after treatment of the naphtha solution with sulphuric acid is much thinner and lighter-coloured than that obtained with complete extraction. It is more aromatic, the odour of these compounds not being so much disguised. It has a specific gravity of 0·9511, 25°—25°, and slightly fluorescent and excessively sticky. Where exposed to the air for a year or more this heavy oil hardens very decidedly, as is the case with the soft asphalt in the Pitch Lake, and increases in specific gravity.

### FURTHER TREATMENT WITH ACIDS.

If the treatment of the naphtha extract of the bitumen of Trinidad asphalt is continued with fuming sulphuric acid there is a very decided reaction, and if the shaking with the acid is violent and prolonged the entire amount of hydrocarbons in solution is absorbed or decomposed in much the same manner as has been observed for the naphthenes of Russian petroleum.

If treated in the cold with fuming acid but a portion is removed, and the remaining hydrocarbons have mostly a sweet odour like paraffine oil.

The examination of these oils was carried out on material obtained in another way.

### Bitumen soluble in Chloroform but insoluble in Naphtha.

After the complete extraction of the Trinidad asphalt with naphtha it was subjected to the action of hot chloroform and the bitumen obtained which is not soluble in naphtha. This is a glossy black substance, extremely friable, readily reduced to powder, which will not melt without decomposition and is very difficult to free from mineral matter. The most carefully made preparation, after the solution in chloroform had stood six months, contains 5·5 per cent. It is soluble in the softer bitumen from the naphtha extract and in heavy petroleum residuum. It has a specific gravity, ash free, of 1·1210.

Its composition is—

| | Per Cent. |
|---|---|
| Carbon | 82·00 |
| Hydrogen | 7·82 |
| Sulphur | 10·86 |
| Nitrogen | ? |
| | 100·00 |

The larger part of the sulphur is, therefore, in this part of the bitumen, and it is probably due to the sulphur, as will be seen in further investigations, that this bitumen is so hard and brittle and only soluble in chloroform and bisulphide. It has, in itself, no cementitious properties as it is too brittle and will not melt, but in the proper proportions when mixed with the softer hydrocarbons it imparts, from a technical point of view, certain valuable properties to the asphalt.

It is more or less readily changed by oxidation into the substance insoluble even in chloroform which has already been described, and in this change there is probably a loss of sulphur. It is also rendered insoluble and removed from its solution by the action of sulphuric acid or aluminium chloride.

### Further Investigations of the Hydrocarbons of Trinidad Asphalt.

The further examination of the hydrocarbons and their derivatives which make up Trinidad asphalt have been carried out with material which is more satisfactory for an initial investigation than the entire bitumen of the asphalt as it comes from the lake.

In the refining or driving off of the water from the crude pitch in preparing it for use the water in it is volatilised either over a free fire or by means of superheated steam. In either case it goes off as steam, carrying some of the lighter and more volatile portions of the asphalt over with it, which can be recovered by condensing the steam. In this way I was enabled to obtain quite a large quantity of oil from some land asphalt, refined over a fire, in Washington in 1890, and am now able to collect any desired amount from the steam-refining plant of the Barber Asphalt Paving Company, at Long Island City. The results in abstract of the examination of these oils is as follows:

### Lighter Constituents of Trinidad Land Asphalt.

The oil condensed from the steam given off by the cargo of land asphalt from the brig "Teneriffe" while it was being refined over a fire in Washington in 1891 was brown in colour and had a distinctively strong odour characteristic of sulphur compounds and unsaturated hydrocarbons. Any attempts to distill or fraction it under ordinary atmospheric pressure resulted in decomposition with the volatilisation of cracked oils of very rank odour, the decomposition of sulphur in the neck of the apparatus, and the conversion of the residue in the retort into a heavy tar or maltha, showing the ease with which the lighter portion of the bitumen of Trinidad asphalt is altered into a true asphalt.

It was found, however, that the most satisfactory way in which to obtain the lighter portions, and to fraction this and similar heavy oils was to heat them to a degree much below their boiling point in a retort, conducting a stream of carbonic acid through them at the same time. In this way the lower boiling substances came over first and, volatilising a drop a minute, the oil could be fractioned to a very high point, leaving only a small residue, and with no apparent decomposition that was revealed by any cracked or strong odour in the distillates. There was, however, a slight deposit in the neck of the retort of the purplish substance recognised by Peckham in his investigations of the California oils, as of a nature similar to pyridine and, if carelessness was not avoided in heating and supplying carbonic acid, there might be a splitting off of water and of sulphur from the action of the oxygen of the air.

Some of the land asphalt oil fractioned in this way by mere volatilisation gave the following results:—

*Original Oil from the Land Asphalt.*

| Fractions Original. | Temperature of Oil. | Per Cent. | Specific Gravity 25/25. |
|---|---|---|---|
| | ° C. | | |
| 1 | 150 | 6·0 | 0·9051 |
| 2 | 150 | 7·8 | 0·9109 |
| 3 | 150 | 11·5 | 0·9156 |
| 4 | 150 | 14·1 | 0·9223 |
| 5 | 215 | 9·4 | 0·9275 |
| 6 | 215 | 12·5 | 0·9311 |
| 7 | 215 | 13·0 | 0·9421 |
| 8 | 215 | 9·7 | 0·9513 |
| 9 | 215 | 12·9 | 0·9647 |
| Tar | .. | 4·4 | .. |
| Loss | .. | 2·4 | .. |

And another lot, fractioned at 215° C. all the way through in lots of 15 per cent., until 90 per cent. had passed, yielded—

| | | | |
|---|---|---|---|
| 1 | 215 | .. | 0·9116 |
| 2 | 215 | .. | 0·9162 |
| 3 | 215 | .. | 0·9199 |
| 4 | 215 | .. | 0·9357 |
| 5 | 215 | .. | 0·9395 |
| 6 | 215 | .. | 0·9578 |

In the same way the oil obtained from the lake asphalt was fractioned, each fraction being the same volume.

*Oil from Lake Asphalt.*

| Fraction Original. | Temperature. | Specific Gravity at 25° C. | Refractive Index. |
|---|---|---|---|
| | ° C. | | |
| 1 | 150 | 0·8883 | 1·4830 |
| 2 | 150 | 0·8933 | 1·4859 |
| 3 | 150 | 0·8991 | |
| 4 | 165 | 0·9006 | 1·4893 |
| 5 | 165 | 0·9048 | |
| 6 | 180 | 0·9077 | |
| 7 | 180 | 0·9115 | |
| 8 | 180 | 0·9127 | 1·4942* |
| 9 | 180 | 0·9138 | |
| 10 | 180 | 0·9155 | |
| 11 | 180 | 0·9177 | |
| 12 | 180 | 0·9194 | |
| 13 | 190 | 0·9200 | |
| 14 | 215 | 0·9210 | |
| 15 | 215 | 0·9225 | 1·5020 |
| 16 | 215 | 0·9233 | |
| 17 | 215 | 0·9246 | |
| 18 | 215 | 0·9259 | |
| 19 | 215 | 0·9280 | |
| 20 | 215 | 0·9301 | |
| 21 | 215 | 0·9321 | |
| 22 | 215 | 0·9322 | |
| 23 | 215 | 0·9345 | 1·5078 |
| 24 | 215 | 0·9361 | |
| 25 | 215 | 0·9379 | |

* Of mixed fractions.

Or grouped in six fractions—

| | | | |
|---|---|---|---|
| 1 | .. | 0·8883 | 1·4830 |
| 2 | .. | 0·8933 | 1·4859 |
| 3 & 4 | .. | 0·9003 | 1·4893 |
| 5—10 | .. | 0·9106 | 1·4942 |
| 11—20 | .. | 0·9224 | 1·5020 |
| 20—25 | .. | 0·9336 | 1·5078 |

All of these fractions were somewhat deep in colour, the more so as the density increased, but none of them showed any sign of solidification or the presence of paraffin when placed in a freezing mixture. The odour of all the fractions was strong, but with no sign of cracking when carefully prepared. It was distinctively asphaltic and a mixture of the smell of sulphur compounds and unsaturated hydrocarbons.

The oils are distinguished by their high boiling point, specific gravity, and refractive index and attention is at once drawn to the possibility of this being due the presence of naphthenes, as paraffin hydrocarbons of such a density would be solid at ordinary temperatures.

The fractioning of the oils from the lake and land asphalt shows also that there is a decided difference in the lower fractions of the two asphalts, the lake asphalt containing much lighter constituents than that from the land asphalt.

### Treatment of the Oils with Reagents.

Following the usual practice the oils were further investigated as to their nature by treatment with sulphuric acid of different strengths and aluminium chloride. After several successive treatments with common sulphuric acid at ordinary temperatures the land asphalt oil, which lost about 30 per cent. in the operation in the form of a heavy tarry sludge, and even then was still acted upon by fresh acid, was washed with sodic hydrate and distilled in a vacuum of about 60 mm. and gave the following fractions:—

| Fraction. | Boiling Point, 60 mm. | Specific Gravity, 15°/15°. | Iodine Absorption. | Refractive Index. |
|---|---|---|---|---|
| | ° C | | | |
| 0 | 210–220 | 0·8973 | 16·2 | 1·4858 |
| 1 | 250 | 0·9014 | 13·9 | 1·4887 |
| 2 | 260 | 0·9059 | 18·0 | 1·4910 |
| 3 | 270 | 0·9136 | 14·8 | 1·4960 |
| 4 | 280 | 0·9205 | 14·6 | 1·5000 |
| 5 | 290 | 0·9277 | 17·5 | 1·5038 |
| 6 | 300 | .. | 16·4 | 1·5072 |
| Residue | .. | .. | 18·4 | 1·4988 |
| Original oil | .. | .. | 21·4 | .. |
| Treated oil | .. | .. | 19·1 | .. |

The fractions had a slight aromatic odour much like cymol, probably due to decomposition of napthene hydrocarbons by sulphuric acid. The lower ones were light and limpid, the upper ones yellow. The results of their examination shows that, by the action of acid, the specific gravity had been reduced somewhat, but that the action of iodine was not essentially reduced, nor was it much more active on one fraction than another. The fractions, therefore, still contained unsaturated hydrocarbons, but after treatment with fuming sulphuric acid the action of bromine was very much smaller, 2·39 and 3·03 per cent. only being absorbed.

The two lower fractions of the land asphalt oil, boiling at from 210°—220° and from 220° to 250° were, as has been said, still acted upon slowly and slightly by fresh ordinary sulphuric acid. They were, therefore, treated with fuming sulphuric acid which attacked them violently, if the temperature was not reduced, with the evolution of sulphur dioxide. With several treatments at low temperatures and subsequent washing with nitric acid and soda, hydrocarbons were obtained, the first of which was fractioned and the larger portion, boiling between 214°–217° C., examined. The second fraction was examined without fractioning. The results were as follows:—

| —— | 0. | 1. | Theory, $C_nH_{2n}$. |
|---|---|---|---|
| Specific gravity 20° 20° | 0·8823 | 0·8925 | .. |
| Refractive index | 1·4790 | 1·4820 | .. |
| Carbon | 84·48 | 85·63 – 85·68 | 85·71 |
| Hydrogen | 13·68 | 14·29 14·49 | 14·29 |

A determination of the molecular weight of the first fraction by the boiling-point method gave as results, 198 and 184, which correspond to $C_{14}H_{28}$ 196 and $C_{13}H_{26}$ 182, but the molecular refraction observed from the above data for each of these substances, according to Bruhl, is 62·9 and 57·4, and from the formulas by Bruhl's values for C and H, 64·0 and 59·4, the latter one corresponding most closely with the allowance of 1·83 for the double bond so that, while the identification is not certain, the hydrocarbon is probably $C_{13}H_{26}$ which should, if it is a naphthene, have, according to Markowinkoff and Oglobin, a specific gravity of 0·8299 at 0° and a boiling point of about 200° C. There is, therefore, a discrepancy in the specific gravity and boiling point and also in the analytical figures. The two former are not explained, but the latter is undoubtedly due to errors in the combustion as the second fraction gives correct results for a $C_nH_{2n}$ hydrocarbon and no $C_nH_{2n-2}$ hydrocarbon in the first fraction could have withstood the action of strong sulphuric acid. Lack of substance prevented its repetition. It is, of course, possible that the first fraction could be contaminated by the presence of aromatic hydrocarbons produced by the action of the acid as shown by Konoalo (J. Russ. Chem. Soc. **22**, 4 and 118; this Journal, 1888, 925), but if any were found they would probably be removed by it as sulphonic acids or leave some odour. The evidence, as far as it goes, is, therefore, that the lowest boiling fractions of Trinidad asphalt, unacted on by sulphuric acid, are cyclic hydrocarbons closely allied to if not naphthenes. On account of their much greater specific gravity and higher boiling points they seem to differ from those described by Markoinko, but they evidently are of the $C_nH_{2n}$ series, have high densities, like the naphthenes, and are slowly attacked by both ordinary and fuming sulphuric acid. They are not readily acted upon by sulphur, are slowly nitrated by dilute nitric acid, 1·072, when boiled with it, and are converted by strong nitric acid into a resinous substance. Bromine acts upon them little in carbon bisulphide solution at ordinary temperatures and converts them into a tar on heating with evolution of hydrobromic acid.

There is, of course, always the possibility that these hydrocarbons do not exist as such in the asphalt, but are the results of decomposition of others by the action of acid. This will require further investigation.

### Lake Asphalt Oil.

The oil from the lake asphalt was fractioned in a similar way to that from land asphalt but after treatment only with dilute sulphuric acid, one of acid to four of water, and with mercuric chloride and acetate of soda in alcoholic solution to remove sulphur compounds. With a vacuum of 30 mm. fractions, which were bright but had some strong odour, were obtained extending from 145° C. to 210° C., but the residue left in the retort, even at this temperature and vacuum, was converted to a thick tar or maltha, showing that the unsaturated hydrocarbons were polymerised or condensed, even under these conditions, when the sulphur has been removed. The fractions were treated with ordinary and with fuming sulphuric acid and then had the following properties:—

| Fraction Boiling Point, 30 mm. | Specific Gravity. | Refractive Index. | Carbon. | Hydrogen. |
|---|---|---|---|---|
| ° ° | | | | |
| 145 – 170 | 0·8576 | 1·4650 | 86·85 | 13·31 |
| 170 – 180 | 0·8699 | 1·4721 | .. | .. |
| 180 – 190 | 0·8822 | 1·4790 | .. | .. |
| 190 – 200 | 0·8854 | 1·4795 | .. | .. |
| 200 – 210 | 0·8914 | 1·4815 | 85·98 | 13·62 |

Here we have fractions which correspond to those from land pitch oil in boiling point, specific gravity, and refractive index, considering that they were distilled in a more perfect vacuum, but they have a composition more nearly corresponding to less hydrogen than $C_nH_{2n}$ and more nearly to $C_nH_{2n-2}$ which require for $C_{12}H_{22}$ 86·75 and 13·25 hydrogen. Such a reduction in the percentage of hydrogen from that required by $C_2H_{2n}$ hydrocarbons may be readily occasioned by the presence of a small amount of an aromatic substance, and with further treatment, this might be removed. It is most probable, therefore, that these are $C_nH_{2n}$ hydrocarbons, contaminated with those of the $C_nH_{2n-6}$ series, or something similar, as it seems quite impossible that a $C_nH_{2n-2}$ hydrocarbon could have resisted the fuming acid treatment.

### Polymerisation of $C_nH_{2n}$ Hydrocarbons.

The fractions just described were examined immediately after distillation in July. In November, they were again examined and the specific gravity and refractive index were found to have distinctly increased showing spontaneous polymerisation, as has been noticed by F. Heusler as happening with other hydrocarbons.

A small portion of one of the distillates exposed to the sun on the roof for a considerable longer time, was turned quite yellow and deposited a hard sticky resin, the change under these circumstances being more active.

The readiness with which these hydrocarbons polymerise and condense at ordinary temperatures suggests the origin of hard asphalt from petroleums, composed of such hydrocarbons at ordinary temperatures and seems somewhat similar to the phenomena seen at the centre of the Trinidad and Bermudez pitch lakes, where the fresh material oozes out in a soft condition, and is slowly converted to a hard asphalt with the evolution of gas. In the case of the pure hydocarbons, the hardening must be one chiefly of polymerisation, however, as there is no opportunity for sulphur or oxygen to assist in condensation, whereas in the lakes sulphur plainly enters into the reaction as shown by the evolution of hydrogen sulphide.

### Treatment with Aluminium Chloride.

According to F. Heusler (this Journal, 1896, 581), the use of aluminium chloride for the removal of sulphur compounds and unsaturated hydrocarbons from mineral oils is a success, although this has been denied by others. The oil from Trinidad lake asphalt, treated with 10 per cent. of the chloride at 150° C. lost about 15 per cent., with evolutions at first of hydrogen sulphide and then hydrochloric acid. The remainder distilled without decomposition into the following fractions, although before treatment it could not have been distilled except in value without cracking and giving rank smelling distillates.

| Boils 760 mm. | Refractive Index. |
|---|---|
| Per Cent. | |
| 278 | 1·4745 |
| 294 | 1·4861 |
| 311 | 1·4926 |
| 329 | 1·4984 |
| 347 | 1·5073 |
| 364 | 1·5165 |
| 370 | 1·5268 |

There is not a great difference in the physical properties of these fractions from those obtained with acid, but they are more viscous, have a sweeter smell and distill without decomposition at ordinary temperatures. They have not been examined further, except as to the action on them of strong sulphuric acid, which is much less marked than in the case of the oil untreated, no tar being formed.

The result of the action of common sulphuric acid was to remove 30 per cent. of the original oil which had not been taken out by aluminium chloride. The acid was not blackened but turned a deep amber colour and deposited no tar on treatment with water. The remaining oil was light yellow in colour, had the odour of a sweet mineral oil free from unsaturated hydrocarbons.

On further treatment of this oil, while cooled with fuming sulphuric acid, but 37 per cent. of the original oil was left, 17 per cent. having been removed by the acid. If treated with an excess of fuming acid small quantities of the oil would be completely absorbed by it or destroyed with evolution of sulphurous acid.

The larger portion of the oil after the treatment with aluminium chloride and forms acid, boils at ordinary pressure between 290° and 310° C., without decomposition.

From these reactions it is inferred that the oils volatilised with steam from lake asphalte are sulphur derivatives, removed by aluminium chloride, unsaturated hydrocarbons absorbed by sulphuric acid and other unsaturated hydrocarbons removed by fuming sulphuric acid, which are similar to the naphthenes, but that no actual hydrocarbons of the paraffine series are produced by the action of the aluminium chloride on the unsaturated bodies of the original asphalt oil. The reaction is, however, such that the remaining oil can be distilled at ordinary pressure and is much more stable than in its original form. This would seem to show that the most unstable compounds of the asphalt are the sulphur derivatives.

The action of aluminium chloride on the asphalt oils will be examined more carefully in the near future.

### Substances Removed by Acids.

As has been said, the treatment of the asphalt oils with acid results in the formation of a thick acid tar or sludge, containing a large part of the sulphur derivatives of the asphalt hydrocarbons, a large portion of the polymerised or condensed unsaturated hydrocarbons, and some sulphuric acid derivatives, sulphonic acids, &c.

If this sludge is distilled with steam, a small portion of these substances is quickly volatilised and recovered as a bright yellow oil of the very characteristic odour of the fresh lake pitch, and evidently containing sulphur compounds. It contained 12·53 per cent. of sulphur, and, therefore, consisted only in small part of sulphur derivatives, and on testing with isatin and sulphuric acid it gave no indophenine reaction, so that thiophen homologues are not present. It was decomposed and converted into a thick tar on distillation at ordinary temperature, but at 30 mm. was fractioned with ease into three parts, boiling as follows :—

| Fraction No. | Boils 30 mm. | Refractive Index. | Specific Gravity. |
|---|---|---|---|
| | ° C. | | |
| 1 | 60—155 | 1·4965 | 0·9383 |
| 2 | 155—185 | 1·5065 | 0·9617 |
| 3 | 185—270 | ? | 0·9700 |

| Fraction No. | Colour. | Odour. | Per Cent. |
|---|---|---|---|
| 1 | Lemon..... | Sulphur aromatic | 30 |
| 2 | Amber..... | Nearly sweet .... | 50 |
| 3 | Dark amber | Unsaturated..... | 20 |

The greater part of this oil is, therefore, either aromatic or one forming compounds with sulphuric acid, easily decomposed in the same way by steam. The very high density of all the fractions would seem to point to the presence of aromatic substances. They will be further investigated.

### The Acid Tar.

After the distillation with steam, the residue of the sludge was a sticky tar, which, after washing with water, was extracted with naphtha, and yielded a deep brown oil, not easily volatile with steam, quite thin, which had less strong odour of asphalt than that volatilised with steam. It contained sulphur, 6·67, and nitrogen, 0·09 per cent.; therefore, a small amount of sulphur compounds in comparison with the oil volatilised with steam. After extraction with 88° naphtha, the remainder of the sludge was a brittle black solid, which, after again washing with water till free from acid, was extracted with acetone. A brown solid was obtained, which had the following composition :—

| | |
|---|---|
| Sulphur............................ | 11·74 |
| Nitrogen .......................... | 0·45 |

This extraction was followed by one with carbon bisulphide, which gave a glossy blue-black pitch, having the following position:—

| | |
|---|---|
| Sulphur | 11·64 |
| Nitrogen | 0·12 |

The residue, a dull black powder, contained—

| | |
|---|---|
| Sulphur | 10·51 |
| Nitrogen | 0·45 |

It is, therefore, evident that the compounds in the sludge, while partly substances decomposable by superheated steam and volatile with it, are largely polymerisation and condensation products of the unsaturated hydrocarbons and sulphur compounds of the oil. Experiments with the bitumen soluble in carbon disulphide, but not in naphtha, have shown also that this readily soluble bitumen is entirely converted, quickly, into a brown powder quite insoluble in the ordinary solvents, by shaking with acid. It is, therefore, plain that there is a tendency in the unsaturated hydrocarbon and sulphur compounds which compose the bitumen of Trinidad asphalt to be converted very easily by condensation or polymerisation, under certain conditions, into substances which are represented ordinarily by the organic matter found in our analysis, which is insoluble in ordinary solvents for bitumen.

### Effect of the Acid Treatment on Heavy Petroleum Oils.

The examination of the asphalt oils by successive treatment with sulphuric acid of different strengths, leads to results which show that apparently there are no saturated hydrocarbons in asphalt. To confirm these results and determine the suitability of this method for arriving at such a conclusion, the heavy residuum oil from Pennsylvania petroleum, which is carefully prepared with steam without cracking, especially for use in the paving industry, was examined in the same way. This oil has the following properties:—

| | |
|---|---|
| Specific gravity | 0·920 – 0·930 |
| Flash point, closed | 425°—400° F. |
| Volatile from 20 grms. in 7 hours at 400° F. | 2·0 —5·0 per cent. |

It cannot be distilled without cracking under ordinary pressure, as it boils above 300° C., but it can be *in vacuô* to a certain point, or be volatilised slowly after the manner employed with the asphalt oils. Fractions prepared in this way gave the following results:—

| Fraction. | Volatilized at | Specific gravity, 20° C./20° C. | Refractive Index. | Paraffine Scale. |
|---|---|---|---|---|
| | ° C. | | | ° |
| 1 | 245 | 0·8857 | 1·4930 | At 15 |
| 2 | 260 | 0·8889 | 1·4935 | 20 |
| 3 | 275 | 0·8916 | .. | 30 |
| 4 | 275 | 0·8920 | .. | 39 |
| 5 | 275 | 0·8953 | .. | 35 |

All these fractions were filled with paraffin scale below 60° F., and had a density and refractive index corresponding to the lower fractions of asphalt oil. They show that such a residuum oil from Pennsylvania is satisfactory for comparing the asphalt hydrocarbons with those of petroleum of equal density.

The original residuum oil was separated by naphtha of 0·64 density into two portions, 96·5 per cent. being soluble, and 3·5 per cent. insoluble. The naphtha soluble hydrocarbons were treated with ordinary sulphuric acid and with fuming acid. The results of the treatment were the separation of a brown insoluble powder by naphtha, the recovery of an oil, little changed in appearance from the naphtha solution, while the oils obtained by the treatment of the naphtha solution with acids were respectively a bright amber coloured vaseline, with some paraffine scale and a soft paraffine like substance semi solid at ordinary temperatures.

To determine the nature of the different fractions and the changes which had resulted in the oil, an examination was made with the following results:—

| Fraction. | Naphtha insoluble. | Naphtha soluble. | After $H_2SO_4$ Treatment. | After $H_2SO_4 + SO_3$ Treatment. |
|---|---|---|---|---|
| Per cent. of original | 3·5 | 96·5 | 82·9 | 52·6 |
| Specific gravity | .. | 0·9126 | 0·9010 | 0·8733 |
| Refractive index | .. | 1·515 | 1·507 | 1·482 |
| Carbon | 91·45 | 87·96 | 87·76 | 84·74 |
| Hydrogen | 7·34 | 12·01 | 12·13 | 15·33 |
| Sulphur | 0·71 | 0·24 | 0·24 | 0·21 |
| Nitrogen | 1·09 | 0·99 | .. | .. |

The brown powder insoluble in naphtha is discovered to be very poor in hydrogen, and also contain the nitrogen of the oil and most of the sulphur. The composition of the oil soluble in naphtha before the acid treatment shows that it contains a considerable amount of unsaturated hydrocarbons since the hydrogen amounts to but 12 per cent. Oxygen is not present in determinable amount.

About 12·6 per cent. of these unsaturated hydrocarbons are removed by ordinary sulphuric acid, with but little change in the composition of the residue, although there is a decrease in the specific gravity and refractive index. It is fair to conclude that the substances removed had a composition like the average composition of the original oil, but a greater density and refractive index.

Treatment with fuming sulphuric acid, the results of the analyses of the residue show, has removed all the unsaturated hydrocarbons to the amount of 30 per cent. of the original residuum and left oils or paraffines, having the average composition of the saturated series, with a much further reduction in density and refractive index. Calculation of the density of the hydrocarbons removed, from the percentage of less, shows that it is 0·936, as would be expected for less saturated substances.

The result of the examination is to prove that the paraffine hydrocarbons, when they exist in a heavy mineral oil, can be isolated by treatment of the oil in naphtha solution with fuming sulphuric acid and subsequent analysis. It is, therefore, justifiable to assume that the asphalt oils contain no paraffines.

### California Maltha.

The heavy petroleum occurring at Bakersfield, California, which, on evaporation, becomes a thick asphaltic maltha, was examined in the same way as the Trinidad oil by volatilization into fractions at a temperature below its boiling point. The resulting oils had the following specific gravity at 20° C. and refractive index for the D ray at the same temperature:—

| Fraction. | Specific Gravity, 20° C. | Refractive Index. |
|---|---|---|
| 1 | 0·8354 | 1·4574 |
| 2 | 0·8471 | 1·4630 |
| 3 | 0·8582 | 1·4690 |
| 4 | 0·8674 | 1·4750 |
| 5 | 0·8776 | 1·4815 |
| 6 | 0·8888 | 1·4876 |
| 7 | 0·9001 | 1·4930 |
| 8 | 0·9080 | 1·4993 |
| 9 | 0·9174 | 1·5041 |
| 10 | 0·9236 | 1·5084 |
| 11 | 0·9319 | 1·5134 |
| 12 | 0·9366 | 1·5185 |

The first fraction was examined by treating it with common sulphuric acid when it lost 8 per cent. and had a specific gravity of ·8590, showing the removal of substances of little density. It had a strong aromatic odour resembling cymol.

It was then treated with fuming sulphuric acid when only 28 per cent. of the original oil was left, and on continued treatment all the oil could be absorbed. Before this occurred a few cubic centimeters were washed, dried, and burned, with the following result, as compared to the composition of the original maltha:—

| —— | Original. | After Acid. |
|---|---|---|
| Carbon | 84·31 | 86·86 |
| Hydrogen | 12·41 | 13·10 |
| Sulphur | 1·40 | .. |

The original material and the final product of the acid treatment are, therefore, composed of unsaturated hydrocarbons, but the latter may not be the same that occur in the original maltha as it smells strongly aromatic which would point to its being a decomposition product.

The fractions are all thin oils not solidifying at low temperatures and evidently contain no paraffine or paraffine hydrocarbon. They are less resistent to reagents than the oils of Trinidad asphalt and are probably less stable.

The acid which absorbed this oil so freely was worked up for sulphonic acids, but only a sticky amorphous product obtained such as is found to be the case with the naphthenes.

### The Bermudez Asphalt Deposit.

From the mouth of the Orinoco, the north-eastern coast of Venezuela, which faces Trinidad, is low and consists of of vast mangrove swamps, through which run deep tidal estuaries. That portion forming part of the State of Bermudez extends inland for many miles. It lies on the opposite side of the Gulf of Paria from Trinidad. About thirty miles in an air line from the coast the asphalt deposit, known as the Bermudez Pitch Lake, is found at the point where a northern range of foot hills comes down to the swamp. The Guanace river, a branch of the San Juan, one of the large cañons or estuaries of this region, at about 65 miles, in its winding course, from its mouth, runs within three miles of the deposit, but it is five or six miles to a suitable wharfage site. On the other hand, towards the north a road runs to the hills and to the village of Guaryquen. These are the means of communication with the deposit. The so-called lake is situated between the edge of the swamp and the foot hills in what might be termed a savanna. It is an irregular shaped surface with a width of about a mile and a half from north to south and about a mile east and west. Its area is a little more than 900 acres, and it is covered with vegetation, high rank grass and shrubs, one to eight feet high, with groves of large moriche palms, called morichales. One sees no dark expanse of pitch on aproaching it as at the Trinidad pitch lake, and except at certain points where soft pitch is welling up, nothing of the kind can be found. The level of the surface of the deposit does not vary more than two feet and is largely the same as that of the surrounding swamps. In the rainy season, it is mostly flooded and at all times very wet, so that any excavation will fill up with water. These conditions make it difficult to get about upon it or to excavate pitch easily.

It is readily seen that this deposit is a very different one from that in the pitch lake of Trinidad. It seems to be in fact merely an overflow of soft pitch from several springs over this large expense of savanna and one which has not the depth or uniformity of that at Trinidad.

Being on a level with the mangrove swamps and with foot hills on its other side any large amount of asphalt could hardly be held in position here, as in the old crater in Trinidad, but would burst out into the swamp and be lost, and, as far as borings have been made, they seem to indicate but a small depth anywhere as compared with that of the Trinidad lake.

At different points there is at most a depth of 7 ft. of material while the deepest part of the soft maltha is only 9 ft. and the average of pitch below the soil and coke only 4 ft. At points there is not more than 2 ft. of pitch, and in the morichales or palm groves it is often 5 ft. below the surface. At several points scattered over the surface are areas of soft pitch or pitch that is just exuding from springs. The largest area is about 7 acres in extent and of irregular shape. It has little or no vegetation upon it, and from the constant evolution of fresh pitch is raised several feet above the level of the rest of the deposit. This soft asphalt has become hardened at the edges but when exposed to the sun is too soft to walk upon. The material is of the nature of a maltha and it is evidently the source of all the asphalt in the lake, from these exudations the pitch having spread in every direction, so that no great depth of pitch is found even at this point.

A careful examination of the surroundings shows that in one respect there is a resemblance between the point of evolution of the soft pitch at the Bermudez and at the Trinidad lakes. Gas is given off in considerable quantities at both places, and in cases consists partly, at least of hydrogen sulphide. At the Bermudez lake I was unable to determine whether it was accompanied by carbonic dioxide, but the odour of hydrogen sulphide was strong.

The consistency of the soft pitch at the centre of the Bermudez lake is much thinner than that of the Trinidad lake. It will run like a heavy tar and does not evolve gas in the same rapid way or harden as quickly after collection. It therefore does not retain the gas which is generated in it, nor does the deposit as a whole do so to the same extent as the Trinidad pitch. Where, however, the surface of the soft pitch has toughened by exposure to the sun and air and where gas is given off beneath it, it is often raised in dome like protuberances, the beehives which were spoken of by visitors to the Trinidad lake. These have a thin wall of pitch and are filled with gas which readily burns, and have been seen 2 ft. or more in height and 18 in. in diameter. They are of course found only near the soft spots.

Although the pitch at the Bermudez lake is too soft to entangle and hold permanently the gas which is given off, where the pitch of medium consistency is covered with water it does not escape so readily, and thus often raises in the pools of water a mushroom-like growth of pitch by the reduction of the gravity of the mass from the included gases. These mushrooms correspond completely, except in size, with those described by Manross as existing at the Trinidad lake when he visited it. It seems, therefore, that we have to-day several of the phenomena represented at the Venezuelan lake which the hand of man has destroyed at Trinidad.

There is, however, no evidence of the same simultaneous boiling up of water with the fresh soft pitch that has been determined at the Trinidad lake, but that there is none at all is not certain as at the time I visited the locality heavy rains were falling which prevented the detection of a small amount. It seems, however, improbable as the soft pitch contains little or no water and the trace found in the samples collected are probably derived from rain.

### Hardening of the Main Mass of Pitch.

The soft pitch after it exudes at the centre of the Bermudez lake undoubtedly hardens slowly on exposure but the condition of the surface of the main mass, which is very hard and rough, and of the harder borders of the soft spots is due to other causes also.

The edges of the areas of soft asphalt are covered here and there with masses of glance pitch and with black and brittle cinders or coke, and which seem to have been produced from the maltha by fire. This is evidently the case since the rank growth of grass which is very dry in the dry season is particularly adapted for a rapid and intense combustion. Such fires have been even recently started intentionally and accidentally and to them are due the condition of the present surface of the deposit and the character of much of the pitch.

The general surface of the lake is very irregular and hard. There are many very narrow and irregular channels or depressions from a few inches to four feet deep, filled with water, and not being easily distinguished one often falls into them. At the foot of the growth of grass and shrubs are ridges of pitch mingled with soil and decayed vegetation, which have been plainly coked and hardened by fires of the nature which has been mentioned. When this hardened material which forms only a crust is removed, asphalt of a kind suitable for paving is found. The crust is from a foot and a half to two feet in depth and very firm, while the asphalt underneath would not begin to sustain the weight which that of the Trinidad pitch lake does easily. There are breaks in the crust here and there through which soft pitch exudes as has been described.

It appears, therefore, that the Bermudez deposit owes its existence to the exudation of a large quantity of soft maltha, which is still going on and which has spread over a great extent; that this has hardened spontaneously in the sun, and has also, by the action of fire, been converted over almost the entire surface into a cokey crust of some depth beneath which the best material lies, and that here and there are scattered masses of glance pitch produced in a similar way from less violent action of heat. There is no evidence of a general movement and mingling of the mass of this deposit in any way that would produce a uniformity of composition as seen in the Trinidad pitch lake, although there is a certain amount of gas evolved at the soft spots where maltha exudes and some gas cavities are found in the general mass of the pitch beneath the crust.

As the asphalt below the crust of the deposit is the only portion of value for paving, the question has arisen as to its uniformity. In order to determine this a collection of samples was made at various points on the lake beneath the crust and they have been submitted to analysis.

All the samples represent pitch considered suitable for paving except Nos. 2, 7, 14, 19, and 34, at the bottom of the table No. 2 being glance pitch. These are excluded from our review of the results.

### Analyses of Samples of Bermudez Asphalt.

| Sample No. | Crude Material. | | Refined Material. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Loss 4 days, 212° F. | Loss 7 hours, 400° F. | Loss 7 hours, 400° F. | Softens ° F. | Flows ° F. | Mineral Matter. | Organic, not Bitumen. | Bitumen by Difference. | Soluble in 88° Naphtha. | Total Bitumen thus soluble. | Degree of Softness. | Specific Gravity, 77°/77° F. |
| | Per Cent. | Per Cent. | Per Cent. | | | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | | Per Cent. |
| 1 | 11·68 | 10·65 | 10·05 | 158 | 170 | 1·03 | 2·79 | 96·18 | 72·61 | 75·49 | 2 | 1·054 |
| 3 | 17·70 | 11·60 | 14·25 | 158 | 170 | 0·81 | 2·95 | 96·24 | 72·13 | 74·95 | 3 | .. |
| 4 | 34·05 | 7·37 | 9·40 | 160 | 170 | 1·14 | 1·90 | 96·96 | 67·10 | 69·20 | 0 | .. |
| 5 | 15·15 | 10·38 | 11·06 | 160 | 170 | 1·50 | 1·08 | 97·42 | 72·56 | 74·48 | 2 | .. |
| 6 | 18·68 | 8·02 | 7·16 | 170 | 180 | 1·67 | 6·45 | 91·88 | 65·62 | 71·42 | 4 | 1·070 |
| 8 | 14·85 | 12·36 | 14·86 | 160 | 170 | 2·78 | 0·62 | 96·60 | 71·31 | 73·82 | 1 | .. |
| 9 | 14·74 | 12·20 | 13·47 | 160 | 170 | 1·50 | 0·90 | 97·60 | 70·89 | 72·63 | 1 | .. |
| 10 | 19·82 | 12·08 | 12·82 | 160 | 170 | 0·88 | 1·09 | 98·03 | 71·17 | 72·60 | 3 | .. |
| 11 | 11·89 | 10·29 | 9·57 | 165 | 180 | 1·20 | 1·89 | 96·91 | 69·53 | 71·74 | 4 | .. |
| 12 | 17·63 | 10·08 | 10·88 | 165 | 188 | 1·55 | 2·56 | 95·89 | 69·80 | 72·79 | 2 | .. |
| 13 | 15·87 | 10·73 | 11·64 | 165 | 180 | 1·50 | 3·26 | 95·24 | 70·80 | 74·34 | 2 | 1·025 |
| 15 | 15·80 | 11·66 | 9·76 | 170 | 180 | 1·45 | 0·78 | 97·77 | 69·95 | 71·54 | 2 | .. |
| 16 | 20·05 | 10·96 | 12·15 | 154 | 170 | 1·57 | 2·40 | 96·03 | 69·41 | 72·29 | 3 | .. |
| 17 | 14·80 | 9·71 | 11·70 | 150 | 160 | 1·30 | 2·27 | 96·43 | 71·40 | 74·04 | 3 | .. |
| 18 | 16·72 | 13·60 | 16·05 | 150 | 160 | 1·35 | 1·40 | 97·25 | 73·05 | 75·12 | 1 | 1·007 |
| 20 | 20·40 | 9·85 | 10·88 | 160 | 168 | 1·30 | 1·45 | 97·25 | 68·64 | 70·58 | 2 | .. |
| 21 | 12·60 | 11·65 | 12·24 | 150 | 160 | 1·86 | 1·70 | 96·44 | 69·30 | 71·86 | 3 | .. |
| 22 | 14·00 | 12·75 | 15·11 | 160 | 168 | 1·55 | 2·24 | 96·25 | 69·40 | 72·13 | 3 | 1·005 |
| 23 | 25·15 | 9·75 | 10·18 | 160 | 170 | 1·31 | 2·64 | 96·05 | 67·88 | 70·66 | 2 | .. |
| 24 | 24·27 | 9·71 | 10·22 | 160 | 168 | 1·80 | 2·25 | 95·95 | 68·62 | 71·52 | 2 | .. |
| 25 | 11·78 | 9·60 | 11·69 | 160 | 168 | 1·93 | 1·68 | 96·39 | 70·68 | 73·32 | 2 | .. |
| 26 | 19·02 | 11·60 | 14·00 | 160 | 168 | 2·70 | 2·20 | 95·10 | 71·14 | 74·80 | 3 | 1·038 |
| 27 | 16·17 | 9·70 | 13·65 | 150 | 160 | 1·72 | 1·84 | 96·44 | 72·50 | 75·17 | 3 | .. |
| 28 | 46·20 | 4·72 | 5·81 | 170 | 180 | 1·43 | 2·38 | 96·19 | 65·20 | 67·78 | 0 | .. |
| 29 | 19·99 | 11·55 | 11·95 | 155 | 168 | 1·68 | 1·24 | 97·08 | 70·40 | 72·52 | 3 | .. |
| 30 | 14·20 | 10·70 | 15·10 | .. | 135 | 3·65 | 1·60 | 94·75 | 72·54 | 76·55 | 1 | .. |
| 31 | 14·20 | 8·69 | 13·16 | 150 | 160 | 3·26 | 0·96 | 95·78 | 70·82 | 73·94 | 3 | .. |
| 32 | 22·55 | 9·11 | 7·35 | 160 | 175 | 3·33 | 6·02 | 90·65 | 63·40 | 69·94 | 4 | 1·060 |
| 33 | 18·12 | 10·15 | 13·89 | 140 | 150 | 3·60 | 1·22 | 95·18 | 70·98 | 71·57 | 3 | 1·075 |
| 35 | 10·72 | 10·75 | 8·80 | 160 | 170 | 1·71 | 1·70 | 96·59 | 70·06 | 72·53 | 2 | .. |
| 36 | 33·10 | 7·74 | 10·20 | 160 | 172 | 0·50 | 0·98 | 98·52 | 69·98 | 71·03 | 2 | 1·020 |
| 2 | 0·67 | 1·36 | .. | 188 | 201 | 1·24 | 2·92 | 95·84 | 58·52 | 60·00 | Pitch | .. |
| 7 | 16·57 | 12·25 | .. | 150 | 160 | 3·90 | 0·94 | 95·16 | 73·98 | 77·74 | 1 | .. |
| 14 | 20·01 | 11·79 | .. | 150 | 160 | 1·15 | 1·36 | 97·47 | 72·10 | 73·97 | 2 | .. |
| 19 | 19·73 | 11·89 | .. | 150 | 160 | 1·13 | 0·94 | 97·93 | 72·58 | 74·11 | 3 | .. |
| 34 | 15·80 | 14·00 | .. | 120 | 140 | 3·85 | 1·30 | 94·85 | 73·80 | 78·22 | 1 | .. |

### Extremes of Determinations in the Analyses of Bermudez Asphalt.

*Crude Material.*

| —— | Highest. | Lowest. |
|---|---|---|
| Loss four days, 212° F. .......... | 46·20 | 10·72 |
| Loss seven hours, 400° F. ........ | 13·60 | 4·72 |

*Dried Material.*

| | | |
|---|---|---|
| Loss at 400° F., seven hours ..... | 16·05 | 5·81 |
| Softens, ° F. .................... | 170° | 140° |
| Flows, ° F. ...................... | 188° | 135° |
| Mineral matter ................... | 3·65 | 0·50 |
| Organic not soluble .............. | 6·45 | 0·62 |
| Bitumen .......................... | 98·52 | 90·65 |
| Bitumen soluble naphtha .......... | 73·05 | 63·40 |
| Per cent. of total thus soluble... | 76·55 | 67·78 |
| Specific gravity ................. | 1·075 | 1·005 |

### Methods of Analyses.

Necessity has compelled the adoption of somewhat different methods of analysis with the Bermudez samples from those employed in the case of the Trinidad material since the sticky nature of this asphalt prevents its being freed from water by powdering and exposure to the air. It was only possible, therefore, to dry the samples by heating them to such a point as to soften the bitumen and remove the water, this point being kept as low as possible, but undoubtedly with the loss of gas and hydrocarbons. The dried samples were then used far analysis. In addition, the amount of water, gas, and volatile oil lost by the crude material on maintaining it for a certain length of time at 212° and 400° F. was determined, as well as the loss suffered by the dried material at the latter temperature. The softening and flowing point and the degree of softness at ordinary temperatures were also determined, 1 softest, 2 soft, 3 medium, and 4 hard, as well as the specific gravity of a few samples. The determinations of the loss at high temperatures are not as accurate as the others, but with such wide variations as were found, is of interest and value.

From the preceding results of analyses we see that the Bermudez asphalt is quite a different material from that of the Trinidad lake. It is practically free from mineral matter; in fact the softest material from the soft spot at the centre before it was in any way contaminated with extraneous matter, has no more than 0·02 per cent. It has, as collected, varying amounts of water and the loss on heating to 100° C. for sometime may reach as high as 46 per cent. Here again, however, it appears from an examination of the freshly collected softest material that the water is adventitious and the loss largely light oils, as this soft material contains but a trace of water. The amount of organic matter insoluble is, probably, also adventitious, as this is not found in the soft pitch, but it may in part be derived from changes in the bitumen itself from age and exposure. The portion of the total bitumen soluble in naphtha is large but not constant in the specimens from the different parts of the lake, but of the fresh material nearly the entire amount is soluble.

A mere glance at the results then shows that there is a great lack of uniformity in the condition and character of the material found scattered over the surface of the Bermudez deposit, and it appears that this bed of asphalt is one that was originally, and is now being, derived from exudations from maltha springs, scattered over the area which the deposit covers; that the original thick oil or maltha was a pure bitumen free from mineral matter and water which has become altered by changes within itself and by external causes, until today it exists in the form found in the so-called lake. There is here, as in the Trinidad pitch lake, a present source of supply which is adding every year to the main body of pitch.

### Character of the Bitumen of Bermudez Asphalt.

An investigation of the nature of the bitumen of Bermudez asphalt has been partially carried out in the same way as with the Trinidad material, and has furnished some results of interest.

The softest and freshest asphalt from the point where it exudes has the following composition:—

| — | I. | II. |
|---|---|---|
| Carbon | 82·88 | .. |
| Hydrogen | 10·79 | .. |
| Sulphur | 5·87 | 5·37 |
| Nitrogen | 0·75 | .. |
| | 100·29 | .. |

The striking features of this bitumen is the very high percentage of sulphur, nearly as great as that in Trinidad bitumen, and the notable percentage of nitrogen, while in other respects it shows only evidences of being composed largely of unsaturated hydrocarbons. On comparing the original bitumen with that found in the general supply of the lake, as used for pavements, an interesting change is seen. This bitumen extracted from the melted and refined pitch by chloroform, has the following composition as derived from the combustion of two different preparations.

*Entire Bitumen Bermudez Paving Asphalt.*

| — | I. | II. |
|---|---|---|
| Carbon | .. | .. |
| Hydrogen | .. | .. |
| Sulphur | 3·93 | 3·41 |
| Nitrogen | 0·75 | 0·82 |

Here a remarkable alteration is seen. The hardening of the pitch or very likely the process of melting and refining has removed a very considerable part of the sulphur, nearly 2 per cent. The sulphur must have been given off as hydrogen sulphide, and in its evolution condensation has taken place, and a consequent hardening of the bitumen.

The nitrogen remains unchanged.

Sulphur, therefore, in this case, as in that of Trinidad asphalt, seems to play an important part in the hardening of the bitumen. It is also found in larger amount on that portion of the Bermudez bitumen which is insoluble in naphtha but soluble in chloroform as with Trinidad asphalt, the bitumen soluble only in chloroform having the following composition:—

| | Per Cent. |
|---|---|
| Carbon | 87·19 |
| Hydrogen | 8·47 |
| Sulphur | 4·83 |
| Nitrogen | .. |
| | 100·49 |

### Sulphur in Asphalt, and its Influence.

The interest, which the presence of so much sulphur in the bitumen of Trinidad and Bermudez asphalt, and especially in the least soluble and harder portions aroused, led me to examine a large number of asphalts and malthas of varied origin, which were available in the collection of the Barber Asphalt Paving Company, with a view of determining how general the presence of a large percentage of sulphur is in bitumens, which are hard and of high melting point.

At the same time complete ultimate analyses were made of these bitumens, in order to show that oxygen is not a constituent of that portion of an asphalt soluble in chloroform or bisulphide of carbon, to determine how generally nitrogen is one, and what differences there may be in the saturation of the hydrocarbons.

The determination of sulphur was made on the pure bitumens after the method, described by Mabery, of combustion in a current of oxygen, and absorption of the sulphur dioxide formed in a standard solution of alkali. In several instances, especially Trinidad pure bitumen, the results were checked by determinations made by oxidation with nitric acid.

The analyses for carbon and hydrogen were made with all the precautions which a long experience with this kind of work has shown to be necessary. They were determined by combustion with manganese oxide $Mn_2O_3$ with a layer of lead chromate and a copper coil on account of the presence of sulphur and nitrogen. The copper coil and the substance in a platinum boat were introduced into the tube after the tube and its contents had been raised to a red heat in current of oxygen and allowed to cool. The combustion was carried on very slowly in a stream of oxygen, and the absorption of the water and carbonic acid carefully provided for and protected. Nitrogen was determined readily by the Kjeldahl method.

The results are shown on next page.

It appears in a general way from the preceding results that all the glance pitches, or brittle asphalts, Nos. 1, 3, 4, and 5, contain from 9·76 to 8·28 per cent. of sulphur, the hard asphalts, not flowing at ordinary temperatures, from 6·47 to 3·93 per cent., while the malthas or soft asphalts and heavy petroleums do not contain more than 2·29 per cent. There are, however, certain exceptions. The soft Bermudez and Montana asphalts should, from their consistency, be classed with the malthas, but are placed by themselves, because they are rapidly transformed under certain conditions to their harder forms, which are included under the asphalts, probably because of the high percentage of sulphur they contain.

The Uvalde Company bitumen is also an exception, in that it is of only medium hardness, although having 9·60 per cent of sulphur. This bitumen is, however, peculiar and differs from the asphalts in some of its other properties as well.

Those natural bitumens also, which are usually classed by themselves, albertite, gilsonite, grahamite, elaterite, and ozokerite, are distinguished by a low percentage of sulphur and a high percentage of carbon, with the exception of elaterite which appears, therefore, to be more nearly allied to the asphalts. They are, probably, therefore, of different origin from the asphalts, and as an illustration of such a difference, the origin and method of formation of the artificial asphalts included in the table will serve.

The so-called Pittsburg flux is produced by heating heavy petroleum oil, such as Pennsylvania residuum with sulphur. Hydrogen sulphide is given off, and the oil is condensed to a peculiar tough and sticky substance, but slightly affected

ULTIMATE COMPOSITION OF THE BITUMEN OF ASPHALTS, MALTHAS, &c.

| —— | Consistency. | In Extracted Bitumen. | | | | |
|---|---|---|---|---|---|---|
| | | Sulphur. | Carbon. | Hydrogen. | Nitrogen. | Total. |
| *Hard Asphalt.* | | | | | | |
| Nevada | Pitch | 9·76 | 79·58 | 9·31 | 1·30 | 99·95 |
| Texas, Uvalde Company | Hard | 9·60 | 80·32 | 10·09 | 0·28 | 100·29 |
| Italy, Ancona | Pitch | 9·39 | 79·56 | 8·80 | 1·40 | 99·15 |
| Egypt | „ | 8·52 | 80·87 | 10·42 | 0·19 | 100·00 |
| Cuba, Bejucal | „ | 8·28 | 82·98 | 9·44 | 0·09 | 100·79 |
| California, Waldorf | Hard | 6·48 | 82·77 | 10·62 | 0·35 | 100·21 |
| „ La Patera | „ | 6·23 | 83·30 | 9·88 | 0·70 | 100·11 |
| Trinidad Lake | „ | 6·16 | 82·33 | 10·69 | 0·81 | 99·99 |
| Tobago | Pitch | 6·16 | 83·37 | 10·54 | 0·50 | 100·57 |
| Turkey | Hard | 6·14 | 83·98 | 9·19 | 0·09 | 99·40 |
| Montana, hardened | „ | 5·31 | .. | .. | .. | .. |
| California, Santa Maria | Cheesy | 5·69 | 82·79 | 11·58 | 0·48 | 100·54 |
| Trinidad, land | Hard | 4·98 | 83·65 | 10·76 | 0·45 | 99·94 |
| France, South | „ | 4·78 | 82·07 | 11·58 | 1·56 | 99·99 |
| Venezuela, Bermudez, hard | Medium | 3·93 | .. | .. | 0·75 | .. |
| Sicily, rock | „ | 3·69 | 84·95 | 11·34 | 0·33 | 100·31 |
| Kentucky | „ | 3·39 | 84·16 | 11·52 | 1·58 | 69·65 |
| Texas, Rio Grando | „ | 3·28 | .. | .. | .. | .. |
| *Malthas rapidly hardening.* | | | | | | |
| Venezuela, Bermudez, Maltha | .. | 5·87 | .. | .. | .. | .. |
| Montana, Maltha | .. | 3·92 | .. | .. | .. | .. |
| *Malthas.* | | | | | | |
| Venezuela, Pedernales | Soft | 2·29 | .. | .. | .. | .. |
| Ohio, Residuum | „ | 2·26 | .. | .. | .. | .. |
| Trinidad, Guayaguayare | „ | 2·00 | .. | .. | .. | .. |
| Indian Territory, Chicakasaw | „ | 1·98 | 85·65 | 12·37 | .. | 100·00 |
| Trinidad, Union, Maltha | „ | 1·75 | .. | .. | .. | .. |
| Mexico | „ | 1·48 | .. | .. | .. | .. |
| Indian Territory, Ardmore | „ | 1·47 | 87·40 | 11·05 | 0·65 | 100·57 |
| California, Asphalto | „ | 1·40 | 84·31 | 12·41 | 1·35 | 99·47 |
| „ Alcatraz | „ | 1·32 | 85·72 | 11·83 | 1·21 | 100·08 |
| Texas, St. Joe | „ | 1·13 | 87·27 | 11·79 | 0·23 | 100·42 |
| Pennsylvania, residuum | „ | 0·60 | 87·44 | 12·50 | .. | .. |
| *Gilsonite, Elaterite, Albertite, Ozokerite.* | | | | | | |
| Elaterite, Utah | Brittle | 5·83 | 80·00 | 12·23 | 1·78 | .. |
| Gilsonite, Utah | „ | 1·79 | 89·28 | 8·66 | 0·79 | 100·52 |
| Albertite, Nova Scotia | „ | 1·20 | 85·53 | 13·21 | 0·42 | 100·33 |
| Oxokerite, soluble in alcohol | Tough | 0·00 | 85·25 | 15·09 | 0·00 | 100·34 |
| *Artificial Asphalts.* | | | | | | |
| Pittsburg flux | Tough | 4·10 | 83·05 | 10·58 | 2·38 | 99·84 |
| Sludge asphalt | Brittle | 4·87 | 87·17 | 7·94 | 0·00 | 99·98 |
| Byerlite, hard | „ | .. | 89·88 | 10·12 | .. | 100·00 |

by temperature changes, and only melting at very high temperature, which has been suggested as a substitute for asphalt. Here the formation of the artificial asphalt is due to the condensation of the unsaturated hydrocarbons by the action of sulphur, and while much is evolved, several per cent. are left in the product. This substance, being formed largely from the saturated paraffin hydrocarbons, in consequence, quite different from the true asphalts in many of its properties. It will not pull into strings in melting and is very short.

The sludge asphalt made from the sludge tar of the kerosene refiners is much more like natural asphalt as it pulls out to strings; like the latter melts easily, and has the general physical properties of ordinary asphalts. In this case these properties are due to the fact that the artificial product is derived from the unsaturated hydrocarbons of the petroleums, rather than the paraffins, as with Pittsburg flux, and to the presence of sulphur in the reaction which takes place, nearly 5 per cent. being left in the substance.

In the hard Byerlite an artificial asphalt is found, which is of still different origin, being produced by the action of oxygen on the heavy oils of petroleum. In this way a substance is obtained in which sulphur is absent, and has also taken no part in the formation. It seems to bear the same relation to the other artificial asphalts that gilsonite does to glance pitch of asphaltic origin, and has many similar physical properties.

The results of our examinations, therefore, seem sufficient to show that sulphur plays an important part in the hardening of asphalts, although like the artificial asphalts, some natural bitumens occur which have become hardened in another way, and, perhaps, by oxygen. In connection with the discussion of the nature and origin of natural asphalt these facts seem to be of considerable interest.

## OXYGEN IN ASPHALTS.

According to Dana's Mineralogy, "Asphaltum or mineral pitch is a mixture of different hydrocarbons, part of which are oxygenated"; and it is stated that "the fluid kinds change into the solid by the loss of a vaporizable portion on exposure, and also by a process of oxidation, which consists first in a loss of hydrogen, and finally in the oxidation of a portion of the mass." Many other writers on the subject also express the same views. For the purpose of determining how far these statements were to be relied upon, since my earlier investigations of the bitumens of Trinidad, Bermudez and some other asphalts had not shown that they contained oxygen, I have had complete ultimate analyses made of most of the pure bitumens, previously examined for sulphur, and the results have been given in the same table. It is evident that among this large number of malthas and asphalts there are none, the soluble bitumen of which contains oxygen to an appreciable extent, although they all contain sulphur and nitrogen. It is, however, easy to explain how the earlier analysts reported oxygen as a constituent of many asphalts. As shown by several recent investigators, the complete combustion and determination of carbon and hydrogen in the higher hydrocarbons is an extremely difficult thing, the tendency being to the formation of acetylene and unsaturated hydrocarbons, instead of carbonic acid and water, so that there is a deficiency in the amount of the latter found in an analysis, unless the greatest care is used. This has been commonly and naturally stated as oxygen. At the same time most analyses ignore the presence of sulphur and nitrogen, so that the amount of these elements is added to the stated percentage of oxygen. It is not surprising, therefore, that oxygenated hydrocarbons are frequently spoken of as occurring in asphalt, and it is equally plain that none exists in the bitumens of which they are composed. With the amount of sulphur present in most of them this is not surprising, as this element easily substitutes oxygen in most of the hydrocarbon derivatives.

Oxygen is present in asphalts only in those which contain organic matter insoluble in chloroform, as has been shown in the examination of the material in Trinidad asphalt, which is not bituminous and not soluble. Here the result of oxydation of the hydrocarbon derivatives has been such that it can be no longer classed as bitumen being insoluble and merely a dry powder. It is not impossible that oxygen may have acted on some hydrocarbons under certain conditions, but in that case the result has been the splitting off of water, the oxidation of the hydrocarbons to a condition in which they are no longer soluble, and to be considered a part of the bitumen; or by the substitution of oxygen by sulphur, which would easily take place in the presence of this element, and which has been shown to occur in Trinidad asphalt with the formation of the organic matter, not bitumen, which contains oxygen.

Where oxygen has acted upon natural hydrocarbons in the same manner as sulphur, with a resulting condensation, as may be the case with gilsonite and similar substances, it has not remained as a constituent of the bitumen, but has been removed as water. Oxygen, therefore, while it may have had a part in the production of hard natural bitumens, as sulphur seems undoubtedly to have done, has not, like sulphur, remained as one of the constituents of these substances.

### ACTION OF ACIDS ON ASPHALTS QUANTITATIVELY DETERMINED.

In the use of sulphuric acid of different strengths as a reagent for the treatment of asphalt oils, the action was such that it seemed of interest to determine quantitatively, as far as possible, what this action would be upon the entire bitumen. These results, it seemed, would prove of relative interest at least, and probably of technical value. Of course, an asphalt cannot be directly treated with acid successfully. To do this satisfactorily the bitumen is disolved in a solvent, such as naphtha, which is not strongly affected by the acid. Naphtha was selected for the purpose because, although the entire bitumen of most asphalts is not soluble in it, the part that is not soluble may be neglected, as it would be entirely removed by the action of acid. It seemed, therefore, only necessary to study the action of the acid on the naphtha solution. This has been done with various asphalts, and with acids of various strengths, in the following manner:—

A weighed amount—2 grms.—of any asphalt was treated with boiling 88 naphtha until extracted, and then diluted to 250 c.c. It was then shaken twice for exactly five minutes, at ordinary temperatures, with two portions of the sulphuric acid. After this treatment, the naphtha solution with its remaining bitumen was washed once with water, then with alkali, and finally with water, the naphtha solution being then evaporated in a weighed platinum dish dried for 10 minutes and 100° C., and the bitumen unacted upon by acid determined. Following are the results, including some obtained with a similar treatment of heavy petroleum oils from Pennsylvania, Ohio, and Russia for comparison:—

*Per Cent. of Naphtha-soluble Bitumen in Asphalt and Heavy Oils unacted upon by Acid.*

| Name. | Total Bitumen. | Naphtha-soluble Bitumen. | Percentage of Total Bitumen thus soluble.* | Percentage of Naphtha-soluble Bitumen unacted on by | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | 25 per cent. NaOH. | 3 to 1 $H_2SO_4$. | 2 to 1 $H_2SO_4$. | 1 to 1 $H_2SO_4$. | Conc. $H_2SO_4$. | Fuming $H_2SO_4$ + $SO_3$. |
| California consolidated.... | 66·9 | 47·78 | 71·10 | 81·8 | 71·8 | 67·1 | 64·4 | 54·4 | 4·0 |
| Bermudez lake........... | 96·5 | 67·27 | 69·70 | 80·8 | 89·7 | 80·9 | 78·8 | 38·9 | 19·4 |
| Trinidad „ ........... | 57·1 | 34·55 | 60·50 | 90·1 | 89·8 | 87·0 | 79·3 | 38·9 | 19·1 |
| Alcatraz maltha (1894).... | 97·9 | 85·19 | 87·00 | 100·0 | 93·7 | 88·4 | 87·0 | 64·2 | 35·7 |
| Penn. Residuum.......... | 100·0 | 95·68 | 95·71 | 97·4 | 95·5 | 95·6 | 95·5 | 89·5 | 53·3 |
| Ohio „ .......... | 100·0 | 97·05 | 97·10 | 99·5 | 94·9 | 93·4 | 93·6 | 86·4 | 62·6 |
| Russian „ .......... | 100·0 | 100·00 | 100·00 | 100·0 | 93·6 | 92·7 | 92·2 | 79·3 | 68·8 |

or, calculated on another basis,—

*Per Cent. of Total Bitumen removed by Reagents.*

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Californian consolidated... | .. | .. | .. | .. | 48·7 | 52·1 | 54·0 | 61·2 | 97·1 |
| Bermudez lake ....... ... | .. | .. | .. | .. | 13·5 | 13·6 | 45·1 | 72·8 | 86·7 |
| Trinidad „ ........... | .. | .. | .. | .. | 15·7 | 17·3 | 52·1 | 76·5 | 78·6 |
| Alcatraz maltha .......... | .. | .. | .. | .. | 17·7 | 23·1 | 24·3 | 44·2 | 69·6 |
| Penn. Residuum.......... | .. | .. | .. | .. | 8·5 | 8·5 | 8·6 | 14·4 | 49·0 |
| Ohio „ .......... | .. | .. | .. | .. | 7·9 | 9·1 | 9·2 | 16·2 | 39·2 |
| Russian „ .......... | .. | .. | .. | .. | 6·4 | 7·3 | 7·8 | 20·7 | 31·2 |

* More thorough extraction would have increased these percentages in some cases.

It is, of course, found that the heavy petroleum oils from Pennsylvania, Ohio, and Russia are the least affected, as they are largely made up of paraffin hydrocarbons and naphthenes not readily acted upon by acid in dilute naphtha solutions. Among the asphalts the malthas are least attacked by common acid, showing that they lie between the heavy petroleum oils and the asphalts, while the harder the asphalt, the smaller is the per cent. of bitumen unacted upon by acids, these corresponding relatively to the amount soluble in naphtha.

### EFFECT OF HEAT ON MINERAL OILS.

In a recent article (Municipal Engineering, Aug. 1897) I have shown that there is a difference in the way different asphalts, malthas, and heavy petroleum oils are affected by being maintained at a high temperature for a number of hours. While all of them volatilise something when heated to the conventional temperatures of the asphalt industry—325° and 400° F. for seven hours—there is a very considerable difference in the appearance of the residues after heating.

Petroleum residuum of the kind best suited for the asphalt-paving industry, and made especially for it, may be heated for seven hours at 400° F. and lose but 2 to 5 per cent. of volatile oil, while the residue will still continue to flow at ordinary temperatures. Other petroleum residuums may lose a much higher percentage of oil up to 15 per cent. and leave a residue, which will not flow owing to the amount of scale paraffin it contains; but in neither case is the resulting residue solid and brittle. Many natural heavy tarry oils known as malthas or soft asphalts will, however, not only lose a large amount of volatile oil when heated to 325° or 400° F., up to 20 or 30 per cent., but they will leave behind a residue which is a brittle glance pitch, showing the very different nature of the hydrocarbon of which it is composed and those making up the residuum of ordinary petroleum. Malthas of a medium character are also found, so that it is a very useful means of determining the character of any heavy mineral oil to examine it in this way.

It appears that there is a very decided difference in the character of the bitumens found in various asphalts, malthas, and heavy petroleum oils, which is shown in several ways by the preceding methods of treatment. The amount of total bitumen which is soluble in 88° naphtha is, in the first place, indicative of how far the action of condensation and polymerisation has gone on, and of how large a part of the bitumen has been converted into the hard and brittle form. For example, in the heavy petroleams and malthas the larger portion of the bitumen is soluble in naphtha, while in the asphalts this may be reduced to. 60 per cent. or less, and in the glance pitches to 40. It is possible, therefore, to gain considerable assistance in judging of the nature of a new asphalt by such a determination, or at least, as to some of its properties of technical interest. In another way the determination of the amount of the hydrocarbons soluble in naphtha, which are unacted upon by acid, help to show the nature of the substances making up this part of the bitumen. And again, the stability of the hydrocarbons is shown by their power of resisting high temperatures with little loss and without change in their physical properties, and especially their consistency.

Perhaps none of the tests which have been given possess any actual scientific merit, but they at least enable us, when they are combined, to compare different asphalts and to judge of their technical value. It must also be remembered that the results have no absolute value, and can be used only as a relative means of comparison, since the solvents and conditions may vary from time to time. In making examinations of this kind, therefore, it is desirable that a well-known asphalt, maltha, or oil should be subjected to a parallel analysis, alongside of the material under investigation.

### Conclusions as to the Nature and Origin of Asphalt.

The natural bitumen, which is known as asphalt, is composed, as far as we have been able to learn, of unsaturated hydrocarbons and their sulphur derivatives with a small amount of nitrogenous constituents. Asphalt may, therefore, be defined as any hard bitumen composed of unsaturated hydrocarbons and their derivatives, which melts on the application of heat to a viscous liquid; while a maltha or soft asphalt may be defined as a soft bitumen which, on heating, leaves an asphalt, or becomes by natural causes converted into one. The line between the two classes cannot be sharply drawn.

Asphalts are distinguished by the large amount of sulphur they contain, and it is to its presence that many of the important characteristics and perhaps, in part, the origin of this form of bitumen is due. The soft asphalts or malthas contain much less sulphur than the harder ones, or, if the former are rich in sulphur, they are then in a transition stage and will eventually become hard. But a small portion of the constituents of a hard asphalt are volatile even *in vacuo*, but they can be separated by solvents into an oily portion, which is soft, or softens readily when heated, and a harder portion, which does not melt by itself without decomposition, and is a brittle, solid, but soluble in the oily or softer portion. The harder and least soluble portion always contains the larger part of the sulphur. It seems, therefore, that sulphur is the effectual hardening agent of natural asphalts, in the same way that it is of artificial asphalts which are produced by heating a soft natural bitumen with sulphur.

The portion of asphalt soluble only in chloroform or carbon bisulphide is entirely absorbed by sulphuric acid, showing that it contains no paraffins or saturated hydrocarbons.

That portion which is soluble in naphtha can be, to a great extent, volatilised *in vacuo*. The lighter constituents are found to be largely unsaturated hydrocarbons of at least two classes—those absorbed or converted into tars by sulphuric acid, and those but slightly acted upon by it, together with sulphur compounds partly precipitated by mercuric chloride and partly recoverable from their combination with sulphuric acid by steam.

The hydrocarbons absorbed by sulphuric acid and converted into tars by it may belong to several series, less saturated than the $C_nH_{2n}$, and it is probable that some of them or their derivatives are aromatic or cyclic compounds. Nothing further is known of them at present. Those not easily acted upon by acid are of very high density and refractive index for their boiling point and molecular weight, and correspond in no way with any saturated or open chain hydrocarbons. They are evidently cyclic hydrocarbons similar to the naphthenes, but having an even greater density than these, as far as they are known, for the same molecular weight. The amount of these comparatively stable hydrocarbons unacted upon readily by sulphuric acid varies in different asphalts from comparatively little to 30 per cent. or more. In the malthas there is a still larger proportion. The greater part of an asphalt bitumen is, therefore, of unsaturated and unstable bodies liable to constant change, while even the most stable constituents, the cyclic hydrocarbons, are slowly polymerised on standing.

### Origin of Asphalt.

Asphalt is in the process of formation today. It plainly does not originate as such, but is a secondary product, resulting from the transformation of lighter forms of bitumen, malthas, or even thinner oils, into harder bitumen by condensation and polymerization, a reaction in which sulphur seems to take an important part.

Asphalt is probably derived from unsaturated hydrocarbons, although the condensation of paraffin hydrocarbons by sulphur or of a mixture of saturated and unsaturated hydrocarbons in the same way would yield unsaturated hydrocarbons as products, but would require a high temperature. Polymerization would probably take place more readily in the unsaturated series.

No high temperatures seem necessary for the change once initiated, as it is seen going on today in the Trinidad pitch lake and in Venezuela. Gas is commonly evolved and is largely hydrogen sulphide, the natural result of the condensation of hydrocarbons by sulphur. Carbonic acid accompanies it which would seem to point to a reaction between hydrocarbons and inorganic sulphates and the latter as the source of the sulphur in the bitumen of asphalts, but this is only theory based on the presence of sulphates in the water emulsified with Trinidad pitch, while there is quite as much hydrogen sulphide given off by the Bermudez asphalt which contains no water or sulphates as it originates.

Whether its origin is in sulphates or inorganic sulphides or whether, as suggests itself, it is derived from the same substances, either animal, vegetable, or inorganic, from which the hydrocarbons themselves are formed, it is plainly an important factor in the production of asphalt and especially the harder ones.

It seems justifiable, therefore, to suggest that where mineral oils, composed of unsaturated hydrocarbons, originate under such circumstances as to be subjected to conditions favorable to condensation and polymerization asphalt will be formed, not necessarily immediately, but in the course of time.

For the primary origin of asphalt one must, of course, go back to that of petroleum. This subject has lately been discussed at length, and both experiments and arguments brought forward in favor of the animal or vegetable origin of petroleum or its inorganic derivatives. Recent scientific journals are filled with the literature of the question, so that it will not be necessary to summarize the reasoning here, but merely to add such suggestions, derived from the results of our investigations, as may be utilized in favor of one or the other theories.

The presence of nitrogen in all the asphalts examined may be considered, by those advocating the animal or vegetable origin of petroleum, as confirming their views, but it seems possible also to account for it from an inorganic source.

The theory of the general origin of all mineral bitumen in a base of unsaturated hydrocarbons, suggested by Hensler, seems to be supported by the unsaturated nature of the constituents of the asphalts and malthas; but on the other hand, the formation of saturated hydrocarbons from the unsaturated oils of Trinidad asphalt by the action of aluminium chloride, has not yet been confirmed.

The thermal origin of the bitumen of Trinidad lake asphalt is strongly suggested by the presence of a thermal water, containing borates and iodides, in an emulsion of definite composition with bitumen and mineral matter, while the transportation of bitumen from place to place by means of springs, is illustrated by the mineral spring which rises at the centre of the Trinidad lake with the freshly issuing pitch, and to the collection of bitumen in pockets, even under the sea where springs break out. In no case, however, have any signs of more than normal temperatures been observed in any of these springs. Steam, therefore, if an element in the movement of bitumen, must be associated with it at such a point as to indicate a thermal origin of the bitumen itself.

Finally, the theory of Ochsenius that the formation of petroleum is to be attributed to the action of strong salt solutions on organic matter, might also be considered as strengthened by the presence of such a strong brine accompanying the emulsion of soft asphalt in the Trinidad lake.

These suggestions are given for what they are worth as slight contributions to the knowledge of a subject which is still so far from being understood. My own investigations have been confined simply to a study of the nature and origin of asphalt, the importance of sulphur in its genesis and the effect which it has upon its character.

I am indebted to Mr. J. H. Byerley and Mr. R. H. Parker, my assistants, for the careful laboratory work they have done under my direction.

### Discussion.

Mr. H. C. Woltereck asked if it had ever been observed that sulphuretted hydrogen was originally separated as thio-carbonic acid. Several years ago, in Europe, in two thermal springs which were strongly sulphurous, it was observed that sulphuretted hydrogen was not present originally as such but as thio-carbonic acid.

He thought the question was originally raised concerning a spring in Hungary, and that it was finally proved that in water taken from a depth of 50 feet no sulphuretted hydrogen nor free carbonic acid was present, while it could always be proved to be present in surface water. That would seem to him to indicate its thermal nature.

Mr. R. C. Woodcock asked whether Mr. Richardson found much mineral matter dissolved in the chloroform extract, and whether there was much sulphide of iron in the Trinidad asphalt.

Dr. Schweitzer said that the author had omitted to mention the brilliant theory of Prof. Moissan about petroleum and its origin. Prof. Moissan attributed the origin of petroleum to metallic carbides, because carbides produce different hydrocarbons on contact with water; for example, carbide of aluminium produced methane; carbide of calcium, acetylene, carbide of uranium, a mixture of hydrogen, methane, and ethylene. The formation of carbides would be due to volcanic action. As evidence of this theory Moissan stated that petroleum was found at Riom, in France, at a depth of 1,200 metres, and that asphalt was found in the rock at Limagne, together with basaltic tufa.

Mr. H. C. Woltereck asked if lima oil did not form an exception as to the sulphur. He said he thought the content of Ohio oil was between 5 and 6 per cent.

The Author, in reply to Mr. Woltereck, stated that he was not familiar with the facts mentioned, but that he had had no similar experience with the mineral waters of the pitch lake, and that the only thing he could say in answer to his question regarding hydrogen sulphide in the water was that if crude Trinidad asphalt were heated, after it had ceased to evolve gas by itself, it continued to give off, at a temperature just below that of boiling water, very large volumes of carbonic acid, and then that ceased and large volumes of hydrogen sulphide were given off. As to Lima oil the heaviest portion had only 2 per cent. of sulphur.

As to the amount of mineral matter dissolved in the chloroform extract, the specific gravity of the chloroform being so much higher than that of the naphtha, it was useless to try to separate the inorganic matter in a centrifugal machine, so that the only way it could be extracted was by sedimentation. He had had chloroform extract standing for six months, and at the end of that time it had 5 per cent. mineral matter still left in it. When it was analysed it was found that it could not be in solution, because of the silicate of alumina it contained. The chloroform extract contained so much mineral matter that it seemed to him extraordinary. He supposed it was in such a very fine state of subdivision that no ordinary filter paper or thick asbestos felt would retain it. As to the amount of sulphide of iron in the Trinidad asphalt, he had not been able to determine whether it was present there as sulphide of iron or ferrous-sulphate. In the water it was ferrous-sulphate. In reply to Professor Peckham's suggestion there was iron pyrites in the neighbourhood of the lakes; he did not know of any deposit of iron pyrites there. Moissan's theory was really Mendeléef's, and the one to which he himself was most inclined.

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* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENT.

*Filter Presses and similar Filtering Apparatus* [*Water, Chemical Liquors, Sugar Juices, &c.*], *Impts. in and relating to.* J. Williamson, 65, West Regent Street, Glasgow. Eng. Pat. 21,934, Oct. 3, 1896.

To prevent clogging or choking of the interstices of the filter-cloth, &c., the apparatus is provided with movable brushes or the like, adapted for cleaning the surfaces of the filtering media, the admission of the unfiltered liquid being stopped and the filtering chambers emptied or partially emptied during the cleaning operation. The brushes or brush-wheels may be mounted on a central shaft, by which they are rotated or oscillated in contact with the surfaces of the filtering media, or they may be mounted on a reciprocating shaft and reciprocated in contact with the said surfaces. Means are also described for intermittently and automatically bringing the brushes into operation whilst suspending filtration.—R. A.

## II.—FUEL, GAS, AND LIGHT.

*Gas Lighting: Arranging Illumination.* Illustr. Zeits. für Blechindustrie, 26, [39], 1350.

To ensure uniform illumination throughout a room, the illuminants should be arranged so as to throw an equal degree of light on each unit of floor space. A convenient unit of light per square metre of floor space is 3 candles, measured at the source of light; and in such case the height of the burner above the floor should be $\frac{2}{5}\left(\frac{a+b}{2}\right)$; *a* being the length and *b* the breadth of the apartment. If the ratio between these values exceed 3:2, then the area should be parcelled into squares and each illuminated separately.

These data suffice for flat and round burners of the ordinary kinds, but are inapplicable for the modern type of burner. Cohn finds that a 10 metre-candle-power light is required to give the same degree of illumination for reading and writing as supplied by daylight. In the case of shaded lights, it must be remembered that opal globes cause a loss of 40—60 per cent. and frosted glass a loss of 36 per cent. of light. Averaging the former loss at 50 per cent., it follows that 15 metre-candles are needed per sq. metre of floor space; and, since the Auer light gives a light of 55 candles when the gas pressure is 34 mm., one such light will suffice for $3\frac{2}{3}$ sq. metres.—C. S.

*A New Illuminant.* U.S. Cons. Reps., Dec. 1897, 626.

Ernest Salzenberg, director of the gasworks of the city of Crefeld, has invented an improvement in incandescent gas-burners, which relates to the production of incandescent gaslight, based upon the discovery that, when the pressure of the gas is considerably increased upon the incandescence body, the said body emits a golden-yellow light, very agreeable to the eye, displaying objects in their natural colours. The gas is supplied to the burner at a pressure of about $3\frac{1}{2}$ atmospheres, the burner, to withstand this high pressure, being of special construction. A single incandescent jet of the ordinary size can emit a light of much more than 1,000 candle-power. The light is of such intensity that a person is enabled to read the finest print at a distance of 100 to 150 feet. The inventor claims that the cost of his incandescent light of 1,500 candle-power is only $4\frac{1}{2}$ cents per hour, while that of the ordinary electric light of 400 candle-power is (in Germany) 14 cents per hour. In the apparatus constructed by Salzenberg, a hydraulic pressure of 3·5 atmospheres, and even more, may be forced through the improved Auer burner. The invention is, however, only applicable where waterworks exist.

*Carbides, New Methods for Preparing, by the Action of Calcium Carbide on Oxides.* H. Moissau. Comptes Rend. 1897, 125, [22], 839—844.

Calcium carbide acts energetically on oxides which are fused. Metals which, like lead, tin, and bismuth, do not form carbides, are liberated, and may remain free, or else combine with the substances present, according to the conditions of the experiments.

The carbides of aluminium, manganese, chromium, molybdenum, tungsten, titanium, and silicon, were prepared by the new method. They had been previously prepared by the direct union of the elements in the electric arc, or by the reduction of the oxides by carbon.—N. H. J. M.

*Sodium Carbide, Properties of.* C. Matignon. Comptes Rend. 1897, 125, [24], 1033—1035. (See this Journal, 1897, 634.)

Sodium carbide is a white powder, insoluble in all solvents, of density 1·575, and not sensitive to a blow or to friction. Dry air and oxygen are without action upon it at the ordinary temperature, but on gentle heating it burns with formation of sodium carbonate. Hydrogen is without action on the carbide; chlorine attacks it, with incandescence and formation of carbon; bromine reacts violently; iodine gives rise to $C_2I_4$. Phosphorus between 50°—180° C. produces sodium phosphide. Sodium carbide, thrown on water, reacts explosively, and carbon is deposited; with small quantities, acetylene is obtained in theoretical amount. With hydrochloric acid gas, the following reaction arises:—

$$C_2Na_2 + 2HCl = 2NaCl + C_2 + H_2,$$

but if the carbide be suspended in ether, acetylene is the product. Carbon and sulphur dioxides induce incandescence, carbon being separated. At 150°, sulphuretted hydrogen attacks sodium carbide—

$$C_2Na_2 + 2H_2S = 2NaSH + C_2H_2.$$

Oxidising agents form, with the carbide, explosive mixtures. Certain chlorides and iodides, mercury and lead sulphates, react violently when mixed with the carbide in a mortar; explosions often result.

The alcoholic iodides and bromides attack sodium carbide at about 180° C.; symmetrical acetylenes, $RC \vdots CR$, are produced in general; but with isobutyl iodide, which is unstable at 180°, isobutylene is formed. Monobrombenzene is without action at 180°. Primary and secondary alcohols produce acetylene and sodium alcoholates. The aldehydes, acetones, and esters react violently, with production of acetylene. The acid anhydrides and chlorides also react on warming.—A. C. W.

*Acetylene, Action of Nitric Oxide on.* S. Zinno. Luce e Calore, 1897, 1, 118; Chem. Zeit. Rep. 1897, 21, 298.

A tube, with two right-angled bends, connects the top of a flask having two lateral tubulures with a two-necked Woulff's bottle containing cold water. At one tubulure dry nitric oxide is admitted, and as soon as air is expelled by it, dry acetylene is admitted at the other tubulure, and the mixed gases are passed through the water for a long time. In addition to nitric and nitrous acids, the water will then be found to contain cyanic acid, arising thus:—

$$C_2H_2 + 2NO = 2CNOH.$$

On igniting the gas escaping from the Woulff's bottle at the orifice of a drawn-out tube, if acetylene be in excess, the flame will be of much higher illuminating power even than that of acetylene alone; but if nitric oxide be in excess, it will be red, and will lead to a most violent explosion of the mixture in the bottle.—J. A. B.

*Acetylene.* V. B. Lewes. J. Gas Lighting, 1897, Nov. 30, 1177—1179.

The manufacture of calcium carbide is first referred to, and it is stated, as the result of the author's personal experience, that for a consumption of one electrical horse-power per hour, there can be produced from 0·3 lb. to a little over 0·5 lb. of carbide, the variation depending upon the working of the furnace and the way in which the mixture of lime and powdered carbon (generally coke) has been prepared. A good working mixture is 100 parts by weight of lime and 68 parts of carbon. The ingot of carbide which is formed in the furnace, consists mainly of pure crystalline calcium carbide, but it is nearly always surrounded by a crust in which the carbide contains a certain proportion of imperfectly converted constituents, and which therefore gives a lower yield of acetylene than the carbide itself; in breaking up and packing the carbide, the poor crust is, as far as possible, picked out and rejected. A certain amount of loss thus occurs during the breaking and packing; in one case, whilst the furnace return was 0·504 lb. per kilowatt-hour, the amount after breaking, sorting, and packing was only 0·406 lb. 1 ton of carbide is obtained from 1·79 ton of the mixture of lime and coke, and one electrical horse-power obtained from water-power costs about 4*l.* in England. In practice, 1 lb. of carbide gives an average of 5 cb. ft. of acetylene gas. One of the chief essentials in the manufacture of carbide is the purity of the raw materials. Good metallurgical coke should be used, in which the phosphorus and sulphur present amount only to the smallest traces, since, if the carbide manufactured be impure, and the acetylene gas generated therefrom contain any considerable proportion of phosphuretted hydrogen, siliciuretted hydrogen, or sulphuretted hydrogen, there ensues a certain amount of danger of spontaneous ignition, whilst the products of combustion from the impurities are injurious to health, and destructive to furniture, draperies, &c. in a room. The author's personal experience is, that those specimens of carbide which contain sufficient impurities to render them dangerous, are of foreign manufacture.

In all of the innumerable patents for the generation of acetylene gas, one of the three following methods has been adopted for bringing together the calcium carbide and the water:—(1.) Water is allowed to drip upon the carbide, in which case the carbide is in excess until near the end of the reaction. (2.) The level of the water is altered so as to allow it to come in contact with the carbide, which is held in a chamber or cage; in this case, a considerable volume of water might be in contact with the carbide acted upon. (3.) The carbide is dropped into a large volume of water, in which case the water is always in excess. In the first method intense heat is developed, which tends to polymerise some of the acetylene into liquid products, and which also, by decomposing all the impurities present in the carbide, renders the gas extremely impure; generators made on this principle must have elaborate arrangements for repressing the heat evolved by the action of the water on the carbide. The second method has some of the disadvantages of the first, though not to so great an extent. not only is there no undue rise of temperature, but the gas evolved has to bubble through a certain amount of water, which condenses and washes from it the impurities. The generator should also be so constructed that under no possible conditions could the pressure rise above a few inches of water, whilst the pressure in the pipes leading from the apparatus should never reach above 2 or 3 inches. With these precautions in regard to the selection of a generator, and with pure carbide and a low pressure in the service-pipes, acetylene is stated to be as safe for domestic lighting as coal-gas.

The author considers that, taking into consideration the serious accidents which have occurred, the placing of liquefied acetylene and high-pressure acetylene gas under the Explosives Act is perfectly justifiable. With regard to the use of pure acetylene as an illuminating agent, one of the chief difficulties has been the large quantity of smoke which is produced. This difficulty, however, it is stated, has been overcome by the use of a form of burner (made by Naphey, New York and Walmsley, Chicago) in which two small jets of acetylene are brought out in such a way that, as they leave the burner, they are surrounded by a tube of air, which prevents the acetylene coming in contact with the burner-tube. These two jets then meeting, splay one another out into a flat flame; and, as far as experience has at present gone, these burners show no tendency to become choked or smoke. For the purposes of railway carriage and tram car lighting, diluted acetylene appears to be more valuable than acetylene itself, more especially in view of the fact that acetylene gas under a certain pressure has been placed under the Explosives Act. When acetylene gas is diluted, its explosive power rapidly decreases, and a mixture of 30 per cent. of acetylene with the diluting gas is quite safe under the pressure which exists in railway carriage cylinders. The author, after a large number of experiments, in the course of which he examined the efficiency of hydrogen, carbon monoxide, water-gas, air, carbon dioxide, and nitrogen, came to the conclusion that the best diluent was a mixture of 30 per cent. of methane mixed with hydrogen, carbon monoxide, or water-gas; this diluting gas, when mixed with 10 per cent. of acetylene, gave a 20-candle power gas, capable of being burnt in ordinary gas-fittings, distributed through the mains and pipes, and behaving in every way like coal-gas. It was also found that with a mixture containing enough acetylene to make a brilliant illuminant, suitable for compression in cylinders for carriage and tram lighting, a temperature which would melt the metal cylinder would be required to cause explosion. The author considers that for country houses and small towns and villages, the diluted acetylene gas, which could be manufactured and sold at a fair profit for 3*s.* 6*d.* per 1,000 cubic feet, presents considerable advantages over coal-gas and other illuminants.—A. S.

*Fire-Damp Explosions: Ammonium Salts as Preventatives, in Blasting Operations.* Oesterr. Zeits. Berg- u. Hüttenw. 1897, **45**, 568.

*See under XXIV., page 77.*

## PATENTS.

*Air and Water Vapour, Impts. in or relating to the Production of a Mixture of, especially applicable for Gas Producers.* L. Mond, Northwich. Eng. Pat. 28,588, Dec. 14, 1896.

This mixture is especially intended for use in the manufacture of ammonia from coal and other nitrogenous fuel, with the simultaneous formation of producer gas, as described in several earlier patents by the same inventor. (This Journal, 1896, 643 and 529; also 1886, 235.) The air may be heated by means of hot gases in a continuous or intermittent superheater, and then brought into intimate contact with hot water in a scrubber. Or the water may be heated under pressure to above 100° C., and then brought into intimate contact with air, by preference partially charged with water vapour, in an apparatus under ordinary pressure. When it is desired to utilise the heat of waste gases at a distance, the heat may be transferred to water or other liquid, which is then carried the distance required in pipes, and the heat afterwards transferred therefrom in a heat transmitter, either to other water, which is then brought in contact with air; or to air, which is brought into contact with heated water.—R. S.

*Coal-Gas, Enriching, Impts. in the Method of and Apparatus for.* R. H. Whiteley and W. Ellis, Chesterfield. Eng. Pat. 16,890, July 16, 1897.

This invention relates to a simple method of enriching coal-gas by means of tar, the object being to extract from the tar as large a quantity of the light hydrocarbons, which it contains, as possible, and to add those light hydrocarbons to the gas before it passes to the gas-holder. The invention consists in breaking up the tar into spray, or otherwise dividing it in such a manner as to make it present a larger surface to the gas passing over it. The patentees prefer to deal with the tar while it is still hot, but it may also be heated after it has been collected and cooled. According to one method, they interpose, in the foul-gas main leading from the hydraulic main, one or more mechanical agitators, rotated at a speed sufficient to break up the tar, which is still liquid and hot, into fine spray, or to cause the tar to expose a large surface for the evaporation of its light hydrocarbons. The gas is thereby thoroughly brought into contact with every particle of tar before it reaches the condensers, and it thus carries away a large proportion of light hydrocarbons.—D. B.

*Petroleum Burners, Impts. in.* M. Habermann, Baumschulenweg, Berlin, Germany. Eng. Pat. 23,058, Oct. 7, 1897.

A device for providing a petroleum lamp with a small light, suitable for a night light, when the circular main wick is turned down, in which the two wicks are automatically and alternately raised and lowered, whereby also each is lighted from the other as it is raised.—R. S.

*Liquid Hydrocarbons, Impts. in or relating to the Burning of.* T. Clarkson, Sutton, Surrey, and H. C. Capel, Dalston Lane, Middlesex. Eng. Pat. 1794, Jan. 22, 1897.

The hydrocarbon is passed through a vaporiser, then through a jet nozzle having a regulator needle, to a flared tube arranged in a mixing chamber, in the top of which is a horizontal gauze or perforated metal partition, and above that the burner. The tube leading to the vaporiser is in connection with a closed air chamber to prevent back pressure. The mixing chamber has a siphon at its lowest part for carrying off condensed oil. The burner consists of a plate having a series of parallel troughs alternating with a series of slits, through which the vapour rises. In another form, also shown, the burner grid is formed hollow, and the vaporiser is combined with it. The oil-feeding tube may pass through a steam generator heated by the burner. To start the burner, two blow-pipe flames are employed—one to heat the vaporiser, the other the mixing chamber. The blow-pipe jets are directed on to asbestos wicks in boxes, fed with liquid hydrocarbon from an overhead divided tank, the division supplying the blow-pipe burner to heat the mixing chamber being much the smaller, whereby it stops action sooner than the other.—R. S.

*Gas Producers, Impts. in.* W. Climie, Lennoxtown, Stirling, and A. Watson, jun., Bellshill, Lanark. Eng. Pat. 6132, March 9, 1897.

Within a vertical cylindrical iron or steel chamber is arranged a second similar chamber, the space between the two forming a complete water jacket. Through the inner chamber is a central vertical tube, forming a communication between the water jacket at the top and the bottom. The inner chamber is fed with fuel through feeding hoppers, and on its base is a fire grate having a number of tubular openings into it, admitting air for combustion and steam from jets, whilst through the tubes, or some of them, there are arranged vertically projecting pokers, by the action of which clinkers are broken up, or their formation is prevented. The producer gas passes away from the top. To gain access to the grates, doors having special closing devices are employed.—R. S.

*Coke Ovens, Impts. in.* G. Hilgenstock, Dahlhausen-on-the-Ruhr, Germany. Eng. Pat. 22,120, Sept. 27, 1897.

Coke ovens of the beehive form, especially intended for making long-grained coke with the recovery of the by-products, are constructed with a broad, flat base and with little height. In the brick foundations there are arranged flues which pass under the bottoms of the ovens, and into these flues gaseous fuel is led through nozzles which are in communication with a duct below, each duct being situated in a passage, whereby both ducts and nozzles are easily accessible.—R. S.

*Graphite [from Acetylene], Process for the Production of.* F. J. Bergmann, Neheim-on-the-Ruhr, Germany. Eng. Pat. 23,957, Oct. 18, 1897.

Acetylene is heated at about 150° under about 5 atmospheres pressure with hydrogen peroxide or other suitable oxidising agent. Or a metallic carbide is similarly heated with aqueous solution of the peroxide, and the hydrate of the metal, which is in this case mixed with the separated carbon, is dissolved out with acid.—J. A. B.

*Carbides, Means [Rotatable Electrodes, &c.] for Manufacturing; Impts. in.* J. W. Kenevel, Brooklyn; C. A. Spofford, New York; and J. H. Mead, Brooklyn—all of U.S.A. Eng. Pat. 19,512, Aug. 24, 1897.

"Consists broadly in passing the prepared material ['substantially two parts of lime to one of carbon or coke'], consisting of carbon and base—such, for example, as lime—between movable or rotatable electrodes, which are suitably connected to a source of electricity to secure the necessarily high temperature." Ten claims. Claim 1. "Means for manufacturing carbides, consisting of a suitable casing within which are positioned a pair of rotatable electrodes [carbon sleeve, discs or pins on metal core or sleeve, &c.] arranged in a substantially horizontal plane, mechanism for rotating the same, and means for connecting the electrodes with a source of electricity, in combination with means for conducting the prepared material between the electrodes, &c."
—J. C. R.

*Gas, Impts. in the Manufacture of.* W. L. Wise, London. From A. Piatti and Co., Rome. Eng. Pat. 2129, Jan. 26, 1897.

A mixture of colophony and calcium oxide is taken, preferably one of 10 parts of the former, 80 of the latter, 5 of magnesium carbonate, and 5 of calcium carbide. This is subjected to the action of water, or acidulated water, and the gas evolved is used for lighting or heating, with or without carburetting.—J. A. B.

*Acetylene Gas, Impts. in the Manufacture of, and in Apparatus therefor.* A. J. de P. Hargreaves, Paris. Eng. Pat. 27,194, Nov. 30, 1896.

Carbide falls from a suitable hopper on to a revolving screw in a horizontal tube, and is conveyed through a controlling device, consisting of fixed and revolving segments of discs, to a perforated hopper mounted on a vertical revolving axis above a plate on the same axis within a generating vessel containing water. The plate is slightly below the level of the water, and has a gauze rim to retain the carbide until disintegrated. The gas generated passes through the perforated hopper to a space above the generator, and thence through four purifiers for the removal of ammonia, sulphuretted hydrogen, phosphuretted hydrogen, and moisture. Lime deposit is removed from the generator by a screw conveyor similar to the feeding screw. The generator has a water jacket, a safety valve, and a cistern which automatically replenishes the generator with water.
—J. A. B.

*Acetylene Gas Generator, Purifier, and Container; Impts. in and connected with.* T. R. Fowler, Seacombe, county of Chester, formerly of Liverpool. Eng. Pat. 28,206, Dec. 10, 1896.

Detachable cages containing carbide are hung on a rod which screws through a nut in the crown of a bell floating in the annular space around a tank of water into which the cages are depressed for the generation of gas. A purifying chamber is contained within the tank. An outer perforated cage prevents the cages becoming accidentally detached.
—J. A. B.

*Acetylene, Improved Apparatus for the Production of.* L. Tèdé, Angers, France. Eng. Pat. 658, Jan. 9, 1897.

A bell fixed within an outer cylinder containing water, is surmounted by a receptacle for pulverised carbide, which passes through a way controlled by a valve into the water beneath the bell. The valve can be kept closed by a screw; but when the apparatus is in action, it is opened and closed automatically by a float on the water beneath the bell.
—J. A. B.

*Acetylene Gas, An Improved Apparatus for Generating and Purifying.* R. J. Moss, Abingdon, Berks. Eng. Pat, 1254, Jan. 18, 1897.

An annular bell gasometer in a square tank with sides extending above the bell has in its central cavity a bell-shaped generating chamber, supported on the top of the gasometer by a flanged head. The generating chamber contains wire cages holding carbide, through which and the water in the tank, passes the gas-exit pipe leading to a condensing chamber, whence the gas passes to the gasometer, and thence to a second condensing chamber and the point of consumption.—J. A. B.

*Acetylene Gas, Improved Portable Apparatus for Generating and Burning.* W. P. Thompson, High Holborn, Middlesex. From A. Gobron, Paris. Eng. Pat. 1784, Jan. 22, 1897.

A carbide container is placed below a water reservoir in which is fixed a bell, and the whole is surmounted by the burner, to which the gas passes by a tube from the container. Surplus gas passes by another tube to the bell, and depresses the level of the water therein until it is below the mouth of the pipe which conveys the feed-water through a porous plug drop by drop to the carbide, and thus prevents further action until the surplus gas is consumed. A special valve on the water-feed pipe is actuated by the burner tap, so that water can only pass to the carbide when the tap is open. Surplus gas can also pass by a safety valve to holes around the burner, and be ignited at the latter.—J. A. B.

*Acetylene, Impts. in and relating to Apparatus for the Production of, from Calcium Carbide.* T. Thorp, Whitefield, near Manchester, and T. G. Marsh, Manchester. Eng. Pat. 1929, Jan. 25, 1897.

In apparatus in which granulated carbide is dropped into water, carbide is apt to be crushed between the faces of the feeding valve, and to interfere with the action of the latter. According to this invention the carbide falls from a hopper on to an endless band, which is moved forward by the fall of the gas holder. From the band it falls intermittently into a counterweighted scoop, which discharges it into water in the generator. A feeding valve is thus avoided. An air-pump piston or bellows for feeding the carbide hopper, a safety water-level gauge-glass, and a discharge valve operated by a float are also claimed.—J. A. B.

*Generating [Acetylene] Gas by the Reaction of Solid Substances with Water or other Liquid, Impts. in Apparatus for; especially applicable to the Generation of Acetylene Gas by Contact of Water with Metallic Carbides.* J. C. Mewburn, London. From A. M. Scott, Woodstock, Ontario, Canada. Eng. Pat. 1952, Jan. 25, 1897.

Water under a constant head flows through a sprinkler on to carbide in a vessel provided with a trapped pipe for the removal of any water which is not immediately taken up by the carbide. The gas generated flows into a bell-holder, which maintains a lower pressure than that corresponding to the head of water entering the generator, until the bell comes in contact with a weight suspended at a suitable height. The weight increases the pressure exerted by the holder, and the flow of water to the generator is thus stopped. The gas passes from the holder, through a pressure regulator, to the burners. Safety devices are claimed.
—J. A. B.

*Acetylene Gas, An Improved Portable and Automatic Apparatus for the Production of, for Lighting Purposes.* A. H. Barthez, Algiers. Eng. Pat. 9294, April 12, 1897. (Under Internat. Convention.)

A basket floating within a bell gas-holder receives carbide through a pipe in the crown of the bell from compartments in a wheel, which is rotated through a fixed distance, and one sealed compartment containing carbide is opened each time the bell descends through a given distance. The bell operates the wheel through a suitable counterweighted lever and ratchet and pawl gearing.—J. A. B.

*Acetylene and other Gases rich in Carbon, A Burner for.* G. Lebrun and F. Cornaille, both of Paris. Eng. Pat. 20,571, Sept. 7, 1897. (Under Internat. Convention.)

A hollow cylindrical nipple with paraboloidal tip is made with thin walls of glass or other feebly-conducting material, and is sealed in a metal socket, the base of which is centrally perforated. The emission orifice at the tip is trumpet-shaped externally and rounded internally. The object of the invention is to minimise the heating of the burner and the accumulation of carbon deposits during combustion.
—J. A. B.

*Acetylene Gas, Improved Apparatus for Producing.* A. Floeck and F. Messedat, both of Cologne. Eng. Pat. 22,730, Oct. 1, 1897.

Water passes from a reservoir, through a pipe, to a chamber communicating by a perforated top with a chamber containing carbide, which is attacked by the water until the pressure of the generated gas forces the water back. The gas is purified by passage through a drying chamber containing suitable absorbent material.—J. A. B.

*Acetylene Gas, An Automatic Apparatus for the Production of.* F. Bosshardt, Manchester. From A. Molet, Buenos Aires, Argentine Republic. Eng. Pat. 23,198, Oct. 9, 1897.

Fragments of calcium carbide are put into a hopper, the bottom of which declines towards a shoot leading into a chamber containing water. The gas generated collects in this chamber, and forces water therefrom into an upper chamber. The level of the water in the generating chamber determines, by means of a float, the vibration of the bottom of the hopper, so that, when the store of gas is small, carbide is shaken into the shoot, and more gas is generated. The float puts in operation an electric hammer or mechanism attached to the bottom of the hopper, and thus causes the latter to vibrate.—J. A. B.

*Acetylene Gas Generators, Impts. in.* L. S. Buffington, Minneapolis, U.S.A. Eng. Pat. 23,802, Oct. 15, 1897.

Carbide cartridges consisting of two cylindrical cups engaging telescopically, and therefore capable of expansion, are fed with water dripping from a needle point. The water may reach the carbide in the cartridge through either a feed spout or absorbent material, while the gas generated escapes through perforations. The cartridge is contained in a chamber beneath the reservoir, from which it is supplied with water through an adjustable needle valve. Reading, cycle, and stand lamps, and a large generator acting in unison with the rise and fall of a gas-holder, are described.
—J. A. B.

*Auto-Luminous (Incandescent) Compositions, Impts. in.* A. Syssoyeff and L. Braly, Lyons. Eng. Pat. 24,822, Oct. 26, 1897.

A solution is prepared from "any luminous solution" by adding in suitable proportions, a mixture of salts of iridium and vanadium. For example, to a solution of 100 parts of "soluble salts of thorium" and 1 part of cerium salts, is added a solution of 0·2 part of iridium salts and 0·002 part of vanadium salts. An organic fabric is dipped into this solution, dried, and coated at any suitable part with a second solution ("preferably reduced to a syrup-like consistence") containing salts of the platinum group; as, for example, rhodium salts, 50 parts; ruthenium salts, 50 parts; cerium salts, 2 parts; and vanadium salts, 1 part. After destroying the organic support, the body is heated in a stove along with alcohol vapour and then with pure hydrogen, to complete the reduction; it will then become automatically incandescent when brought into contact with a combustible mixture of gas and air. The claims are for the above prescriptions and for the process of manufacture.
—H. B.

*Incandescible Materials, Processes for Manufacturing.* W. L. Voelker, New Jersey, U.S.A. Eng. Pat. 22,625, Oct. 2, 1897.

Equal parts by weight of oxides of calcium and magnesium are dissolved in nitric acid (to which "a trace" of chromic acid may be added "to impart a ruddy tint to the light"), the solution is evaporated, and the nitrates decomposed to "the combined oxides of magnesium and calcium" by heating the mass in a crucible. The mass is then fused for a short time in an electrical furnace, or otherwise, and allowed to cool; or it may be at once spun into filaments, which may be knitted into mantles.—H. B.

*Incandescible Materials and Mantles, and the Process of making the same; Impts. in.* W. L. Voelker, New Jersey, U.S.A. Eng. Pat. 22,626, Oct. 2, 1897.

Mantles are made in the usual way by impregnating knitted fabric with a 25 per cent. solution of "commercial thorium nitrate" or nitrate of another rare earth. After burning off, the mantle is dipped into an alcoholic solution of calcium and magnesium nitrates, which is prepared by dissolving "combined oxide of calcium and magnesium" (see preceding abstract) in nitric acid, and using one part by weight of the dry nitrate to three parts by weight of alcohol. The mantle is again heated over a Bunsen flame till the nitrate is decomposed into the "combined oxide of calcium and magnesium."—H. B.

*Mantles for Incandescence Gas Lighting, Impts. in.* H. Ottenbruch, Coblenz. Eng. Pat. 24,815, Oct. 26, 1897.

"The essential feature of the improved incandescence body," which is for use with gas under pressure, "is that the same is interwoven with platinum wires . . . such wires passing radially from the contracted upper portion of the mantle and downward in a vertical direction." For specially strong mantles, platinum wire fabric or netting is "connected in any suitable and obvious manner" with an ordinary mantle. Preferably the mantles are corrugated longitudinally.
—H. B.

*[Incandescence] Illuminant Appliances for Gas and other Burners, Impts. in.* J. F. Burgess, Regent Square, W.C. Eng. Pat. 1287, Jan. 18, 1897.

Mucilage, or a substance which contains mucilage, is to be heated till all combustible materials are consumed, the ash is to be dissolved in "pure or aqueous acetate or nitric acid," and mantles are to be prepared by impregnating cotton fabrics with the strained solution. A combination of the "refractory vegetable substance" with rare earths or lime may also be used.—H. B.

*Incandescing Media for use in Incandescent Gas Lighting, Impts. in the Manufacture of.* R. Langhans, Berlin. Eng. Pat. 501, Jan. 7, 1897.

There is claimed the manufacture of incandescing media for use in gas lighting, in which vanadiniferous thoria is employed, the thoria containing from 2 to 5 per cent. of vanadium pentoxide. For utilising the material, any of the known ways may be employed, such as impregnating a fabric with a solution containing thorium and vanadium, or first making a body from thoria and then impregnating it with aqueous vanadiniferous solution, &c.—H. B.

*Incandescent Mantles for Gas Burners, Impts. in.* F. R. Foster, Milwaukee, U.S.A. Eng. Pat. 11,739, May 11, 1897.

To the outside of a suitable combustible base, preferably in the form of a network hood, is applied a thin layer of graphite, on which is formed a thin layer of copper by electro-deposition. There is next applied a coating of platinum, and finally "a somewhat heavier coating" of ruthenium. The mantle is then highly heated, to burn away the combustible base. The mantle and process of manufacture are claimed.—H. B.

*Petroleum Incandescence Lamps, Impts. in Burners for.* M. Frank, Frankfurt-on-Main, Germany. Eng. Pat. 417, Jan. 6, 1897.

On a petroleum container is fitted a wick tube and burner, carrying a circular wick and provided with the usual central air supply. Above the wick tube is the usual concentric deflector. The lamp having been lit, there is slipped over the wick tube a dome-shaped cap, open at the top and carrying the incandescence mantle and a comparatively high chimney. The cap is provided at its base with two sets of air-inlets, the one supplying an air current between the outer surface of the cap and the inner surface of the chimney, and the other supplying a current which is directed by the inner surface of the cap against the lower edge of the flame. When the cap is slipped over the luminous circular flame, the latter is immediately rendered non-luminous. The combination of the parts in a burner is claimed.—H. B.

*Inverted Regenerative Incandescent Gas Burners or Lights.* H. A. Kent, Bounds Green, N. Eng. Pat. 3667, Feb. 11, 1897.

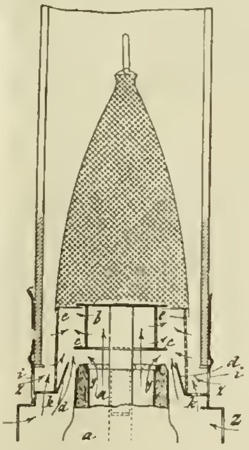
The burner claimed, which is designed to give a downward light free from shadows, comprises a tube which rises upwards, and is at its upper end turned over and downwards for a short length. At the lower end of the longer limb is situated a gas-inlet nozzle and air-inlet holes of the usual type. The short turned-down limb carries an inverted burner from which depends a mantle or the like, of refractory material, the whole being surrounded by a globe.
—H. B.

*Carbonaceous Agent [Naphthalene] for Enriching Methylated Spirit and the like, A Process for Producing.* H. Hempel, Berlin, Germany. Eng. Pat. 16,232, July 8, 1897.

The object of this invention is to produce a soluble medium which, when added to spirit, shall render the same rich in carbon, so that it will burn with a luminous flame. Volatile oils, such as turpentine, are best suited for the purpose; and in order to increase the amount of carbon contained therein, without simultaneously augmenting the amount of resin, solid hydrocarbons, and particularly naphthalene, are employed. To prevent the generation of smoke, the naphthalene is treated with sulphuric acid, to render it completely soluble in cold water, and owing to the increased solubility it is said to combine more intimately with the spirit.
—D. B.

*Incandescent Oil Vapour Lamps, Impts. in or relating to Burners for.* L. Durra, Brussels. Eng. Pat. 23,127, Oct. 8, 1897.

Surrounding the top of the burner tube *a*, containing the circular wick, is a conical tube *d*, which directs an air current of sufficient velocity to prevent the vapours from igniting on the outer side of the wick; the inner air current *y*, of less velocity, allows a flame to burn at the inner side of the wick. The cap *b* is perforated laterally, and carries the flange *c*; the perforated tube *e* is preferably slightly conical, to let the mantle be slipped over it easily. There are air-inlets at *i*, *k*, and *z*. For convenience in lighting the lamp, the upper parts may be raised by such means as the usual device of a key and levers. When the lamp is in action, owing to the conflicting currents of hot and cool gases passing through *b* and *e* respectively, "the flame is divided so as to 'curl' or curve, so that no sharp tongues are formed," and the flame spreads uniformly. The lamp as a whole, and the various combinations of parts, are claimed.—H. B.



*Electric Arc Furnaces [Automatic; Carbides, &c.], Impts. in.* The Acetylene Illuminating Co., Ltd., London, and P. R. Day, Foyers, Inverness, N.B. Eng. Pat. 27,323, Dec. 1, 1896.

*See under* XI. A., *page* 52.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Shale Oils, Scotch; Composition of: A Contribution to the Theory of the Formation of Tar.* F. Heusler. Ber. 1897, **30**, 2743—2752.

With regard to the formation of aromatic hydrocarbons from bituminous substances by destructive distillation, the author is of opinion that their origin is not entirely due to synthesis from pyrogenetic intermediate products, such as acetylene, but he attributes their existence largely to primary products of the distillation of substances, present in coal, which belong to the aromatic series.

It has been found that the light fractions from lignite tar contain benzene and its homologues, and even naphthalene, in large quantities, whilst the corresponding fractions obtained from shale tar yield but little benzene and apparently no naphthalene. Quantitative experiments have given the following results, fractions boiling at about 110°:—

| —— | Lignite Tar. | Shale Tar. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Paraffins | 16·0 | 42·0 |
| Naphthenes | 4·0 | 10·0 |
| Aromatic hydrocarbons | 45·0 | 7·3 |
| Ethylenes | 31·0 | 39·0 |

These results accord with the fact that brown coal is essentially of vegetable origin, and is, as a rule, associated with fossil resins, which on distillation yield oils, the nature of which points to their relationship to the aromatic hydrocarbons. The bituminous shales of Scotland, on the other hand, are of animal origin. According to Nicholson they belong to the lower carboniferous series, and rest upon, or form part of, the Burdiehouse limestone, an estuarial formation rich in the remains of fishes. They are succeeded by the higher beds of the oolitic limestones, which are of marine origin. It is probable that the paraffin shales were formed under water, and that the carbon which they contain is of animal origin. This explains the small percentage of aromatic hydrocarbons present in the oils obtained from shale by distillation, and affords evidence of the formation of ammonia in considerable quantity. It is also interesting to note that besides pyridine and pyrrol, nitriles of the fatty acid series have been detected—a circumstance which clearly demonstrates the analogy of shale oil to bone oil.

The experimental part of the paper is devoted to a description of the methods resorted to for the purpose of identifying the presence of paraffins, naphthenes, aromatic hydrocarbons, ethylenes, and fatty acid nitriles in shale oil. —D. B.

*Petroleum, Californian; Composition of.* C. F. Mabery. Amer. Chem. J. **19**, [9], 796—804.

*Ventura County Oil.*—The characteristics of the crude oil are: sp. gr. at 20° C., 0·8882; colour, dark, nearly black—brownish in transmitted light; bromine absorption, 17·72 per cent., *i.e.*, higher than Pennsylvania or Ohio oil, but approximately the same as Oil Springs (Canada) oil. Composition:—Carbon, 85·6; hydrogen, 12·84; sulphur, 0·84; nitrogen, 0·53 per cent.

*Distillates under Atmospheric Pressure.*

| Boiling Point. | Yield. | Bromine Absorption. | Specific Gravity. |
|---|---|---|---|
| | Per Cent. | | |
| Below 150° C. | 9·7 | 5·60 | 0·7395 at 20° C. |
| 150°—200 | 29·1 | 6·00 | 0·7823 „ |
| 200°—250° | | 9·17 | 0·8252 „ |
| 250°—300° | | 18·15 | 0·7591 „ |
| Residue | 61·2 | .. | 0·9528 at 45° C. |

Those formed above 250° C. are coloured, and exhibit an odour of decomposition, both characteristics increasing with the temperature. The residue is a thick solid tar, and its qualities and amount explain the difficulty experienced in preparing burning oil from this petroleum.

The constituents boiling above 150° C. were separated into fractions at intervals of 25° by distillation *in vacuo* (50 mm.), the specific gravity ranging from 0·7342 (fraction up to 100° C.) to 0·9071 (fraction 250°—275°). The fractions all remained undecomposed, retaining the natural odour of the crude oil. The highest fraction deposited paraffin in quantity when exposed to a freezing mixture, but very little was obtained from the next lower fraction.

In examining the distillates below 200° C. for the preponderence of the $C_nH_{2n+2}$ series, it was found, after repeated distillations under ordinary pressure, that the fraction 78°—81° contained a large proportion of benzene, and toluene was detected in the fraction 108°—112°. The specific gravity of the fraction 97°—100° corresponded approximately to Russian distillate (95°—100°), both before and after purification (*viz.*, 0·7341 : 0·718, and 0·7268 : 0·733), and, from this similarity and its composition, apparently consisted of heptanaphthene. The fraction 117°—119° yielded a nitro-product (presumably nitroxylene), and appeared to consist of octonaphthene. The values yielded by the fraction 123°—125° are believed to preclude the presence of any considerable proportion of normal octane, and in such case the fraction would probably be separable into higher and lower constituents by further distillation. Both in density and composition, the fraction 159°—160° approximated to decanaphthene, and the purified fraction 169°—172° agreed very closely with the theoretical percentage composition for the $C_nH_{2n}$ series.

*Fresno County Oil.*—This is of a lighter and more greenish hue than Ventura oil, and has a lower specific gravity, *viz.*, 0·8423 at 20° C. Composition:—Carbon, 86·24; hydrogen, 13·08; sulphur, 0·21; nitrogen, 0·11 per cent. Bromine absorption, 9·07 per cent.

*Fractions distilled under Atmospheric Pressure.*

| Boiling Point. | Yield. | Bromine Absorption. | Specific Gravity at 20° C. |
|---|---|---|---|
| | Per Cent. | | |
| Below 150° C. | 33·06 | 17·21 | 0·7829 |
| 150°—200° | 24·79 | 14·23 | 0·8268 |
| 200°—250° | 20·66 | 32·10 | 0·8725 |
| 250°—300° | 12·40 | 21·68 | 0·9090 |
| Residue | 9·09 | .. | 0·9356 |

Both the specific gravity and bromine absorption are here greater than in the distillates from Ventura oil. —C. S.

## PATENT.

*Hydrocarbons, Liquid, and similar Substances; Impts. relating to the Distillation and Breaking up of.* P. J. Dawes, Greenwich, Kent. Eng. Pat. 2515, Jan. 30, 1897.

The apparatus consists of a still made of copper, iron, or other metal, which is heated by means of a copper coil. The still is connected with two feed pipes leading from an elevated store tank. One feed enters the still near the top, and is used for charging purposes only; the other feed goes through the side, close to the bottom, and is fitted to a coil of copper pipes leading upwards just above the centre of the still, where it terminates in a revolving or stationary rose or suitable jet. At the bottom of the still a pipe is fixed, with valve for removing the heavy grades of spirit. Having filled the still half full with liquid from the top feed, steam is passed through the coil, and as soon as distillation commences, liquid is introduced in a continuous stream through the bottom feed. In its upward passage through the coil, it absorbs heat from the hot residue, and when it reaches the revolving jet, it sprays a warm liquid on to the boiling mass below, and in doing so the light portions are volatilised. The valve at the bottom of the still is then regulated, to allow the residue at the bottom to pass out at a convenient rate.—D. B.

## IV.—COLOURING MATTERS AND DYES.

*Friedel and Crafts' Method, Modification of.* A. Verley. Bull. Soc. Chim. 1897, **17**, 906—914.

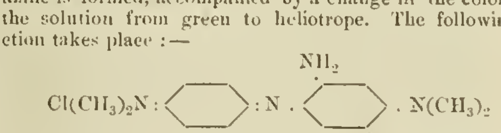
Although the yields of aromatic hydrocarbons effected by means of the aluminium chloride synthesis are satisfactory, such is not the case when attempts are made to obtain oxygenated compounds. For instance, the action of acetyl chloride on benzene in presence of aluminium chloride only gives about 20 per cent. of the amount of acetophenone theoretically obtainable. The author's modification consists in using a larger amount of aluminium chloride than that prescribed by Friedel and Crafts, and in working under reduced pressure at a low temperature. Thus he obtains cresylmethyl ketone, as follows:—1 kilo. of toluene and two molecular proportions of aluminium chloride are placed in a flask, which is then exhausted, and after cooling to 0° C., two molecular proportions of acetyl chloride are added drop by drop. The reaction proceeds steadily, the temperature rising to 5°—10° C. When the whole of the acetyl chloride has been added, the temperature is allowed to rise to 20° C., the vacuum still being maintained. On pouring the melt into water slightly acidulated with hydrochloric acid, two layers are formed, the upper consisting of toluene and cresylmethyl ketone. The former is separated by fractional distillation, and 162 grms. of the ketone are obtained, boiling at 168° C. under 16 mm., and at 215° C. under 760 mm. pressure. The specific gravity at 0° C. is 1·021. Only the para compound is obtained. Several other examples are given.—T. A. L.

*Nitrotoluenes, The Separation of.* G. A. Schoen. Bull. Soc. Ind. Mulhouse, 1897, 391—392.

In a sealed communication, dated Oct. 22, 1886, the author describes the commercial separation of *o*- and *p*-nitrotoluene as carried on in the works of The Fabriques de Produits Chimiques de Thann et de Mulhouse. The process is a fractional distillation in a cast-iron retort, heated indirectly by means of superheated steam, or by circulation of mineral oil, &c., and provided with a Savalle's column. The distillate is redistilled until an almost pure *o*-nitrotoluene passes over, and the residues on cooling deposit crystalline *p*-nitrotoluene.—R. B. B.

*Nitrodimethylanilines, Reduction Products of.* E. Noelting and E. Fourneaux. Ber. **30**, 2930—2947.

Dimethylaniline is nitrated by dissolving 2 kilos. in 40 kilos. of sulphuric acid (66° B.) and running in a mixture of 1,930 grms. of nitric acid (38° B.) and 600 grms. of sulphuric acid. The next day the melt is poured into 50 litres of water containing 50 kilos. of ice, and treated with soda lye until red crystals of *m*-nitrodimethylaniline begin to precipitate. The *p*-derivative is filtered off, whilst the *m* derivative is precipitated from the filtrate on the addition of more alkali. The mother-liquor is then steam-distilled and the distillate extracted with toluene. On shaking this with moderately concentrated hydrochloric acid and saturating the acid liquor with ammonia, a further quantity of *m*-nitrodimethylaniline is obtained, together with a little dimethylaniline, and the toluene solution when evaporated, gives *p*-nitrodimethylaniline and traces of a yellowish oil. The *m*-derivative is easily purified by recrystallisation from alcohol, the *p*-derivative being sparingly soluble, but it is more difficult to obtain the *p*-derivative free from meta. This is best effected by fractional crystallisation from alcohol and extraction with petroleum ether, which dissolves the *m*-derivative. The *m*-derivative, on reduction with iron and hydrochloric acid, yields dimethyl-*m*-phenylenediamine, but feebler reducing agents give the following products :—Alcoholic potash yields a mixture of *m*-azoxy- and *m*-azodimethylaniline, zinc in an alkaline alcoholic solution or sodium amalgam gives the same mixture or *m*-hydrazodimethylaniline, whilst sodium methylate, which only reacts in a very concentrated solution, gives *m*-azoxydimethylaniline. The hydrazo compound, when treated with acids, yields a tetramethylbenzidine together with dimethyl-*m*-phenylenediamine and a base which has not been identified. The azoxy derivative is prepared by heating 20 grms. of *m*-nitrodimethylaniline with 4 grms. of sodium in 50 grms. of methyl alcohol, first to 100° C. and subsequently to 125° C until a homogeneous mass is obtained, more alcohol being added if necessary to prevent the mass drying. The melt is then diluted with water, the azoxy derivative filtered off and recrystallised from alcohol, benzene, or petroleum ether. It forms orange-red crystals, melting at 89° C., and gives colourless salts. The same substance is obtained by methylating *m*-azoxyaniline. When dissolved in 100 per cent. sulphuric acid and heated for three hours to 110° C., the colour changes from orange to red, which probably indicates an isomeric change to an oxyazo compound, although no crystalline product could be isolated. In order to obtain *m*-azodimethylaniline, a solution of 100 grms. of *m*-nitrodimethylaniline in 500 c.c. of alcohol is gradually treated with 125—150 grms. of zinc dust and 30—50 c.c. of soda lye (38° B.). The mixture is finally heated for about 20 minutes on the water-bath, and the filtered solution, on cooling, deposits red needles, which melt at 118° C. The salts are orange, more sparingly soluble and more stable to water than the azoxy salts. Attempts to prepare the dimethyl derivative by methylating *m*-azo-aniline only yielded a violet dyestuff, presumably an induline, although the action of methyl iodide on *m*-azodimethylaniline yielded the normal iodomethylate. The orange-yellow crystals of this substance turn brown at 190° C. and melt at 230° C. On treatment with moist silver oxide, a very unstable ammonium base is obtained, which decomposes on evaporating its solution, into *m*-azodimethylaniline and methyl alcohol. By reducing *m*-azodimethylaniline with sodium amalgam in an atmosphere of carbonic acid or of hydrogen, it is possible to obtain *m*-hydrazodimethylaniline, which is tolerably stable when dry. It crystallises from ether in white needles melting at 100° C., and is converted by the action of mineral acids into two or three isomeric benzidine, diphenyline, or semidine derivatives. Tetramethyldiamidobenzidine is best obtained by reducing *m*-azodimethylaniline with sodium amalgam in alcoholic solution, adding hydrochloric acid, warming, and pouring into ammonia. The benzidine is precipitated together with a little zinc oxide, and is purified by recrystallisation from benzene-petroleum ether. The yield is about 60 per cent., soluble bases being formed to about 25 per cent. Tetramethyldiamidobenzidine crystallises in colourless needles melting at 166° C., and gives the following characteristic reaction. A drop of nitrite added to a very dilute acid solution of the base, shows an intense violet coloration, which rapidly changes to brown. The benzidine, when treated with methyliodide and heated for half an hour on the water-bath, yields the di-iodomethylate of tetramethyldiamidobenzidine, which, when converted into the hydrobromide and heated with methyl iodide, decomposed with moist silver oxide, treated with hydriodic acid, and finally with ammonia, gives the di-iodomethylate of octomethyldiamidobenzidine ; colourless needles melting at 190° C. The prolonged heating of tetramethyldiamidobenzidine with an excess of methyl iodide and the theoretical amount of sodium carbonate, yields a tetra-iodomethylate of octomethyldiamidobenzidine, which melts at 205° C. Tetramethyldiamidobenzidine will combine with one or two molecules of a diazo compound to form dyestuffs, and it is noteworthy that the sulphonated derivatives (for instance, from sulphanilic acid) are weaker dyestuffs than the non-sulphonated. The product obtained from equimolecular proportions of diazobenzene chloride and tetramethyldiamidobenzidine forms orange-coloured needles melting at 221° C., easily soluble in benzene, sparingly in petroleum ether. The shades on animal fibres and on tannin are brown. The disazo derivative is a very similar substance, but dyes redder shades. Tetramethyldiamidobenzidine also reacts with nitrosodimethylaniline hydrochloride to form an indamine, but in an acetic acid solution oxidation takes place rapidly, and an azine is formed, accompanied by a change in the colour of the solution from green to heliotrope. The following reaction takes place :—



The zinc chloride double salt forms lumps similar to Methyl Violet. The aqueous and alcoholic solutions are heliotrope. The dyestuff gives bright bluish-violet shades on unmordanted cotton.

The isomeric diphenyline derivatives are very similar, but dye fuller shades; they do not, however, go on to unmordanted cotton.

The reduction of *p*-nitrodimethylaniline proceeds much less satisfactorily than that of the *m*-derivative. The *p*-azoxydimethylaniline is best prepared according to O. Fischer and Wacker (Ber. **21**, 2612), whilst the alkaline reduction gives a product, after frequent purification, which forms orange needles melting at 265° C. This substance has also been obtained following the action of nitrosodimethyl aniline on phenylhydrazine, by passing nitrous acid through an alcoholic dimethylaniline solution, by combining dimethylaniline with dimethylamido-*p*-diazobenzene chloride, and by methylating *p*-amidobenzene-azodimethylaniline.

—T. A. L.

### *Yellow Colouring Principles of Various Tannin Matters. IV.* A. G. Perkin. Proc. Chem. Soc. 1897, [183], 170.

Cape sumach, the leaves of the *Colpoon compressum*, is used in South Africa as a substitute for sumach (*Rhus Coriaria*) under the name of "Pruim-bast." According to H. Procter (private communication), it contains 23 per cent. of a catechol tannin. Its dyeing property is due to the presence of a new glucoside, *osyritrin*, $C_{27}H_{30}O_{17}$, pale yellow needles, melting point 185°, which is decomposed by acid into quercetin and glucose—

$$C_{27}H_{30}O_{17} + 2H_2O = C_{15}H_{10}O_7 + 2C_6H_{12}O_6.$$

This is not identical with viola-quercetrin, $C_{42}H_{42}O_{24}$, which exists in the *Viola tricolor florensis*. The tannin, obtained as an orange-coloured, transparent mass, is a glucoside, yielding with acid, an anhydride or phlobophane and a sugar. By fusion with alkali, protocatechuic acid is formed. A re-examination of gambier catechu (*Uncaria Gambier*) corroborated the statement of Löwe (Zeits. Anal. Chem. 1874, **12**, 127) that this contains quercetin. Acacia catechu not previously examined, was found to contain the same colouring matter.

The dyeing properties of a commercial sample of Venetian sumach (*R. Cotinus*), are due to myricetin, and not quercetin, as stated by Löwe (*loc. cit.*). This result will be corroborated by the examination of a specially picked sample.

Valonia (*Quercus Ægilops*), divi-divi (*Cæsalpinia Coriario*), myrabolans (*Terminalia chebula*), agarobilla (*Cæsalpinia brevifolia*), pomegranate rind (*Punica granatum*), and gall-nuts (*Quercus infectoria*), owe their tinctorial property to ellagic acid, and contain no member of the quercetin group. It is here pointed out that the plants examined hitherto, contain respectively, a tannin and colouring matter, which yield on decomposition identical acids, and in some cases the same phenol.

### *Yellow Vegetable Colouring Matters.* A. G. Perkin. Proc. Chem. Soc. 1897, [183], 198; also J. Chem. Soc. 1897, **71**, 1194.

The *Rhus rhodanthema*, a tree growing to the height of 70 or 80 feet, is indigenous to northern New South Wales. The colouring matter, $C_{15}H_{10}O_6$, is identical with fisetin. A *glucoside* of fisetin, $C_{36}H_{30}O_{16}$, is also present: it is decomposed with difficulty by boiling dilute acids. This closely resembles fustin, $C_{58}H_{46}O_{23}$ or $C_{36}H_{26}O_{14}$, the fisetin glucoside of *R. Cotinus* (Schmid, Ber. 1886, **19**, 1753), but differs from it in percentage composition. Gallic acid was also isolated, evidently as a decomposition product of gallotannic acid contained in the wood.

*Berberis orturensis*, a plant resembling *Berberis vulgaris*, flourishes in Cyprus. It was found to contain berberine, but no colouring matter of the mordant yellow class.

The perianths surrounding the seeds of *Rumex obtusifolius* contain a trace of quercetin, which is interesting, as in many roots of this species, methylanthraquinone derivatives also exist. It is also pointed out that the leaves and green stems of madder (*Rubia tinctoria*) contain a yellow colouring matter, which will be examined.

### *Bismarck Brown II.* E. Täuber and F. Walder. Ber. **30**, 2899—2902.

In continuation of their former paper (Ber. **30**, 2111; this Journal, 1897, 905), the authors now find that commercial Bismarck Brown consists principally of *m*-phenylenedisazo-*m*-phenylenediamine. They have extracted this product from Bismarck Brown base, and have also obtained it by combining tetrazotised *m*-phenylenediamine with *m*-phenylenediamine in presence of an excess of sodium carbonate. This latter operation is carried out as follows:—80 c.c. of fuming hydrochloric acid are mixed with five times the weight of ice, cooled externally, and treated with a solution containing 15 grms. of sodium nitrite. To this solution is then added a cold solution of 9 grms. of *m*-phenylenediamine hydrochloride together with 10 c.c. of fuming hydrochloric acid. After five minutes, the solution is gradually run into a well-cooled solution of 11 grms. of *m*-phenylenediamine base together with 200 grms. of crystallised sodium carbonate in about 3 litres of water. The precipitate obtained, is boiled with sodium carbonate, dissolved in very dilute warm hydrochloric acid, filtered, saturated with ammonia, and the base dried over sulphuric acid, and then extracted with benzene. Brownish-yellow crystals separate from the benzene on cooling, melt at 118° C., and, on recrystallisation from phenol and benzene, yield darker crystals, containing phenol, melting at 136° C., and identical in every respect with the substance already extracted from Bismarck Brown base.—T. A. L.

### *Malachite Green Series, New Method of Formation of Dyestuffs of the.* E. Noelting. Ber. **30**, 2588—2590.

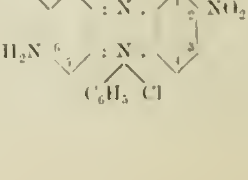
According to Ger. Pat. 41,751 of 1887 (Farbwerke Höchst), a general method is given for the production of dialkylamidobenzophenones by condensing benzanilide with a tertiary aromatic amine by means of phosphorus oxychloride. The Auramine formed is then hydrolysed by dilute acid. If, however, the benzanilide be replaced by salicylanilide, the reaction takes a different course: a Malachite Green derivative is produced, and with dimethylaniline, the same product is formed as that obtained by O. Fischer by condensing salicylaldehyde with dimethylaniline. There are gradually added to a mixture of 15 grms. of salicylanilide and 40 grms. of dimethylaniline, 15 grms. of phosphorus oxychloride, the temperature being kept at about 125° C. The melt is then heated for six hours on the water-bath, dissolved in water containing a little hydrochloric acid, and the dyestuff salted out. A yield of 24 grms. was obtained, equal to 89·6 per cent. of the theoretical. The anilide of *o*-cresotic acid—

$$1.3.4—C_6H_3.CH_3.OH.OCNHC_6H_5$$

yields a similar product, whilst the anilides of *m*- and *p*-hydroxybenzoic acid, *o*-nitro- and *o*-amidobenzoic acids, do not give Malachite Green derivatives. On the other hand, the anilide of *o*-hydroxynaphthoic acid (m.p. 216° C.) reacts as above. Equal weights of the anilide, dimethylaniline, and phosphorus oxychloride were mixed as already described, and heated for 3—4 hours on the water-bath. A green dyestuff is produced, which is very sparingly soluble in water, and easily in alcohol. On sulphonation with fuming sulphuric acid, it is rendered soluble, and dyes silk, wool, and tannined cotton a yellowish green. It would appear that the condensation of tertiary amines with benzanilide, to form dyestuffs of the Malachite Green series, is dependent upon the presence of an *o*-hydroxyl group in the benzoic acid radicle.—T. A. L.

### *Rosinduline, Fifth Isomeric.* F. Kehrmann and O. Feder. Ber. **30**, 2637—2641.

By condensing amido-β-naphthoquinone (oxynaphthoquinone-imide) with that nitro derivative of *o*-amidodiphenylamine which is obtained by partial reduction of ordinary 1.2.4-dinitrodiphenylamine by means of alkaline sulphides, a nitro-rosinduline is obtained having the following constitution:—

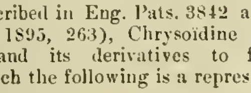


By removing the amido and reducing the nitro group, salts of a new Rosinduline (2-amidophenylnaphthophenazonium) are obtained, ordinary Rosinduline being 6-amidophenylnaphthophenazonium according to the above scheme. The condensation is effected by boiling together 5 grms. of nitro-amidodiphenylamine hydrochloride, 4·5 grms. of oxynaphthoquinone-imide, and 400 c.c. of alcohol until all is dissolved; 10 c.c. of concentrated hydrochloric acid are then added, and the whole is allowed to cool. The precipitate, which deposits after standing two hours, is extracted by boiling with water and a little hydrochloric acid, and the pure Nitro-rosinduline chloride separates from these combined extracts on the addition of concentrated hydrochloric acid. It forms a dark red-greenish metallic powder having the formula $C_{22}H_{15}N_4O_2Cl$, much more sparingly soluble in water than rosinduline chloride, and much yellower in shade. The alcoholic solution has a yellowish fluorescence. Concentrated sulphuric acid gives a yellowish-green solution, which, on dilution with water, passes through bluish-green to red.

2-Nitrophenylnaphthophenazonium is obtained as follows:—Nitro-rosinduline chloride is dissolved in concentrated sulphuric acid, and so much water is subsequently added that the solution becomes greenish-blue. Sodium nitrite in concentrated solution is then added until a clear blood-red solution is obtained. This is then poured into twice its volume of alcohol, when a vigorous evolution of nitrogen takes place, and after standing two hours, the ferric chloride double salt $C_{22}H_{14}N_3O_2Cl.FeCl_3$ is precipitated as a sandy powder by adding a concentrated solution of ferric chloride. The nitrate is obtained by adding sodium acetate to a solution of the iron double salt, and filtering the solution into dilute nitric acid (sp. gr. 1·1). This salt is stable in presence of nitric acid, but when boiled with water, a precipitate of Nitrorosindone is formed. The chloride forms greenish needles, soluble in concentrated sulphuric acid with a reddish-violet colour, which turns yellowish-red on the addition of water. The platinochloride, bichromate, and aurochloride are all insoluble reddish-yellow powders. On reduction of the chloride with stannous chloride in an alcoholic solution, Isorosinduline chloride No. 5 is obtained, having the formula $C_{22}H_{16}N_3Cl$. This product forms blackish-violet needles, which dissolve in pure water to a brownish-violet solution. The addition of concentrated hydrochloric acid turns the solution yellowish-red. The salt dissolves in concentrated sulphuric acid with a reddish-violet colour, which, on addition of water, turns yellowish-red, and then violet-red. The nitrate $C_{22}H_{16}N_3NO_3 + H_2O$ possesses the characteristic property of being nearly insoluble in water acidulated with nitric acid, and forms greyish-violet needles.—T. A. L.

### *Triazine Derivatives of Chrysoïdine and of o-Amido-azotoluene.* E. Noelting and F. Wegelin. Ber. 30, 2595—2604.

As already described in Eng. Pats. 3842 and 3843 of 1894 (this Journal, 1895, 263), Chrysoïdine condenses with benzaldehyde and its derivatives to form colourless triazines, of which the following is a representative:—



$$\begin{array}{l} C_6H_5.N\;.\;N\diagdown \\ \quad\;\;\cdot\qquad\cdot\;\;\rangle C_6H_3.NH_2 \\ C_6H_5.CH.N\diagup \end{array}$$

These products, containing a free amido group, can be diazotised and combined with various components. The triazines can, moreover, be sulphonated, or can be prepared from sulphonated Chrysoïdines, and their alkali salts possess a very sweet taste. The azo dyestuff from one of these, combined with salicylic acid, is known as Chrome Fast Yellow. In order to test whether the sweetness was due to the presence of the amido group, this was replaced by iodine, by means of Sandmeyer's reaction, but the taste remained. Similarly, three isomeric triazine monosulphonic acids prepared from sulphonated Chrysoïdines were also sweet, but a fourth from Chrysoïdine and *m*-sulphobenzaldehyde appeared to taste bitter. By condensing *o*-, *m*-, and *p*-nitrobenzaldehydes with Chrysoïdine in a methyl alcoholic solution, nitrotriazines of a yellow colour are obtained, which yield amidotriazines on reduction, and these on diazotisation and combination with 2.1′.3′-amidonaphthol sulphonic acid G, and with 1.1′.3.3′-amidonaphthol disulphonic acid H, yield dyestuffs for unmordanted cotton, giving violet to blue shades, unstable, however, to acids. The combinations took place in alkaline solution, and it was observed that an increase in strength was noticeable, passing from the ortho to the para derivatives. Similar triazines have also been obtained from *o*-amido-azotoluene (from *p*-toluidine) and benzaldehyde derivatives. Thus, from *o*-nitrobenzaldehyde, a product is obtained crystallising in yellow needles, and melting at 230° C.; the *m*-derivative, somewhat less soluble, melts at 228° C.; whilst that from *p*-nitrobenzaldehyde melts at 264° C. The *m*-hydroxybenzaldehyde derivative forms white needles melting at 265° C. The action of phthalic anhydride on *o*-amido-azotoluene was also investigated, since in certain reactions this behaves similarly to benzaldehyde; it was found, however, that the yellow crystalline compound melting at 220° C. was a simple acidyl derivative.—T. A. L.

### *Artificial Indigo.* O. N. Witt. Chem. Ind. 1897, **20**, [20], 154—157.

Under the name of "indigo. pure," synthetically prepared Indigotine has recently been sent into commerce by The Bad. Anilin und Soda Fab. It appears in the form both of powder and paste, and, compared with the natural product, offers the following advantages:—

*First*, owing to an extremely fine state of division, the author states that it enables the tedious and expensive operation of grinding, necessary in the preparation of Indigo for use, to be dispensed with. *Secondly*, as the product is pure, with its use, a more effective control of the vats can be maintained than was formerly possible; moreover, the amount of Indigotine consumed from time to time may be determined with exactitude. *Thirdly*, it is easily tested and valued.

The product is already offered in commerce at about the same *pro ratâ* price as good qualities of ordinary Indigo. It is therefore cheaper than the refined Indigos of commerce, which are sold at a higher rate than ordinary Indigos.

It is found in practice that, whilst the new product, in dyeing, behaves towards wool, similarly to natural Indigo, it does not dye cotton so well as the latter does. This is owing to the presence in Indigo of impurities which act as mordants for the vegetable fibre. In connection with this discovery, the further discovery is made that certain compounds—notably albuminoids, especially glue, and fatty matters, amongst which Turkey-red oil may be particularly mentioned—act even more powerfully in this capacity than the substances contained in Indigo. These compounds may be added to the dye-vats, but it is more practicable to impregnate the fibre with them before dyeing, as is the usual practice when dyeing with mordants. The cotton thus prepared not only dyes in a satisfactory manner, bu also exhibits, when dyed, a fine purple shade, which contrasts greatly with the shade ordinarily obtained, and especially with the weak greenish shade obtained from the artificial product in the absence of such mordanting compounds.

The synthetical product is also advantageously employed in calico printing, and in the manufacture of indigo extract and carmine. For the latter purpose, it is pointed out that the manufacturers (The Badische Anilin und Soda Fab.) of the new product have for some time past employed refined natural Indigo, which answers better than the crude dyestuff. The results obtained, however, with the synthetical product are so much better, that the firm has recently decided to use it exclusively.—E. B.

### *Chloride of Nitrogen, Action of, upon Aniline, and also Methyl- and Ethylaniline.* W. Hentschel. Ber. 1897, **30**, [17], 2643.

*See under* XXIV., *page* 79.

## PATENTS.

*Colouring Matters [Tartrazine], Impts. in the Manufacture of, and of New Intermediate Products.* J. Y. Johnson, London. From The Badische Anilin und Soda Fab., Germany. Eng. Pat. 765, Jan. 11, 1897.

The process given in Eng. Pat. 5693 of 1893 (this Journal, 1894, 382) for the preparation of Tartrazine gives such a small yield of dyestuff as to be practically useless. According to the present method, which gives a largely increased yield, the hydrazone formed according to the above patent is converted by splitting off alcohol into a pyrazolone derivative, which is then combined, before or after saponification, with diazosulphanilic acid. The following are the quantities employed :—About 9·5 kilos. of phenylhydrazine sulphonic acid, 7·5 kilos. of crystallised sodium acetate, and 9·4 kilos. of oxalylo-acetic ether are suspended in about 50 litres of water, agitated, and warmed to 50° C. until a clear solution results. The solution then contains the hydrazone, and, after cooling to the ordinary temperature, about 8—10 kilos. of calcined soda are added until the solution reacts distinctly alkaline. The formation of the pyrazolone derivative (tartrazinogen-ester sulphonic acid) proceeds rapidly, and is complete when successive samples show no increase in the amount of precipitate thrown out by dilute sulphuric acid. The whole melt is then precipitated with dilute sulphuric acid, and the sulphonic acid is filtered off, pressed, and dried. In order to saponify it, 167 kilos. of the sodium salt are dissolved in about 2,000 litres of water and warmed for about half an hour on the water-bath with 115 kilos. of caustic soda lye (35 per cent. NaOH). The new tartrazinogen sulphonic acid is precipitated with hydrochloric acid and collected in the usual way. By treating the solution of the hydrazone with caustic soda as above, the formation of the tartrazinogen-ester sulphonate and its saponification are effected in one operation. For the conversion into Tartrazine, the alkaline solution of the tartrazinogen sulphonic acid as obtained, has added to it 75 kilos. of crystallised sodium acetate, and subsequently, at 5—10° C., the amount of diazosulphanilic acid equivalent to 34·5 kilos. of sodium nitrite. After standing about 20 hours, the Tartrazine is salted out and collected in the usual manner. The saponification, instead of being an intermediate operation, may be the final one, and for this purpose the tartrazinogen-ester sodium sulphonate solution obtained as above is diluted with water up to 200 litres and treated with the amount of diazosulphanilic acid equivalent to 3·45 kilos. of sodium nitrite. The tartrazine ester formed, after occasional stirring for about 12 hours, is then separated by salting out, and subsequently saponified; or it can be saponified directly. This operation is best effected by heating with caustic alkali on the water-bath.—T. A. L.

*Rhodamine Dialkylamides, Manufacture of.* O. Imray, London. From The Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/M., Germany. Eng. Pat. 1592, Jan. 20, 1897.

By acting with phosphorus oxychloride on Rhodamines and subsequently treating the products so obtained with secondary aliphatic amines, valuable dyestuffs are obtained. For instance, 1 kilo. of diethylrhodamine hydrochloride is mixed with 3 kilos. of phosphorus oxychloride, and the reaction is finished by heating to 80° C. The brownish-red mass, when cold, is extracted with benzene, and the insoluble oil is mixed with about 10 times the quantity of chloroform and diethylamine. After heating for some time to 50° C., the chloroform and excess of diethylamine are distilled off, the residue is extracted with hot water, and the new dyestuff is precipitated from the filtered solution as the zinc chloride double salt. It dissolves in water with a yellowish-red colour and fluorescence, and is unaffected by alkaline carbonates, but easily decomposed by free alkalis.
—T. A. L.

*Colouring Matters [Malachite Green Series], The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 3452, Feb. 6, 1897.

Alkylated diamidodiphenyl carbinols are condensed with sulphonic acids of the benzene series containing an amido group meta to the sulphonic acid group and having a free para position to the amido group. Thus, by condensing tetramethyldiamidodiphenyl carbinol with *o*-toluidine *p*-sulphonic acid, a leuco product is obtained having the formula—

$$[1.2.4.5]CH_3.NH_2.SO_3H.C_6H_2.CH\{C_6H_4N(CH_3)_2\}_2.$$

By employing the process described in Eng. Pat. 26,139 of 1896 (this Journal, 1897, 907), the free amido group is replaced by the sulphinic acid group. On oxidation, this is converted into the sulphonic acid group, and at the same time the leuco compound is converted into the dyestuff. 13·9 kilos. of the leuco compound obtained by condensing tetramethyldiamidodiphenyl carbinol with *o*-toluidine-*p*-sulphonic acid in a dilute sulphuric acid solution, are dissolved in 500 kilos. of a 20 per cent. sulphuric acid solution, and diazotised at a low temperature with 7 kilos. of sodium nitrite. The diazo solution is saturated with sulphur dioxide, and treated with 25 kilos. of copper powder as a moist paste. The solution is then filtered, air is passed through the liquid, and, after liming, the calcium salt is converted into the sodium salt, the solution of which is evaporated. Of this salt, 53·2 kilos. are dissolved in 200 litres of a 50 per cent. acetic acid solution together with 10 kilos. of sulphuric acid, and oxidised by slowly adding 180 kilos. of a 10 per cent. manganese dioxide paste and 50 kilos. of a 20 per cent. sulphuric acid. Under constant stirring the temperature is gradually raised to 50° C., and after some hours the resulting deep blue solution is neutralised by sodium carbonate, filtered, and concentrated. On salting out, the dyestuff precipitates in small bronzy scales. The dyestuffs obtained according to this method give blue to greenish-blue shades fast to alkalis and light, and dye evenly.—T. A. L.

*Colouring Matters [Blue-Black], Impts. in the Production of.* A. Ashworth and J. Bürger, Bury. Eng. Pat. 22,677, Oct. 1, 1897.

Amido-azo compounds of the naphthalene series are diazotised and combined with β-oxynaphthoic acid [m.p. 216° C.], yielding dyestuffs which produce shades on chrome-mordanted wool very fast to soap and light. For example, β-naphthylamine disulphonic acid G is combined with α-naphthylamine, and 50 kilos. of the disulphonaphthalene-azo-α-naphthylamine so obtained are diazotised with 7 kilos. of sodium nitrite and the necessary quantity of hydrochloric acid. The compound so formed is run into an alkaline solution of 19 kilos. of β-oxynaphthoic acid. When the reaction is complete, the new product is salted out, filter-pressed, and dried. It gives black shades on chrome-mordanted or on unmordanted wool.—T. A. L.

*Colouring Matters [Jet Black], Impts. in the Production of.* A. Ashworth and J. Bürger, Bury. Eng. Pat. 23,816, Oct. 16, 1897.

An extension of the foregoing patent, consisting in the substitution of α-oxynaphthoic acid for the β-oxynaphthoic acid in the process already described. The compounds obtained from the α-derivative are said to be more soluble in hot dilute acids, and to give greater depth of shades.
—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Mercerised Cotton, Structure and Properties of.*
E. Hanausek. Dingler's Polyt. J. 1897, **306**, 19—22.

The author has compared microscopically the structure and properties of cotton mercerised according to the process of Thomas and Prevost, with those of ordinary cotton. Fibres taken from cotton yarn which has been mercerised under tension appear stretched, smooth, occasionally knobby or irregular in outline, and usually straight, although the twisted appearance of ordinary cotton may sometimes be observed to a slight extent. The fibres from the outer layers of the yarn exhibit longitudinal pleats, whilst the inner fibres frequently show transverse folds. On the surface of the fibre may often be distinguished an intermittent double longitudinal stripe and occasional granular markings (Fig. 2). The central canal in many places is very narrow, or has disappeared; in other portions it has become wider, and is apparently filled with a granular mass (Fig. 3).

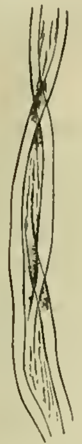


*Fig. 1.*

Cotton fibre, not mercerised.

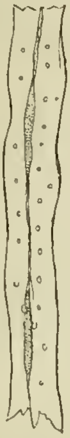


*Fig. 2.*

Mercerised cotton fibre, showing granular markings on surface.

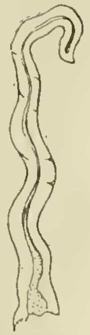


*Fig. 3.*

Mercerised cotton fibre, showing granular contents of central canal.

When mercerised cotton is treated with ammoniacal copper oxide solution, a slight swelling takes place, the central canal usually widens, and the cell wall shows longitudinal striations; but the fibre does not become swollen and constricted in the manner of ordinary cotton, nor are portions of the cuticle dissolved away (Figs. 4 and 5).

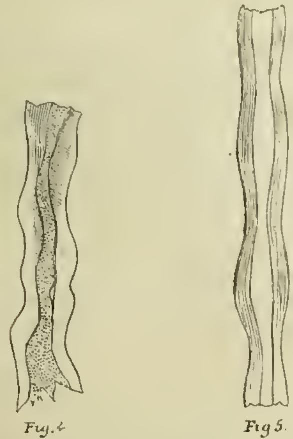


*Fig. 4.* *Fig. 5.*

Mercerised cotton fibres treated with ammoniacal copper oxide. Cell wall showing longitudinal striations.

Mercerised cotton gives a blue coloration with iodine solution and sulphuric acid.

It is found that cotton treated by the process mentioned, retains its silky lustre and scroop, even on long steeping in cold water. The cost of the process at present is 2 marks per kilo. of cotton; and as the strength of the fibre is considerably increased by mercerising, cotton thus treated should find extended use in the manufacture of durable articles of wearing apparel.—R. B. B.

*Animal Textile Materials, Method of Imparting a Silk-like Lustre and Handle to.* Leipziger Färber- u. Zeugdr. Zeit. **46**, [10], 422.

According to the German patent of J. G. Koethe (No. 93,107), the material is treated in an aqueous solution of 5—7·5 per cent. of bromine on the weight of the material, and in this bath the goods are worked for half an hour at 30°—35° C., then rinsed and dyed.

After dyeing, a treatment follows with soap and acid, as in the already known chlorination process (this Journal, 1897, 532), in order to intensify the silk-like properties.

To reduce the cost, the quantity of bromine may be decreased, and an addition to the bath made, after the bromine is all taken up, of $\frac{1}{4}$—$\frac{1}{2}$ per cent. of bleaching-powder solution. For "Mélanges" (mixtures of dyed and undyed wool), bromine alone should be used, since in many cases its destructive action on the dyed colours is less than that of chlorine. It is also stated that bromine does not, like chlorine, impart a yellow tint to the undyed wool.

—R. B. B.

### PATENT.

*Pearl Hardening or Satin Spar* [*Finishing Textile Fabrics*], *Impts. in the Manufacture of.* W. Brothers and W. M. Brothers, Rawtenstall, Lancashire. Eng. Pat. 1577, Jan. 20, 1897.

*See under* XIX., *page* 65.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Sponges, Bleaching of.* Rev. Chim. Ind. 1897, **8**, 337.

Sponges are usually bleached by immersion in several baths of 4 per cent. sulphurous acid until the desired tint is obtained: but hydrogen peroxide, or a 0·5 or 1 per cent. solution of potassium permanganate, is quicker in its action. In the latter case they become stained brown by a deposit of oxide, which is removed by sulphurous acid or a mixture of sodium sulphite and hydrochloric acid. The preliminary treatment consists in extracting all soluble matter from the sponges by soaking them for five or six days in cold water, followed by a bath of 5 per cent. HCl to dissolve calcium salts, and then 24 hours' washing in running water. After bleaching, they are again well washed. As a final operation, they are lightly tinted with Naphthol Yellow, Orange IV., Phosphine, or Auramine.—F. H. L.

*Azo Colours, Insoluble, on Cotton* [*Calico Printing*]; *Discharging.* P. Wolff. Färber-Zeit. 1897, **8**, [22], 341–344.

H. Schmid effects the discharge of Paranitraniline Red on cotton tissues by means of a mixture of stannous chloride and an organic solvent, as glycerin, acetin, &c. (Eng. Pat. 8860 of 1896; this Journal, 1897, 438). He believes that the latter compounds remove from the fibre the amidonaphthol and *p*-phenylenediamine which result from the destruction of the dyestuff, and which would, if left upon the tissue, become oxidised into resinous compounds and be fixed upon it.

The author considers that the action upon the dye is due primarily to the reducing action exerted by the unusually large proportion of stannous chloride (about 660 grms. per kilo.) employed in the process, and that the organic substances used in conjunction with this salt have no solvent action such as has been supposed.

In view of the fact that the application of a concentrated solution of stannous chloride is attended with grave risk of damage to the fibre, besides being expensive, experiments were made with the stannous salts of organic acids, but none of those tried was found to be sufficiently powerful in its action to replace the chloride. A better result, however, was obtained with the sulphocyanide (Ger. Pat. 94,174). A printing mixture containing (? per kilo.) 250—300 grms. of stannous chloride, and an equivalent amount of ammonium or other suitable sulphocyanide, discharges the red on steaming. By the use of stannous chloride and chromium sulphocyanide, colour discharges may be produced, almost all the dyestuffs which are fixed by the chromium mordant being available for use in this connection: a bright blue is, for instance, obtained by the use of Celestin Blue with such a mixture.

Still better results are, however, obtained with zinc dust and sodium bisulphite, which, on steaming for 20 minutes in an atmosphere as moist as possible, yield a perfect discharge.

The claret shade from α-naphthylamine and β-naphthol, also, which has hitherto been considered to be undischargeable by reducing agents, is completely reduced when similarly acted upon. Colour discharges can be effected by adding to the discharge mixture, Methylene Blue B B for blue, Safranine for pink, and Thioflavin S for yellow, Aniline black being printed for black.

The author considers that the insoluble azo dyestuffs, as they are produced upon cotton, are true colour lakes, the oily compounds which are used in their formation entering into chemical combination with them. He attributes to this the difficulty which is met with in their discharge, as compared with that of the soluble azo dyestuffs.—E. B.

### *Alizarin Nitro Derivatives, Reactions of: their Application in Printing.* J. Brandt. With Report by G. van Caulaert. Bull. Soc. Ind. Mulhouse, 1897, 332—386.

There exist at present three commercial derivatives of alizarin and the purpurins. These are β-nitro alizarin, the ordinary Alizarin Orange, α-nitro-alizarin, which, according to O. Witt, is Alizarin Brown of the B.A.S.F., and β-nitro-flavopurpurin sold as Alizarin Orange G (M. L. and B.). The last of these is similar in reactions and properties to ordinary Alizarin Orange, but dyes somewhat yellower shades. α-Nitro-alizarin, when dyed on aluminium mordant with the addition of a calcium salt, yields browner shades than Alizarin Orange, the colours being bright and very fast to soap. On chromium mordant the shade is similar to that obtained with the purpurins. The steam colours produced with α- and β-nitro-alizarin are duller than the dyed colours, and this may be accounted for by the formation of amido-alizarins due to reduction during steaming. This fact may be made of practical value by printing fabrics dyed with nitro-alizarin with a reducing agent, *e.g.*, a stannous salt, the effect of which is to convert the nitro-alizarin more or less completely into the corresponding amido compound. With β-nitro-alizarin (Alumina mordant) the result is a brown on orange ground; with α-nitro-alizarin (Alumina mordant), deep crimson on orange; and with the latter dyestuff on chromium mordant, a violet on a dull bluish-red ground. β- and α-amido-alizarin form respectively the commercial products Alizarin Maroon (B.A.S.F.) and Alizarin Claret R (M. L. and B).—R. B. B.

### *Tin, Action of Nitric Acid on.* R. Engel. Comptes Rend. **125,** [19], 709—711.

Nitric acid of sp. gr. 1·42, diluted with two volumes of water, dissolves tin in the cold, forming stannous nitrate, which gives a brown precipitate of stannous sulphide with hydrogen sulphide. The strong acid diluted with one volume of water also dissolves tin in the cold, forming stannic nitrate. The acid without any water, acts on the tin, giving a white insoluble powder, which, however, dissolves in water after being freed from acid, and is also stannic nitrate. This stannic nitrate, however, in presence of water (and more rapidly when heated) changes into stannic acid, which slowly changes into metastannic acid. This change is never complete however, even on boiling, but there is always formed parastannic acid, $Sn_5O_{11}H_2, 3H_2O$ (Comptes Rend., 27 Sept. 1897), a body which is also formed from metastannic acid by long boiling with water.—J. T. D.

## PATENTS.

### *Dyeing [Khaki Shades], Impts. in.* E. J. Stewart, London. Eng. Pat. 25,581, Nov. 13, 1896.

When the outer husks of the pods of the cotton plant, hitherto regarded as a waste product, are extracted with water, and the extract filtered, a clear brown liquid is obtained, which, on evaporation, yields a glossy brown residue. This residue, it is claimed, possesses very similar dyeing properties to catechu. To produce a fast Khaki dye, myrabolams and ferrous sulphate are added to a boiling solution of the cotton-pod extract; in this liquid the fabric is steeped for some hours, then rinsed in water, worked in a warm solution of potassium bichromate, washed, and dried. A similar process may also be employed for dyeing with catechu or with the bark of the Babool tree.

Fabrics steeped in a weak hot solution of the cotton pod extract and allowed to oxidise in the air without further treatment, acquire a brown or pinkish-brown colour, which is unaltered by boiling soap solution.—R. B. B.

### *Dyeing Purposes, An Improved Liquid Compound [Coloured Soap] for; also applicable for Detergent, Purifying, and Disinfecting Purposes.* C. W. Anderson, H. Benjamin, and J. Mendess, London. Eng. Pat. 25,592, Nov. 13, 1896.

This patent describes a number of liquid and semi-solid preparations, the common basis of which is an aqueous and alcoholic solution of soft soap. When a substantive aniline dyestuff is dissolved in this solution, the improved dyeing compound is produced. A disinfectant and cleansing liquid results from the addition to the soap solution of sugar and a disinfectant, such as carbolic acid or eucalyptus oil. For the purpose of cleansing metals, glass, &c., an addition is made of emery powder or rouge, and when the mixture consists of the soap solution, sugar, and a perfume, with or without a colouring matter, it is stated to be an excellent shaving soap.—R. B. B.

### *Fast Colours upon Vegetable Fibre, Impts. in the Production of.* H. W. Kearns, Baxenden, and J. Barnes, Accrington. Eng. Pat. 27,742, Dec. 5, 1896.

Compounds of titanium are employed to replace salts of antimony and tin in the fixation of tannic acid on cotton, &c., previous to dyeing with basic colours. The method is the same as that already in use when tartar emetic is the salt employed, and the fixing solution may contain any soluble salt of titanium, *e.g.*, the acid oxalate, acid chloride, or the double oxalate of titanium and potassium. Cotton mordanted with tannin and titanium has a yellow or orange colour, which, of course, influences the final shade, and by printing with basic colours on this mordant, coloured patterns are obtained on a yellow ground.—R. B. B.

### *Dyeing Wool or other Animal Fibres in either the Raw or Manufactured State, Impts. in.* F. A. Gatty and Co., Ltd., and V. H. Gatty, Preston. Eng. Pat. 29,150, Dec. 19, 1896.

Wool or other animal fibre is dyed by fixing upon it basic oxides or salts of iron or chromium, or of both these metals. This is effected by steeping in solutions of soluble salts of the metal or metals, rendered basic by addition of alkali. The fixation of basic oxide or salt may be completed either by leaving the saturated fibre in the wet condition without washing for several hours, or by the action of steam or ammonia, or by two or more of these agencies jointly.
—R. B. B.

### *Printed Fabric, Impts. in, and in Means for Printing the same.* H. H. Lake, London. From G. Frattini, Milan, Italy. Eng. Pat. 29,225, Dec. 19, 1896.

The object of this invention is to print designs on fabrics in such a manner that the colour does not penetrate the fabric, but is printed on one side only, thus rendering it possible to print on the other side the same or different designs in the same or different colours.

The fabric is extended on a table and covered with a stencil plate in which the design is cut. A flat or cylindrical brush bearing the colour is caused to pass lightly backwards and forwards over this plate, thus producing the design on the upper side of the fabric only.

In a modification of the process, the stencil plate is rolled into the form of a cylinder, and the brush mounted on a driving shaft within this cylinder. The fabric in this case receives the design as it passes between a pressure roller and the rotating cylindrical stencil plate.—R. B. B.

### *Printing of Metallic Powders on Textile Fabrics to be subsequently Mercerised; Impts. in and in connection with the.* Salis, Schwabe and Co., Ltd., A. Binz, and R. Boral, Middleton, Lancs. Eng. Pat. 29,504, Dec. 22, 1896.

A reserve, consisting of a drying oil, *e.g.*, linseed oil, gum kauri, turpentine, litharge, a trace of manganese dioxide, and a metallic powder, is printed by means of an ordinary

calico-printing machine on those portions of a fabric to be mercerised which are to remain unaffected by the process. The reserve is neither attacked during mercerising nor removed by subsequent washing, and hence a fabric is produced in which the crinkled portions are coloured. The essential feature of the process is the use of linoleïc acid or its glyceride as a reserve against mercerising agents.
—R. B. B.

*Dyestuffs [Blue] on Fibre, Impts. in the Production of.* H. E. Newton, London. From "The Farbenfabriken vorm. F. Bayer and Co.," Elberfeld, Germany. Eng. Pat. 29,827, Dec. 28, 1896.

A PREVIOUS patent (17,591 of 1896; this Journal, 1897, 675) described the formation on the fibre of chromium lakes of Brilliant Alizarin Blue. This is effected by printing a mixture of an oxyindophenol thiosulphonic acid and chromium acetate together with the weak salt of an alkali, such as sodium acetate or thiosulphate, and subsequently steaming, &c. It has been found, however, that the printing colour will not keep for any length of time, and, according to the present patent, the cloth is first padded with the sodium salt, and afterwards printed with the thickened mixture of chromium acetate and thiosulphonic acid; or the order of the two operations may be reversed. In either case, steaming, "malting," and soaping in the usual manner completes the process.—R. B. B.

*Alpha-Naphthylamine [Bordeaux Colour on Fibre] in an Easily-Diazotisable and Stable Form, Manufacture of.* O. Imray, London. From The Farbwerke, Hoechst a/M., Germany. Eng. Pat. 1320, Jan. 18, 1897.

INSTEAD of using α-naphthylamine hydrochloride for diazotising and combining with β-naphthol on the fibre, the patentees make α-naphthylamine sulphate ground to a fine powder, and use this for the purpose. The product is said to be stable to light and air, and not to suffer from the defects of the hydrochloride.—T. A. L.

*Apparatus for Finishing, Stretching, Mordanting, and Drying Textile Fabrics; Impts. in connection with.* M. Beck, Frankfort-on-Main, and Johnsons and Wilcox, London. Eng. Pat. 5350, Feb. 27, 1897.

IN this invention all or any of these operations are performed with one machine. The fabric is first passed between rollers which are placed in the vessel containing the glossing or mordanting material, and then, directly, by means of rollers fitted with spikes or pins, on to the tenter or stretching frame. Means are provided by which, in case of the rollers in the trough exercising undue tension on the fabric, they yield automatically.

The fabric, after leaving the tenter, is passed, still in the stretched condition, through a bath containing water or dilute acid, and finally through another series of rollers.
—C. M.

*Paranitraniline Red and Related Substances, An Impt. or Impts. in White and Colour Discharge, or Enlevage Printing of Dyed.* H. Züblin and A. Zingg, Fratte di Salerno, Italy. Eng. Pat. 13,088, May 27, 1897.

ACCORDING to this improved method, cotton which has been dyed with Paranitraniline red is impregnated with glucose, by padding in a solution at 25° Tw., dried, and then printed with a strongly alkaline solution of naphthol, phenol, glycerin, or other similar substance containing the hydroxyl group, together with a suitable thickening. After printing and drying, the fabric is steamed, passed through acid, well washed, and soaped. For a coloured discharge, there is added to the printing mixture ground indigo or lead oxide, or a mixture of these two substances, and when the fabric is passed through potassium bichromate solution the resulting discharge colours are blue, yellow, and green respectively.

The advantages claimed for this discharge are that clear whites are obtained without injury to the fibre, and that exceedingly fast discharge colours are produced as described.—R. B. B.

*Mordanting of Fibrous Materials, Impts. in or relating to the; Preparatory to Dyeing such Materials.* H. Seidel, Vienna, Austria. Eng. Pat. 19,005, Aug. 17, 1897.

THE improvement consists in the use, as an assistant to metallic mordants, of the substance salted out from the lyes of sulphite cellulose stuff by salts of the alkali or alkaline earthy metals; or the lye itself may be used for this purpose in a more or less concentrated or purified condition. This substance is claimed to be a useful substitute for tartar or lactic acid in the mordanting of wool with potassium bichromate.—R. B. B.

*Bleaching and Washing Woven Goods in the Piece, Impts. in the Method of and Apparatus for Use in.* W. Hadfield, H. Hadfield, and J. J. Sumner, Furness Vale; and E. K. Dutton and Co., Manchester. Eng. Pat. 29,645, Dec. 24, 1897.

WOVEN cloth, in the rope or open form, is boiled in a kier, and then, passing by means of rollers through hot-water baths, it enters the vessels containing the "chemicking" liquid, *i.e.*, bleaching liquor. Thence, after passing through squeezing bowls, it enters a steam chamber in which an acid vaporiser is fitted. At the bottom of the chamber a steam pipe is covered with an outer earthenware pipe, and on to this an acid, usually acetic acid, is allowed to drop by means of a long tube passing through the top of the chamber. The acid evaporates from the hot pipe, and, mixing with the steam, not only intensifies the bleaching action, but also renders the lime left in the cloth, soluble. The cloth is then passed through a steam chamber with no acid in the vapour, and finally through vats containing cold water, to thoroughly cleanse it.

A hood fitted with a chimney is erected over the steam chambers, for carrying off the chlorine fumes.

The whole operation is continuous, so that the fabric on leaving the kiers is not handled until it has passed through the drying rollers.—C. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Sulphuric Acid: Table of Specific Gravities, Strengths, &c.* A. H. Elliott. Published by The Manufacturing Chemists' Association of the United States. H. Bower, Recording Secretary. Philadelphia.

THE accompanying table is the result of a careful selection of figures from standard authorities, and calculations from those figures have been worked out into forms that are constantly needed in a chemical manufactory. The columns with headings marked "Degrees Baumé," "Specific Gravity," and "Per Cent. of $H_2SO_4$," follow the figures of the table of The Manufacturing Chemists' Association. A comparison of these figures with those of Bineau and Kolb shows them to be the latest and most reliable result. The 66th degree of Baumé* is placed at 1·835 sp. gr., not because this is the highest attainable strength, but because this is the specific gravity of the acid sold and branded "66° Oil of Vitriol" in commerce. Acid of a higher specific gravity is difficult to prepare, and is unnecessary for the ordinary uses to which oil of vitriol is put. In other words, "Oil of Vitriol" contains 93½ per cent. of monohydrated acid ($H_2SO_4$), and this is now a settled question among chemical manufacturers.

Starting with the above data, the weight of a cubic foot of acid at the various densities has been calculated, and the figures worked out in the other columns. The nearest degrees of Twaddell's hydrometer corresponding to the various specific gravities have also been added. This latter hydrometer is a much more reliable instrument to place in the hands of a workman than that of Baumé, because the degrees are more easily read, and a difference of a quarter, or even a half degree, does not make as much difference as the same error on Baumé's scale. But since it is a trade custom to use the latter hydrometer, a definite understanding of its value is very important.

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* The total number of degrees of this areometer would be 145. (N = 145·04.)

The boiling points given are according to the figures of Dalton. The specific gravity of sulphuric acid in the accompanying table is that obtained at a temperature of 60° F. (15·5 C.). The number of degrees Fahrenheit causing a rise or fall of 1° Baumé, is given in the last column. Thus, a rise or fall of 40° F. in the temperature of acid ranging from 60°—66° Baumé (at 60° F.), causes a fall or rise of 1° Baumé. In other words, acid of 63° Baumé at 60° F. will stand 62° at 100° F., or it will stand at 64° at a temperature of 20° F., and so on for each set of figures given in the last column.

The term O.V. in the table, is the contraction of "Oil of Vitriol," or acid of 93·5 per cent. of $H_2SO_4$. The term 60° acid means acid containing 77·6 per cent. of $H_2SO_4$.

See also Lunge's "Sulphuric Acid and Alkali," Vol. I., 2nd edition, 115; also 119—124.

| Degrees Baumé. | Specific Gravity. | Per Cent. $H_2SO_4$ | Per Cent. O.V. | Weight of Cubic Foot. | Per Cent. 60 Acid. | Pounds of O.V. in Cubic Foot. | Pounds of 60° Acid in Cubic Foot. | Nearest Degree of Twaddell. | Boiling Point, Degrees C. | Boiling Point, Degrees F. | Correction for Temperature. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1·005 | 0·93 | 1 | 62·8 | 0·73 | 0·62 | 0·45 | 1·0 | .. | .. | .. |
| 2 | 1·011 | 1·87 | 2 | 63·2 | 1·46 | 1·26 | 0·92 | 2·0 | .. | .. | .. |
| 3 | 1·023 | 3·71 | 4 | 63·9 | 2·92 | 2·55 | 1·86 | 4·5 | .. | .. | .. |
| 4 | 1·029 | 4·67 | 5 | 64·3 | 3·64 | 3·21 | 2·33 | 5·7 | .. | .. | .. |
| 5 | 1·036 | 5·61 | 6 | 64·7 | 4·37 | 3·88 | 2·83 | 7·0 | .. | .. | .. |
| 6 | 1·043 | 6·54 | 7 | 65·2 | 5·10 | 4·56 | 3·32 | 8·5 | .. | .. | .. |
| 7 | 1·050 | 7·48 | 8 | 65·6 | 5·83 | 5·25 | 3·82 | 10·0 | .. | .. | .. |
| 8 | 1·057 | 8·41 | 9 | 66·0 | 6·56 | 5·94 | 4·33 | 11·5 | .. | .. | .. |
| 9 | 1·064 | 9·35 | 10 | 66·5 | 7·29 | 6·65 | 4·85 | 13·0 | .. | .. | .. |
| 10 | 1·071 | 10·28 | 11 | 66·9 | 13·16 | 7·36 | 8·80 | 14·0 | .. | .. | 43° |
| 11 | 1·086 | 12·15 | 13 | 67·8 | 15·45 | 8·81 | 10·47 | 17·0 | .. | .. | .. |
| 12 | 1·093 | 13·09 | 14 | 68·3 | 16·75 | 9·56 | 11·44 | 18·5 | .. | .. | .. |
| 13 | 1·100 | 14·02 | 15 | 68·7 | 17·94 | 10·30 | 12·32 | 20·0 | 103 | 217 | .. |
| 14 | 1·107 | 14·96 | 16 | 69·2 | 19·15 | 11·07 | 13·25 | 21·5 | .. | .. | .. |
| 15 | 1·114 | 15·89 | 17 | 69·6 | 20·34 | 11·83 | 14·15 | 23·0 | .. | .. | .. |
| 16 | 1·122 | 16·83 | 18 | 70·1 | 21·54 | 12·62 | 15·10 | 24·5 | .. | .. | .. |
| 17 | 1·136 | 18·70 | 20 | 71·0 | 23·93 | 14·20 | 16·99 | 27·0 | .. | .. | .. |
| 18 | 1·143 | 19·63 | 21 | 71·4 | 25·12 | 14·99 | 17·93 | 28·5 | .. | .. | .. |
| 19 | 1·150 | 20·57 | 22 | 71·8 | 26·33 | 15·79 | 18·90 | 30·0 | .. | .. | .. |
| 20 | 1·158 | 21·50 | 23 | 72·3 | 27·52 | 16·63 | 19·89 | 31·5 | .. | .. | 30° |
| 21 | 1·172 | 23·37 | 25 | 73·2 | 29·91 | 18·30 | 21·89 | 34·5 | .. | .. | .. |
| 22 | 1·179 | 24·31 | 26 | 73·7 | 31·11 | 19·16 | 22·93 | 35·7 | .. | .. | .. |
| 23 | 1·186 | 25·24 | 27 | 74·1 | 32·31 | 20·00 | 23·94 | 37·0 | .. | .. | .. |
| 24 | 1·201 | 27·11 | 29 | 75·0 | 34·70 | 21·75 | 26·02 | 40·0 | 107 | 224 | .. |
| 25 | 1·208 | 28·05 | 30 | 75·5 | 35·90 | 22·65 | 27·10 | 41·5 | .. | .. | .. |
| 26 | 1·216 | 29·98 | 31 | 76·0 | 37·15 | 23·56 | 28·23 | 43·0 | .. | .. | .. |
| 27 | 1·231 | 30·85 | 33 | 76·9 | 39·55 | 25·37 | 30·41 | 46·0 | .. | .. | .. |
| 28 | 1·238 | 31·79 | 34 | 77·4 | 40·75 | 26·31 | 31·54 | 47·5 | .. | .. | .. |
| 29 | 1·254 | 33·66 | 36 | 78·4 | 43·15 | 28·22 | 33·83 | 51·0 | .. | .. | .. |
| 30 | 1·262 | 34·59 | 37 | 78·9 | 44·34 | 29·19 | 34·98 | 52·5 | .. | .. | 30° |
| 31 | 1·269 | 35·53 | 38 | 79·3 | 45·55 | 30·13 | 36·12 | 54·0 | .. | .. | .. |
| 32 | 1·285 | 37·40 | 40 | 80·3 | 47·95 | 33·12 | 38·50 | 57·0 | .. | .. | .. |
| 33 | 1·293 | 38·33 | 41 | 80·8 | 49·14 | 33·13 | 39·70 | 58·5 | 115 | 239 | .. |
| 34 | 1·309 | 40·20 | 43 | 81·8 | 51·54 | 35·17 | 42·16 | 62·0 | .. | .. | .. |
| 35 | 1·317 | 41·14 | 44 | 82·3 | 52·74 | 36·21 | 43·40 | 63·5 | .. | .. | .. |
| 36 | 1·334 | 43·01 | 46 | 83·4 | 55·14 | 38·36 | 45·98 | 67·0 | .. | .. | .. |
| 37 | 1·342 | 43·94 | 47 | 83·9 | 56·33 | 39·43 | 47·26 | 68·5 | .. | .. | .. |
| 38 | 1·359 | 45·81 | 49 | 84·9 | 58·73 | 40·75 | 49·86 | 72·0 | .. | .. | .. |
| 39 | 1·368 | 46·75 | 50 | 85·5 | 59·93 | 42·75 | 51·24 | 73·5 | .. | .. | .. |
| 40 | 1·386 | 48·62 | 52 | 86·6 | 62·33 | 45·03 | 53·98 | 77·0 | .. | .. | 31° |
| 41 | 1·395 | 49·55 | 53 | 87·2 | 63·52 | 46·21 | 55·39 | 79·0 | .. | .. | .. |
| 42 | 1·413 | 51·42 | 55 | 88·3 | 65·92 | 48·56 | 58·21 | 82·5 | 128 | 262 | .. |
| 43 | 1·422 | 52·36 | 56 | 88·9 | 67·12 | 49·78 | 59·67 | 84·5 | .. | .. | .. |
| 44 | 1·441 | 54·23 | 58 | 90·0 | 69·52 | 52·20 | 62·57 | 88·0 | .. | .. | .. |
| 45 | 1·451 | 55·16 | 59 | 90·7 | 70·71 | 53·51 | 64·13 | 90·0 | .. | .. | .. |
| 46 | 1·470 | 57·03 | 61 | 91·9 | 73·11 | 56·06 | 67·19 | 94·0 | .. | .. | .. |
| 47 | 1·480 | 57·97 | 62 | 92·5 | 74·32 | 57·35 | 68·74 | 96·0 | .. | .. | .. |
| 48 | 1·500 | 59·84 | 64 | 93·7 | 76·71 | 59·97 | 71·88 | 100·0 | .. | .. | .. |
| 49 | 1·510 | 60·77 | 65 | 94·4 | 77·91 | 61·36 | 73·55 | 102·0 | .. | .. | .. |
| 50 | 1·531 | 62·64 | 67 | 95·7 | 80·31 | 64·12 | 76·85 | 106·0 | 144 | 292 | 38° |
| 51 | 1·541 | 63·58 | 68 | 96·3 | 81·51 | 65·48 | 78·49 | 108·0 | .. | .. | .. |
| 52 | 1·562 | 65·45 | 70 | 97·6 | 83·91 | 68·32 | 81·89 | 112·5 | .. | .. | .. |
| 53 | 1·573 | 66·38 | 71 | 98·3 | 85·10 | 69·69 | 83·65 | 114·5 | .. | .. | .. |
| 54 | 1·594 | 68·25 | 73 | 99·6 | 87·50 | 72·71 | 87·15 | 119·0 | .. | .. | .. |
| 55 | 1·616 | 70·12 | 75 | 101·0 | 89·89 | 76·05 | 90·79 | 123·0 | .. | .. | .. |
| 56 | 1·627 | 71·06 | 76 | 101·7 | 91·10 | 77·29 | 92·65 | 125·5 | .. | .. | .. |
| 57 | 1·650 | 72·93 | 78 | 103·1 | 93·50 | 80·42 | 96·40 | 130·0 | 177 | 350 | .. |
| 58 | 1·661 | 73·86 | 79 | 103·8 | 94·69 | 82·00 | 98·29 | 132·0 | 180 | 354 | .. |
| 59 | 1·683 | 75·73 | 81 | 105·2 | 97·08 | 85·21 | 102·13 | 136·5 | 186 | 367 | .. |
| 60 | 1·705 | 77·60 | 83 | 106·5 | 100·00 | 88·39 | 106·50 | 141·0 | 192 | 378 | 40 |
| 61 | 1·727 | 79·47 | 85 | 107·9 | 101·88 | 91·71 | 109·93 | 145·5 | 200 | 392 | .. |
| 62 | 1·747 | 81·34 | 87 | 109·2 | 104·28 | 95·00 | 113·87 | 149·5 | 205 | 401 | .. |
| 63 | 1·767 | 83·21 | 89 | 110·4 | 106·68 | 98·25 | 117·77 | 153·5 | 217 | 422 | .. |
| 64 | 1·793 | 86·02 | 92 | 112·0 | 110·28 | 103·04 | 123·51 | 158·5 | 230 | 446 | .. |
| 65 | 1·814 | 88·82 | 95 | 113·4 | 113·87 | 107·73 | 129·13 | 163·0 | 249 | 480 | .. |
| 66 | 1·835 | 93·50 | 100 | 114·7 | 119·87 | 114·70 | 137·49 | 167·0 | 272 | 523 | 40° |

*Ammonia-Soda Process, Influence of the Addition of Salt whilst Carbonating.* K. W. Jurisch. Zeits. f. angew. Chem. 1897, [21], 688—691.

THE origin of the idea of adding to the ammoniacal brine, whilst carbonating, additional salt, is not very clearly traceable. Gossage mentions a "further addition of common salt" in his patent of 1854; but it would seem, from a study of his apparatus, that his addition was made before carbonating had commenced; and the definite recommendation to add solid salt whilst carbonating, seems to have been made by Solvay. The author has examined the influence of this addition of salt on the results of the process, making use of a decinormal silver nitrate solution, a normal soda solution, and a solution of acid of corresponding strength. The carbonated liquor, after withdrawal from the tower, was allowed to settle in a cylindrical test glass, and samples of the clear liquor tested. The chloride content, in those cases in which no addition of salt was made, varied from 42 to 49 c.c. of the silver solution per c.c., with an average of 47·8; while from those towers working with salt-addition, the figures varied from 54 to 63 c.c., with an average of 57. In the case of a British works in which the addition of salt was not made, the original ammoniacal brine required for neutralisation, 37·3 c.c. of the sulphuric acid solution per 10 c.c., and after carbonating, 11·3 c.c.—a fall of nearly 70 per cent.; whilst the same works, later, when salt was added, produced after carbonating, from a brine neutralising 44·2 c.c. of acid, a liquor needing only 6·6 c.c.—a fall of 85 per cent. But the utilisation of the ammonia can under certain conditions be carried very far without adding salt, as the following figures from an Austrian works show :—

| ——— | Salt added. | No Salt added. |
|---|---|---|
| Ammoniacal brine :— | | |
| Free ammonia | 44 to 46 c.c. | 43 to 44 c.c. |
| Tower liquor :— | | |
| Free ammonia | 5 to 9 c.c. | 6 to 11 c.c. |
| Total ammonia by distillation. | 39 to 42 c.c. | 39 to 43 c.c. |
| Chlorine | 56 to 58 c.c. | 43 to 47 c.c. |

Increasing the height of the towers seems to render the addition of salt superfluous: the latest American work has been done without addition of salt. A good ammoniacal brine should have a specific gravity of 1·18 to 1·19, contain free ammonia in 10 c.c. corresponding to 43 or 44 c.c. of normal acid, total ammonia equivalent to 44 or 45 c.c. of acid, and salt in 1 c.c. corresponding to 44 or 45 c.c. of silver solution. Formerly, with the view of preventing loss of ammonia, brines were worked with a smaller proportion of ammonia (42·5 to 37·5 or even 30 c.c. of acid); but there was always a correspondingly small yield of bicarbonate, and where salt addition is to be made during the carbonating, the proportion of ammonia must not fall below the figure given; indeed, it may with advantage be increased by about 2 to 5 per cent. of the total quantity. It is thus clear that the addition of salt during carbonating necessitates an alteration in the composition of the ammoniacal brine. A continuation of the paper is to follow.—J. T. D.

*Nitrous Acid, A New Gas-Volumetric Method for the Estimation of, and other Substances which can be oxidised by Hydrogen Peroxide.* E. Riegler. Zeits. Anal. Chem. 1897, **36**, 665.

*See under* XXIII., *page* 71.

## PATENTS.

*Air and Water Vapour, Mixture of; Impts. in or relating to the Production of a; especially applicable for Gas Producers.* L. Mond, Northwich. Eng. Pat. 28,588, Dec. 14, 1896.

*See under* II., *page* 34.

*Ammonia, Impts. in Process of and Apparatus for Distilling.* H. J. Krebs, Wilmington, U.S.A. Eng. Pat. 17,313, July 22, 1897.

This is a process for obtaining anhydrous liquefied ammonia by a primary, and a secondary, or refining, distillation and condensation, the latter being effected at a lower temperature than the former. The residues of the primary distillation are returned to the still as steam; and in the refining distillation the residue is redistilled. The distillations may be carried out by the aid of pumps at relatively low temperatures and pressures; or preferably without pumps, and with these increased. The process, taken apart from any special apparatus employed, consists in gradually heating *aqua ammoniæ* until the ammonia is evolved, then vaporising the residue and returning the vapour through the solution under treatment, thus raising it to the desired temperature, with the effect of separating ammonia, the further separation of the ammonia from the aqueous vapour being effected by cooling, the reduction of the former to a liquid being by means of cooling and pressure. "The liquid thus produced is refined or further purified by subjecting it to a much lower degree of heat, condensing the vapours so produced, subjecting the residuum of this distillation to a further distillation, and cooling and returning the vapours thus produced to the mass of impure ammonia."—E. S.

*Common Salt and other like Salts, An Improved Process and Apparatus for obtaining, from their Solutions.* E. A. Goddin, Stoke Newington, and R. Lees, London. Eng. Pat. 28,294, Dec. 10, 1896.

The process consists in alternate refrigeration and concentration by heat of saline solutions, so conducted that, by means of heat-interchanging apparatus, the hot solution is cooled on its way to the refrigerator, while the cold solution is heated in its course back to the boiler, the process being thus made continuous. In the case of sodium chloride, the hydrated salt, with 2 atoms of water of crystallisation, is deposited in the cooler, and is subsequently dried by waste heat. A close boiler is used, and the steam under pressure is utilised in working the refrigerating plant as well as otherwise. The elaborate apparatus employed, is described in detail and illustrated.—E. S.

*Bay Salt, An Impt. in the Manufacture of.* A. MacNab, London. From W. A. Willock, Vizagapatam, Madras, India. Eng. Pat. 24,524, Oct. 22, 1897.

Bay salt is soaked in strong brine, containing a small proportion of sodium carbonate, for about a day, and is then washed in brine and dried. The deliquescent magnesium and calcium chlorides present are thus changed into carbonates, which are washed out by the brine.—E. S.

*Sulphuric Acid and other Liquids, Impts. in and connected with Apparatus for Concentrating.* T. G. Webb, Manchester. Eng. Pat. 29,884, Dec. 29, 1896.

The apparatus is of the type described in Eng. Pat. 2343, 1891 (this Journal, 1891, 768), in which the concentrating vessels are arranged on a stepped surface, and the process is continuous. But instead of heating at the higher end of the apparatus, and using coal, coke, or coal-gas, the heat is applied at the lower end, and by means of water-gas. Side chambers are formed in the lower part of the apparatus, in communication with a gas-flue leading to the supply-pipe of a water-gas generator and controlled by a valve. Air channels are also formed in connection with the gas-flue, and the air mixes with the water-gas "and thus creates combustion." slots or openings being provided for the passage of the hot gases into the heating chamber.—E. S.

*Concentrating Sulphuric Acid or the like, Impts. in Methods and Apparatus for.* G. D. Howard, London. From H. Howard, Boston, U.S.A. Eng. Pat. 1762, Jan. 22, 1897.

The invention consists in the automatic regulation of the flow of sulphuric acid through the concentrating apparatus, by causing the density of the distillate to influence a float, which, through suitable mechanism, controls the flow of acid from the supply vessel. The acid is siphoned from a reservoir to the supply vessel, which is horizontally divided into two chambers, communication between which is controlled through two ports by frusto-conical valves reversely arranged, and connected by stems to a balanced lever. The supply vessel is kept substantially full. The acid flows through the ports into the lower compartment, whence the still is supplied. A portion of the distillate from the sulphuric acid is received in a U-shaped tube, in one arm of which a float is arranged, connected by a stiff wire to the outer end of the lever which controls the supply vessel. As the density of the distillate increases, the float rises, and through the action of the lever on the ports, the flow of acid is increased, or *vice versâ*, if the distillate becomes weaker. The apparatus is shown in connection with a platinum concentrating vessel.—E. S.

*Chlorine, Impts. in the [Electrolytic] Manufacture of.* E. J. Hunt, Wednesbury, and Ed. F. Watson, Wolverhampton. Eng. Pat. 29,066, Dec. 18, 1896.

The crude ferrous chloride resulting from the pickling of iron is treated for some days in a wooden tank with scrap iron, and is then heated with more scrap iron in iron tanks, until the ferric chloride present is reduced to ferrous chloride, and the free acid is neutralised. The solution is then concentrated to a density of about 1·4, when it is ready for use. The electrolysing tank is preferably of pitch pipe, in which are secured porous compartments, each formed of two slabs of fire-brick, the edges of which are packed water-tight in grooves formed by battens on the sides of the tank, the bottoms being similarly packed on a false bottom formed of wood. A carbon plate is fixed in each compartment, which is closed at the top by a gas-tight lead cap. Each such anode compartment is charged with a saturated solution of common salt, potassium chloride, or earthy metal chloride. Strips of iron are run around the tops of the sides and ends of the tanks, the strips of the last tank of a series being connected to the negative terminal of a dynamo, the other connection being to the carbons. Rods of iron, bolted at each end to the iron strips, and placed between the porous compartments, have depending into the ferrous chloride solution with which the spaces between the porous compartments are filled, strips of thin sheet iron which form the cathode of the arrangement, and which receive a deposit of pure iron. The carbon plates are connected together in series, and each porous compartment communicates with a pipe carrying the chlorine evolved, to be utilised in making bleaching powder or otherwise. It is necessary that the density of the ferrous chloride solution should not fall below 1·25.—E. S.

*Barium Carbonate, An Improved Process for obtaining, from Native Sulphate of Barium.* H. H. Lake, London. From J. S. Levi and Co., Turin, Italy. Eng. Pat. 1218, Jan. 16, 1897.

Barium sulphide is obtained by heating the sulphate with a reducing agent, such as carbon, iron, or manganese slag, in a reverberatory furnace. The hydrated barium sulphide obtained from the product by lixiviation and crystallisation, is dissolved in water, and precipitated by sodium or potassium carbonate, to obtain barium carbonate; and from the residual liquor, by concentration, an alkali sulphide.
—E. S.

*Caustic Soda or Caustic Potash, Impts. in the Manufacture of, from Carbonate of Soda or Carbonate of Potash.* T. Ryan, jun., A. Dyson, and The United Alkali Co., Ltd., Liverpool. Eng. Pat. 4227, Feb. 16, 1897.

Ferric oxide—preferably roasted iron pyrites after extraction of the copper—is heated in a Mactear furnace, and a concentrated solution of sodium or potassium carbonate is gradually added, with constant stirring. The dry mass is preferably transferred to another furnace to be highly heated, until an alkali ferrite is formed; or both stages of the process may be conducted in one furnace. The product is lixiviated to obtain the caustic alkali, and the residue of ferric oxide is re-used. Ordinary vat-liquor, containing sulphide, may be used in the process. Reference is made to Loewig's Eng. Pat. 4364, 1882.—E. S.

*Silico-Fluorides and Boro Fluorides, Method of Transforming into corresponding Fluorides or Fluorine Compounds.* J. Reich, Vienna II., Austria. Eng. Pat. 21,073, Sept. 11, 1897.

If one molecule of an alkali silico-fluoride be mixed with three molecules of an alkaline earth (as lime, for instance), the mixture, heated to low redness, becomes incandescent, without evolution of gas. The external heating must then be discontinued to avoid fusion. Preferably, more than the theoretical proportion of the earth is used; or a diluent, such as felspar, or alkali-earth fluorides, may be added to the mixture. On lixiviating the mass after the reaction, the alkali fluoride is obtained in solution, which may be treated, if desired, with lime to obtain caustic alkali. When the latter is to be produced however, it is better to employ a mixture of, for instance, lime, calcium carbonate, and carbon with the silico-fluoride; the mass, after sufficient heating, then yields caustic alkali at once on lixiviating. Carbonates, nitrates, and other oxysalts of the alkaline earths, as they do not react by themselves with a silico-fluoride, can only be used together with the oxides of the alkaline earths. The appearance of incandescence on heating the first-described mixture, is promoted by the addition of alumina or silica, which are stated to act as "heat transporters." The same reactions take place on substituting a boro-fluoride for a silico-fluoride in the described mixtures.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

### PATENTS.

*Washing and Cleansing Glass, and Painted and Varnished Surfaces; A Solution for.* T. Arthur, Dunedin, New Zealand. Eng. Pat. 23,110, Oct. 8, 1897.

The solution consists of dilute hydrochloric acid, in which a little common salt is dissolved.—H. H. B. S.

*Polishing Glass, Improved Method of, and Composition for.* B. Swinden, Sheffield. Eng. Pat. 24,317, Oct. 21, 1897.

The method consists in the application of a mixture of hydrofluoric, sulphuric, and hydrochloric acids, mixed in various proportions, according to the nature of the glass.
—H. H. B. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Continuous Kilns for Burning Bricks, Tiles, Sanitary Ware, and other Clay Goods, and for Salt Glazing; Certain Impts. in.* P. Wood, West Bromwich, Stafford. Eng. Pat. 27,406, Dec. 2, 1896.

This is an arrangement of the flues of a continuous kiln, so that any one or more of the kilns of the series may be fired, and the heat regulated as required. A main flue, passing under or through each kiln of the series, serves to convey heat from the producers to, or receive heat from, any one kiln, and converging from this main flue are smaller auxiliary flues controlled by dampers. These in turn are connected with the usual open flue leading to the chimneys, the connections of these being likewise controlled by dampers. There may also be a branch flue for conveying heat from the main flue to the drying sheds.
—H. H. B. S.

*Fireproof Protecting Compound.* S. Jabłónski, Warsaw, Russia. Eng. Pat. 13,065, May 27, 1897.

The compound consists of 280 parts of water-glass, 2 parts of spirit varnish, and 96 parts of a "filling" composition, which may be made of 20 parts of powdered graphite, 50 parts of barium sulphate, and 26 parts of clay. Other substances, however, may be used for the "filling," such as peat-ashes, bone-black, chalk, gypsum, earth, &c. Before the compound is applied, the articles are coated with a mixture of water-glass and potassium carbonate.
—H. H. B. S.

## X.—METALLURGY.

*Platinum, New Process for Attacking. Preparation of Potassium and Ammonium Bromoplatinates.* G. Méker. Comptes Rend. 1897, **125**, [24], 1029—1032.

Platinum resists the action of fused ammonium sulphate; the alkali chlorides and bromides attack it between 250° and 350° C., but only to an infinitesimal extent. With a mixture of these salts, however, and especially with ammonium sulphate and bromide, or ammonium sulphate and potassium bromide, the corrosion is very rapid. When one of these mixtures is heated in a platinum vessel for some moments, the surface of contact becomes bright red, owing to the formation of ammonium bromoplatinate. This salt may be prepared by introducing platinum black or thin foil into a fused mixture of 6 parts of ammonium sulphate and 1 part of potassium bromide, the temperature being kept at about 330° C., washing with cold water, and recrystallising from hot water.

If the bromide be replaced by chloride, some chloroplatinate is formed, mixed with ammonium compounds. With potassium bisulphate instead of ammonium sulphate, a bad yield of potassium bromoplatinate is obtained. The iodoplatinate could not be obtained in this way.—A. C. W.

*Smelting at Broken Hill.* H. Watson. Proc. Inst. Mining and Metallurgy, Dec. 1897.

The ores smelted at Broken Hill are: (1) *Oxidised lead ores*, consisting chiefly of lead carbonate (cerussite); (2) *Manganiferous iron ores*; (3) *Kaolin*, containing too much lead for the leaching process; (4) *Silicious iron ores*, also containing too much lead for leaching. Crude sulphide ore is also added, generally one-sixth of the weight of lead ore. The other constituents of the smelting charge are: *Lead*, precipitated by scrap-iron from its solution as chloride in the wash water from the roasted ore of the leaching works; *Lead carbonate*, precipitated by $Na_2CO_3$ from the hyposulphite solution; *Limestone*, for fluxing, and some old slag.

*The Fluxes.*—The fluxes are: (1) *Manganiferous ironstone*, containing lead and silver; (2) *Silicious ironstone*, also containing manganese, lead, and silver; both obtained from the mines; (3) *Limestone*, purchased.

*Analyses.*

| — | Manganiferous Ironstone. | Silicious Ironstone. |
|---|---|---|
| Pb | 18·0 per cent. | 13·0 per cent. |
| Fe | 15·0 ,, | 14·7 ,, |
| Mn | 21·6 ,, | 16·0 ,, |
| $Al_2O_3$ | 4·4 ,, | 6·2 ,, |
| Zn | 1·8 ,, | 1·7 ,, |
| $SiO_2$ | .. .. | 27·5 ,, |
| Insoluble matter | 15·3 per cent. | 32·0 ,, |
| Ag | 5 oz. per ton | 11 oz. per ton. |

*The limestone* contains 5 to 7 per cent. of $MgCO_3$, and 6 to 7 per cent. of insoluble matter. The limit for $MgCO_3$ is 7·5 per cent., and for anything over this a deduction is made from the price per ton. The limestone is brought to the works by tramway from the quarries, about 40 miles distant.

The objection to much magnesia is that it diminishes the fusibility of the slag to a greater extent than lime, 20 parts of MgO being equal to 28 of CaO in this respect.

A rather thin slag is required for ores containing much alumina and zinc, and the presence of manganese increases the fusibility of the slag. The best slag is a singulo-silicate, or one between a singulo- and a bisilicate. The slags formed in the Broken Hill smelting have that constitution, containing:—

| | |
|---|---|
| $SiO_2$ | 38·4 to 42·8 per cent. |
| Fe | 10·0 ,, 13·1 ,, |
| Mn | 9·8 ,, 12·5 ,, |
| CuO | 16·0 ,, 17·5 ,, |
| $Al_2O_3$ | 9·1 ,, 9·2 ,, |
| Silver | 1·3 ,, 1·7 oz. per ton. |

*The Charge.*—The charges are made up on the half ton, and consist of—

| | Lb. |
|---|---|
| Lead ore, oxidised | 150 |
| Manganiferous iron ore | 125 |
| Silicious iron ore | 150 |
| Limestone | 275 |
| Kaolin | 175 |
| Coke | 125 |
| Crude sulphide ore | 25 |
| Precipitated lead | 25 |
| Carbonate of lead, precipitated | 25 |

*Analyses.*

| — | Oxidised Lead Ore. | Kaolin. | Crude Sulphide Ore. |
|---|---|---|---|
| Pb per cent. | 17·0 | 10·0 | 61·5 |
| Cu ,, | Trace | 0·5 | Trace |
| Fe ,, | 8·0 | 10·0 | 2·0 |
| Mn ,, | 7·0 | 7·8 | 1·7 |
| CaO ,, | Trace | Trace | .. |
| $Al_2O_3$ ,, | 8·0 | 8·8 | .. |
| Zn ,, | 2·0 | 2·8 | 8·2 |
| $SiO_2$ ,, | 45·2 | 49·0 | .. |
| Insol. mat. ,, | 60·2 | 63·5 | .. |
| S ,, | .. | .. | 13·3 |
| Ag per ton | 26 oz. | 26 oz. | 24 oz. |

*The Products of Smelting.*—These are *matte* and *base bullion*. The matte varies in composition, but usually contains 25 to 30 per cent. of Cu, 37 per cent. of Pb, and about 300 oz. of Ag per ton. The base bullion contains 300 oz. of Ag per ton. Both products are shipped to Port Pirie for treatment.

The amount of ore smelted in six months is about 135,000 tons, producing 10,000 tons of base bullion, yielding about 4,000,000 oz. of silver, and 3,000 oz. of gold.

*The Furnaces.*—There are 15 rectangular 80-ton furnaces, water-jacketed, each having 11 twyers.

The bullion is tapped periodically, being led over the moulds by an iron spout. The matte runs out into pots with some slag, from which it is separated when cold.

—R. A. P. D.

*Steel. Further Study on the Influence of Heat Treatment and Carbon upon the Solubility of Phosphorus in.* E. D. Campbell and S. C. Babcock. J. Amer. Chem. Soc. 1897, **19**, [10], 786—790.

The authors have continued their earlier work (this Journal, 1896, **15**, 905), employing an accurate Le Chatelier pyrometer with an error of ±5° at 1,150° C. The solvent used for discriminating between soluble and insoluble phosphorus was slightly acid mercuric chloride, which gave practically as good a result after acting for $1\frac{1}{2}$ hour as after 40 hours. 5 grms. of the steel were placed in an Erlenmeyer flask, 35 grms. of powdered $HgCl_2$ were first added, and then 100 c.c. of 2 per cent. hydrochloric acid solution. After standing for $1\frac{1}{2}$ hour with occasional shaking, the solution was filtered by means of a pump, and washed four times with hot water. The phosphorus in the filtrate was determined by concentration in basic ferric acetate (obtained from added ferric chloride), followed by filtration of the precipitate, solution in nitric acid, and precipitation with ammonium molybdate.

Three samples of steel, which had the following compositions respectively, were examined:—(α) C = 0·10, P = 0·119, Mn = 0·484; (β) C = 0·37, P = 0·16, Mn = 0·82; and (γ) C = 1·22, P = 0·098, Mn = 0·78, S = 0·068, and Si = 0·058 per cent. These were heated to various temperatures and quenched, or were annealed at 900°–1,000° C. and cooled slowly, with the following results as regards the solubility of the phosphorus:—

| Temperature of Quenching. | Percentage of Insoluble Phosphorus in Resulting Steel. | | |
|---|---|---|---|
| | (α) | (β) | (γ) |
| 719° | 68·1 | 68·8* | 89·8 |
| 750° | .. | .. | 52·0 |
| 825°–827° | 66·4 | 41·2 | 18·3 |
| 928°–923° | 67·2 | 30·0 | 15·3 |
| 1,028°–1,023° | 72·2 | 30·6 | 16·2 |
| Annealed | 83·2 | 85·6 | 100·0 |

* Quenched at 728° C.

The effect of the carbon is thus very marked, and the existence of phosphorus in at least two forms in steel is thus confirmed. The influence of this element on the physical properties of steel thus depends as much upon the form in which the phosphorus exists as upon its quantity (compare this Journal, 1897, 542).—W. G. M.

[*Gold.*] *The so-called Lode Formations of Hannans, and Telluride Deposits.* H. P. Woodward. Inst. Mining and Met. 1897.

A minute account is given of the geology of the district and the manner of occurrence of the gold. Where rich lodes outcrop, oxidation occurs on the surface and extends downwards to a considerable depth, the gold in the oxidised portion being derived from the decomposition of tellurides. Small decomposed quartz veins, often ferruginous, intersect the lode mass, and here the lode is always richest, the coarsest gold occurring in quartz, whilst in the absence of such veins little or no gold is met with. In prospecting, the best method is to sink shafts to a depth of 100 ft. upon the supposed line of lode. From this, cross-cuts are driven east and west until a lode is cut, when a level should be driven north and south. The quality of the water, however, is bad, containing not only much common salt, but a quantity of magnesium chloride, which causes "sickening" of the mercury.

The ore is troublesome to treat, because, although the gold is free in the decomposed portions of the lode, it contains magnesium silicates and hydrated oxides of iron, which form slimes amounting to 40 per cent. of the entire lode-stuff. Most of the ore as yet has been treated by wet-stamping and amalgamation, and the process has been so far perfected that the tailings contain less than 8 dwt. of gold per ton, which is considered satisfactory when the nature of the ore, its richness, the fine state of the gold, and the bad quality of the water are taken into account. Cyaniding has been tried, but it was found that when the ore was first passed through the amalgamation barrels, it became a dense mass in the leaching vats, and so retarded filtration. It is, however, proposed first to dissolve the fine gold by cyanide, and then to proceed to amalgamation. Much ore is shipped to South Australia for smelting, with excellent results, when the richness of the material is sufficient to bear the cost of carriage. The undecomposed portions of the ore require special treatment, owing to the presence of the tellurides. The best plan at present appears to be to send the rich ore to smelting works (now under erection at Freemantle), and to treat the low-grade ore by roasting and cyaniding, followed by amalgamation if any coarse gold be present.

Everything at present points to the permanency and richness of these lodes in depth, and it may safely be inferred that this district contains some of the most promising ore bodies yet discovered, either in this colony or elsewhere.—W. G. M.

### *Sulphide Gold Ores, Cyaniding.* R. Recknagel. Eng. and Mining J. 1897, **64**, [20], 580.

The failure in treating sulphide ores by the cyanide process may be attributed to:—(1) The decomposition of cyanide, which makes the cost of treatment too high, even when a satisfactory extraction can be obtained. (2) Unsatisfactory extraction; some sulphide ores, and even concentrates, decompose only a very small amount of cyanide, and in such cases the percentage of extraction is of chief importance. In some cases both of these defects exert an influence against the cyanide treatment.

With regard to the causes of the difference in extraction, although most of the gold contained in metallic sulphides is in the free state, yet some might exist in combination with sulphur, arsenic, antimony, &c., or as a silicate, and thus not be amenable to cyanide treatment, since, although the artificial sulphide of gold is soluble, the natural sulphide — judging from the behaviour of the natural telluride—and the silicate, would probably not be soluble in the cyanide solution applied in practice. In most cases, however, the mechanical conditions under which gold is contained in the ores, rather than chemical difference, will determine the percentage of extraction. Amalgamation previous to cyaniding will overcome the difficulty caused by the coarseness of the gold particles, but when the gold is in a fine state of division, the only remedy is a very fine pulverisation of the ore.

With regard to the high consumption of cyanide, this is not difficult to account for, when a partial oxidation of the sulphides has taken place, and when acid and basic salts have been formed; but when it is due to some of the sulphides being dissolved, an analysis of the ore will not always afford an explanation, since it has been found, on experimenting with natural sulphides, that apparently the same material is acted upon with different degrees of intensity by cyanide solution. The author suggests the following explanation of this apparent inconsistency. The empirical formula of copper pyrites is $CuFeS_2$, which might mean either that $CuS + FeS$ or $Cu_2S + Fe_2S_3$ "combine together"; and in either case, the action of cyanide solution on the ore would be variable, if part of the iron were present in the ferrous state whilst the bulk was in the ferric condition, and *vice versa*. The same replacement might ensue among the two possible copper sulphides. Large interchanges of this kind could of course be detected by chemical analysis, but small ones would escape detection. In the case of the differences in the action of cyanide solution on natural bisulphide of iron, $FeS_2$, the author found that, apart from any admixture, *marcasite*, the variety of $FeS_2$ crystallising in the rhombic system, is always attacked by cyanide to a considerable degree; whilst iron pyrites, crystallising in cubes, is not at all, or only very slightly, acted upon. A. P. Brown (Proc. Amer. Philos. Soc. 1894, **33**, 225—243) has shown that whilst *marcasite* corresponds to the formula $Fe''S_2$, the real formula of pyrites is $Fe''S_2Fe_4'''S_6$, and the author considers it probable that the proportion of four-fifths of ferric iron is not fixed, and that other combined proportions of the two sulphides occur, which would account for all the differences in solubility, since the action of cyanide on $Fe''S_2$ and $Fe_4'''S_8$ seems to correspond with that on $Fe''O$ and $Fe_2'''O_3$, the first being easily soluble, and the latter insoluble. It is probable that similar conditions prevail regarding the dimorphous substances $Ag_2S$, $Cu_2S$, and $ZnS$; and, taking into account the occurence of simple sulphides in sulpharsenites, sulphantimonites, &c., and the frequent partial substitution of one by another, it is very probable that the natural sulphides of heavy metals are not only polymorphous, but that they also exhibit different chemical characters.

The author concludes by advocating the dead-roasting of sulphide ores previous to cyaniding. (See W. McGregor, this Journal, 1897, 803.)—A. S.

### *Cyanide Process, Novelty in.* C. A. Hering. Berg- u. Hüttenm. Zeit. 1897, **56**, 357; through Chem. Zeit. Rep. 1897, **21**, [91], 276.

Netto has evolved a process for the treatment of gold and silver ores by cyanide, a 0·2 to 0·6 per cent. solution of alkali cyanide having the power of dissolving both metals equally. The silver forms $AgK(CN)_2$, which is decomposed by a slight excess of $HCl$, forming $AgCl$, $KCl$, and $HCN$, and the clear liquid drawn off from the $AgCl$ is electrolysed for gold with lead cathodes and carbon anodes; but the gold should not be completely deposited, in order that decomposition of $HCN$ may be avoided. The $HCN$ is reconverted into $KCN$ by the addition of potassium hydroxide. The formation of chlorine during electrolysis, leads to the production of $CNCl$, which is a valuable solvent for gold. Silver is thus said to be obtained free from gold, and gold free from silver, the loss of cyanide amounting to 15—20 per cent. The presence of zinc and lead in the ore does not interfere, but copper must first be removed by roasting and leaching. The process has given good results on a large scale with various Spanish ores.—W. G. M.

### *Gold, Results of the Estimation of, in Ore, and Data in Dry-crushing Experiments.* F. White. The Chemical and Metallurgical Society of South Africa. Oct. 16, 1897.

By the practical extraction of an ore, it was found to contain 9·66 dwt. of gold per ton, 88·86 per cent. of which was extracted in the dry mill, cyanide, concentrates, &c., and the remainder left in the residues. The average assay of the mineral before being dry-crushed was 10·3 dwt. per ton, showing that 6 per cent. of the gold present was unaccounted for. This may partly be due to low results in the residues. Other examples showed in the same way discrepancies between theoretical and practical extraction, which differences are attributed to want of precision in the weights taken, and to errors in assaying, apart, of course, from the consideration of leakages, &c. The results, however, are considered very favourable for the dry-crushing process, and are considered equal to many obtained by wet-milling for the same expenditure of plant, work, and power.

—A. W.

### *Silver Assay, A Modified Method of.* A. E. Knorr. J. Amer. Chem. Soc. 1897, **19**, 811.

*See under* XXIII., *page* 72.

*Phosphorus, Estimation of, in Steel, Iron, and Iron Ores.* J. Ohly. Chem. Zeit. 1897, **21**, [90], 139.

*See under* XXIII., *page* 73.

## PATENTS.

*Gold and Silver and other Metal-bearing Ores and Materials, Refractory; Impts. in the Method of Treating.* J. Campbell, Sydney. Eng. Pat. 26,297, Nov. 20, 1896.

The ore is roasted in a stream of water-gas or Dowson gas in closed retorts, whereby it is rendered more friable, while the base metals and materials are to be carried off as volatile compounds or reduced to a condition which prevents their hindering the subsequent amalgamation. Loss of float gold is minimised. The material may be air-roasted first if desired.—W. G. M.

*Furnaces [Metallurgical; Oxyhydrogen Flame], Impts. in or relating to.* W. P. Thompson, High Holborn, London. From F. Guidi, Rome. Eng. Pat. 27,090, Nov. 28, 1896.

Furnaces for the treatment of metals and the reduction of their ores, in which ordinary firing is combined with the use of oxyhydrogen gases. Each furnace has two chambers —a reverberatory melting one, and a second similar one communicating therewith by a narrow passage, in which the waste heat is utilised, and which is in communication with the chimney. The first chamber is heated to the maximum heat possible by burning ordinary fuel on a grate, or gas from a gasholder. Afterwards a massive valve made of refractory material and having a central metal core, is turned to shut off all connection with the air, the passage to the second chamber is closed, and the oxyhydrogen gases, or one of them, led through tubes into the vault or floor of the furnace. These gases may be those obtained by the electrolysis of water. When discharging, through a hole in the floor, carbonic acid is allowed to enter through a tube to prevent explosions.—R. S.

*Ores, Impts. in Extracting Metals [Gold or Silver] from, and Apparatus therefor.* J. J. Shedlock, Framlingham. Eng. Pat. 27,346, Dec. 1, 1896.

The crushed [gold or silver] ore is delivered in a gentle shower through a hopper at the top of a vertical shaft, at the bottom of which is a burner by which the shaft is heated; on either side of the shaft is a reservoir into which air, steam, chlorine, or other gases may be passed, and from which the gases are discharged through controllable nozzles into the shaft. The ore thus falls through the flame that passes upwards from the bottom, and is subjected throughout its fall to the action of the gases blown across its path. The volatile and suspended portions pass through descending and ascending flues, either directly or through depositing chambers, to a stack. The ore falls into a pointed hopper, which delivers it between air- or water-cooled crushing rolls maintained at a constant distance apart. Thence it is discharged into a horizontal chamber, where it rests upon the surface of mercury or other fluid metal, kept constantly flowing in one direction, and is so conveyed to the opposite end, from which it is removed by jets of air, steam, water, or reducing gas, according to the character of the metal bath used. This bath extracts the precious metal by amalgamation or solution. The bath is constructed with a rotary fan arrangement at one end, which causes a flow of the metal along the bath from that end to the other; and a longitudinal and horizontal partition reaching nearly, but not quite, to each end, provides for the return of the circulating metal. Where silver ores are being treated, they may be mixed with salt before roasting, or chlorine may be injected into the shaft; in this case the solvent would be an aqueous solution of salt or other suitable material. If gold ores are under treatment, the solvent may be aqueous potassium cyanide.—W. G. M.

*Metals to be Welded, Forged, Soldered, Riveted, Perforated, Bent, or the like; Method of Heating.* R. Deissler, Treptow, Berlin, Germany. Eng. Pat. 27,625, Dec. 4, 1896.

For the purpose of riveting, welding, or soldering any metal, the heat is produced on the spot by the chemical reaction of a mixture of either powdered magnesium or aluminium and an oxide, sulphide, or salt of another metal, which will continue to burn throughout the mass after being once ignited at one point. As an example, the mixture may consist of powdered aluminium and oxide of iron, and may also contain a compound of a metal which will be reduced and take part in the soldering. The ignition is started by magnesium ribbon or by a pellet of an igniting mixture consisting of aluminium and barium peroxide or lead oxide, which itself is started by a blowpipe flame. —A. W.

*Metals and Metallic Objects, An Improved Method of Heating.* R. Deissler, Treptow, Berlin, Germany. Eng. Pat. 27,626, Dec. 4, 1896.

The object of this specification is the same as the preceding one, but a carbide is used either with or in place of the powdered aluminium or magnesium. The mixture may consist of calcium carbide, gypsum, and sand, either with or without magnesium. The igniting mass in this case consists of calcium carbide and barium peroxide, which is started by the blowpipe.—A. W.

*Steel and Iron, An Improved Process for Covering or Coating, with Copper or Alloy of Copper.* R. D. Burnie and W. T. Lougher, both of Swansea. Eng. Pat. 29,251, Dec. 21, 1896.

The surface of the steel is thoroughly cleansed from grease and oxide (from the latter by means of acid), washed with a solution of sal-ammoniac or a tinman's flux, and covered with a sheet of tin-foil, lead, zinc, or alloy. The latter is then gradually melted, by immersing the whole in an oven or otherwise, and cooled. The sheet of copper or of its alloy, to be used, is now cleansed and coated with the tin-foil or other sheet in the same way. The two tin surfaces are now painted with flux, placed in contact, firmly clamped, and pressed by a heated roller or other surface, so applied to the copper that the heat passing through the metal suffices to melt the tin beneath, and thus to unite the whole. For ship plates, care must be taken that the steel does not come into contact with the copper at any point, or remain exposed.—W. G. M.

*Metal Alloys [Iron, Copper, &c.], Process for Making.* M. M. Marcus, Lyons, France. Eng. Pat. 28,361, Dec. 11, 1896.

Pieces of clean iron, preferably in the shape of horse shoes, are placed in a strong solution of urea containing also a mixture of 75 per cent. of carbonate and 25 per cent. of bicarbonate of ammonium, whereby a patina of active carbonate of iron is produced on their surfaces. These are then removed and heated, and the molten soft metal, such as copper, tin, or aluminium, is poured on. The soft metal absorbs the patina and, combining with its iron and carbon, produces alloys which are harder and stronger than the original metal. The pieces of iron are cleaned and again used.—A. W.

*Iron (Malleable), Mild Steel, and other Metals, Castings in; Impts. for Use in Producing.* J. Burgess and W. A. Brodie, both of Willenhall, Staffs. Eng. Pat. 30,077 Dec. 31, 1896.

The cavity between the runner and the "git" in the mould is made so that the bottom is at a greater depth than usual below the runner; it is "conoidal" at bottom, and tapers slightly above. The runner enters the cavity tangentially. By this construction the whirling of the metal in the cavity is ensured, and the slag and floating dirt are more thoroughly removed than is at present the case, and are retained in the upper portion.—W. G. M.

*Metals* [*Gold*], *Impts. in Extracting, from their Ores, or in obtaining Solutions of same.* F. W. Streatfeild, London. Eng. Pat. 3303, Feb. 8, 1897.

The pulverised ore, roasted if necessary, is drenched with dilute hydrochloric acid (10 to 30 per cent.) and the mixture subjected to small additions of sodium or other nitrite, solid or in solution, care being taken to avoid the excessive production of nitrous fumes. The whole should be agitated during tho process, when the gold is rapidly dissolved, and may be precipitated by any known method. About 10 lb. of acid per cwt. of ore will be required, and the nitrite necessary may probably vary from 0·25 to 2·5 per cent. on the ore treated.

The use of hydrobromic acid along with a salt of nitrous acid is also claimed.—A. W.

*Converter Furnace, An Improved.* A. E. Thomas, Ormesby. Eng. Pat. 19,764, Aug. 27, 1897.

The principle of this patent is to blow in with the air blast, through the molten iron, any ingredients which may be useful in the refinement of the metal and its conversion into steel. The furnace is of the cupola type with circular blast main, with branch pipes to the twyers leading under the surface of the metal. The ingredients are supplied into the blast through a series of closed hoppers, placed above the blast mains, each one being provided with a stop plug, so that any or all of the ingredients may be employed at one time. When the blast is on and the plug opened, the air circulates round the closed hopper, and carries away with it some of the ingredients into the furnace.—A. W.

*Ores, Mixed Sulphide; Improved Process for Treating.* W. P. Thompson, London. From E. C. Ketchum, Boston, U.S.A. Eng. Pat. 24,121, Oct. 19, 1897.

Mixed ores, containing lead and zinc, are crushed, concentrated, and dead-roasted. Any zinc sulphate that is present may then, if desired, but not necessarily, be leached out. The calcined material is now agitated in large vessels in a solution containing 25 per cent. of caustic alkali, heated to a temperature (say 210° F.) just short of boiling. Here the zinc and lead dissolve, and iron, copper, and silver remain unattacked. The solution is conveyed to a series of electrolyte vats, in the first few of which it is subjected to a current with a voltage of 1·8, which suffices to deposit the lead, but not the zinc; then, when free from lead, it is decomposed by a current of 2·1 volts, which brings down the zinc in reguline form. The lead-depositing vats have carbon or platinum anodes, which are placed in a cell of asbestos or earthenware, containing a pure alkaline solution, so that the ore-solution may not come in contact with them, and thus produce lead peroxide. The lead is in the form of crystals or spongy masses, which are detached at intervals, pressed, and formed into small pigs. In the second series of electrolyte tanks, zinc cathodes are used, which may be remelted with their deposit of zinc, after cutting off a small portion and rolling it out for a new cathode.—W. G. M.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (*A*.)—ELECTRO-CHEMISTRY.

*Electro-Hyposulphite* (*Hydrosulphite*) *Treatment.* E. Urbain. Bull. Assoc. Chim. 1897, **15**, 106.

*See under* XVI., *page* 57.

*Iodoform, Electrolytic Preparation of.* F. Foerster and W. Meves. Zeits. f. Elektrochem. **2**, 1897, 268.

*See under* XX., *page* 66.

### PATENTS.

*Secondary Batteries* (*Envelopes*), *Impts. in or connected with.* [*Rubber Coating.*] J. H. May and F. King, London. Eng. Pat. 25,701, Nov. 14, 1896.

The covers or envelopes of celluloid or the like, employed to retain the active material on the plates of secondary batteries, are given a coating of india-rubber, the solution of which may be either hot or cold, and applied in any suitable manner; or the surface of the envelopes may be first treated with some solvent so as to render them sticky before the application of the india-rubber. Any india-rubber solution, which has covered the perforations in the envelope, may be removed by an air blast.—G. H. R.

*Electrodes* (*Carbon*), *Impts. in.* [*Metallic Connection.*] C. C. Connor, Belfast. Eng. Pat. 26,059, Nov. 18, 1896.

The electrodes are formed of a number of pieces of carbon connected together by having lead or some similar fusible metal or alloy run through or around them, and where great conductivity is required, a rod or wire of copper, iron, or other suitable metal may be embedded in the lead connecting the pieces together. The surface of the carbon is left exposed to the electrolyte, but that of the connecting metal is given a protective coating of Portland cement or other suitable material.—G. H. R.

*Electric Arc Furnaces* [*Automatic; Carbides, &c.*], *Impts in.* The Acetylene Illuminating Co., Ltd., London, and P. R. Day, Foyers, Inverness, N.B. Eng. Pat. 27,323, Dec. 1, 1896.

Data are given on the working of electric furnaces, particularly "those furnaces which comprise a carbon electrode suspended over a fixed crucible or hearth, such as are used for the production of calcium carbide and other substances." The object is "to provide apparatus which will successfully perform the following operations:—(1) The automatic withdrawal of the carbon electrode as the smelted material accumulates beneath it. (2) The automatic raising or lowering of the carbon electrode with sufficient rapidity to prevent any serious fluctuations in the speed of the engine and dynamo. (3) The production of a slight reciprocating movement of the carbon electrode with sufficient rapidity to ensure the manufacture of calcium carbide of high quality, thereby dispensing with the services of the attendant hitherto required for that purpose." Illustrated details of mechanism are supplied. Six claims. No. 1.—"For automatically regulating the arc in electric smelting furnaces, a relay electro-magnet and a motor-reversing switch, the positions of which are controlled by the said relay electro-magnet, the switch being in the armature circuit of an electric motor adapted to move the movable furnace electrode towards and away from the fixed furnace electrode, &c." 2 . . . "means whereby a reciprocating movement is imparted to the movable furnace electrode sufficient to cause the pulverised material to be fed continuously and with sufficient rapidity into the arc."—J. C. R.

*Electrical Storage Batteries and Cells, Impts. in.* [*Paste Plates.*] R. Kennedy, Bradford. Eng. Pat. 28,314, Dec. 11, 1896.

The positive and negative plates are made of pastes formed of dilute sulphuric acid and of red lead and litharge respectively, which are dried slowly, and set in frames of vulcanite or the like, and between perforated sheets of vulcanite or celluloid; or bi-polar plates may be made by uniting layers of positive and negative pastes with or without the interposition of a sheet of lead foil to prevent local action when the battery is at rest. The battery is composed of any suitable number of such plates separated by rubber distance pieces and ebonite forks, the latter being used to space the ebonite supporting plates and stiffen them against vibration. The end plates are formed on lead sheets, to which the connections are attached.—G. H. R.

*Chlorine, Impts. in the* [*Electrolytic*] *Manufacture of.* E. J. Hunt, Wednesbury; and E. F. Fairlie, Wolverhampton. Eng. Pat. 29,066, Dec. 18, 1896.

*See under* VII., *page* 47.

*Batteries, Secondary, Preparing Positive Plates for* [*Nitrites*], *An Improved Method of.* H. W. Handcock and A. H. Dykes, London. Eng. Pat. 1201, Jan. 16, 1897.

The claim is for a "method of forming positive plates [lead] for secondary batteries . . . by the agency of the nitrites of ammonium or organic ammonium bases [" such as Lenthine "] or other organic bases containing NH, $NH_2$ . . . ."

"We find that the best density for the acid [sulphuric] is 1·05, and use by preference the nitrite of ammonium of density 1·1. The two must be mixed quickly."

—J. C. R.

*Ozone, the Manufacture and Production of* [*" Open Ozonisers," &c.*], *Impts. in and connected with, and in Apparatus therefor.* E. Andreoli, London. Eng. Pat. 15,813, July 2, 1897.

. . . . . " A series of open ozonisers arranged in chambers in such a manner that the air or gas which enters the said chambers has to pass evenly between the electrodes of the said ozonisers where it is electrified by a silent electric discharge, the glow of which is reduced to a minimum to avoid the generation of excessive heat and the formation of nitrous compounds." 2. . . . " open ozonisers consisting each of a set of electrodes, held together in a frame by clamps or wooden screws or the like." 3. . . . " the use of duplex dielectric sheets [' two thin sheets of glass placed one against the other '] for preventing polarisation." 4. 5. 6. . . . " flat electrodes ; arrangement of a number of open ozonisers in large chambers over or in apertures ; large chambers or reservoirs which supply direct the ozone in its nascent state where it has to be utilised." The inventor's experience is, " that the most satisfactory results are obtained with point-bearing grids [electrodes], one facing the other, or point-bearing grids opposite flat metallic surfaces ;" and (in his provisional specification) that " a current of 2 ampères discharging through 50 square feet of electrodes gives good results." Patents cited, Nos. 17,426 of 1891, 9631 and 21,794, of 1892, 21,707 of 1893, and 10,372 of 1896 (this Journal, 1893, 452 ; 1894, 162 ; 1896, 726).—J. C. R.

*Batteries* [*Traction*], *Electrical Storage, and Plates therefor ; Impts. in and connected with.* R. Kennedy, York. Eng. Pat. 19,638, Aug. 25, 1897.

" Batteries specially suitable for traction purposes." The claims are .—1. . . . . " plates consisting of a central solid lead sheet painted on one side with litharge and dilute sulphuric acid, and on the other side with red lead and dilute sulphuric acid, and one or more layers or thicknesses of perforated lead sheets also painted with litharge and red lead on each side of the central sheet, the litharge painted sheets being placed on the litharge painted side of the central plate, and the red lead painted sheets on the other side thereof, the whole being electrically connected and made solid round the edges by fusing or soldering," &c. 2. . . . " forming the plates by cleaning lead sheets with dilute [5 per cent. hot] nitric acid, washing them in clean water and painting or coating them while chemically clean and still wet with litharge and red lead and sulphuric acid, and afterwards drying the same [slowly], &c." 3. . . . " lead plates [bolted together] having sides of opposite polarity . . . . in combination with indiarubber washers and non-conducting diaphragms," &c. 4. . . . " forming two pockets or chambers insulated from each other at the bottom of each cell by means of a partially perforated diaphragm," &c. [to prevent short circuiting].—J. C. R.

*Electric Accumulators, the Manufacture of Plates* [*Asbestos Frames and Acetates*] *for ; Impts. relating to.* L. Gnelzow, London. Eng. Pat. 19,846, Aug. 28, 1897.

" 1. A plate for electric accumulators consisting of a frame of asbestos or other insulating material capable of withstanding acid, &c., a lead core inserted into the space of the frame and having horizontal slots with lips bent to one side or the other for preventing the sagging of the active material, &c." " 2. A plate for electric accumulators, in which minium and litharge forming the active material are chemically combined by potassium acetate and sulphuric acid," *i e.* " a solution of potassium acetate in distilled water " and " dilute sulphuric acid " are employed for making a dough with lead oxides.—J. C. R.

*Electric Batteries (Primary), Electrodes, and Method of Making the same ; Impts. in.* R. W. James, London. From the Bell Electric Company, New York, U.S.A. Eng. Pat. 22,044, Sept. 25, 1897.

The battery is contained in a case formed of insulating material. There are bevelled openings, in which pass screws which are cast into the electrodes. These screws are surrounded by rubber gaskets which fit the bevelled openings in the case, and within the case and about the openings are rings or thimbles provided with flanges, on which bear washers, perforated centrally for mounting on the screws, which carry binding nuts. One electrode is formed of a zinc plate, and the other of a core and a head united to it, and both formed of a practically invulnerable alloy, composed of lead, antimony, and a tellurate. On the stem are strung a number of discs of lead, provided with projections to keep the discs apart, thus exposing the core to the action of the electrolyte, and providing space for the deposition of the active material. The latter electrodes are first " toned " electrolytically in a bath of sulphuric acid, water, and bisulphide of mercury. They are then thoroughly washed in water and " formed " in a mixture of an aqueous solution of sulphuric acid, caustic potash, and the sesquinitrate or a higher nitrate of magnesium, and a solution of glyceride of potassium, nitric acid, and water. When the formation is complete, the electrodes are again washed, and then " hardened " by immersing them for a few seconds in a solution of magnesium sulphate, glycerin, and water until they are warm, and then dipping them in an aqueous solution of sulphuric acid till all gas bubbles disappear, after which they are preferably exposed to the action of the electric current for a short time in a bath of diluted sulphuric acid, sp. gr. 1·200. After this process the electrodes are dipped into an aqueous solution of sulphate of magnesium, which is allowed to dry on them, and they are then ready for use in the battery, the electrolyte for which is composed of diluted sulphuric acid, sodium sulphate, and magnesium sulphate. There are 39 claims.—G. H. R.

*Carbon and Zinc Elements, An Electrolyte for, suitable for Lighting and other Purposes.* J. S. Johansen, Odense, Denmark. Eng. Pat. 23,123, Oct. 8, 1897.

The zinc element is surrounded by a paste consisting of equal parts by weight of ammonium sulphate, potassium chloride, and zinc oxide, enclosed in a well sealed jar. The negative is carbon, which stands in a strong solution of potassium chromate.—G. H. R.

## (*B.*)—ELECTRO-METALLURGY.

*Copper Refineries, Electrolytic ; Tank Residues, Composition and Formation of, in.* E. Kolier. J. Amer. Chem. Soc. 1897, **19**, [10], 778—782.

The average composition of the vat deposit obtained (I) during a year's treatment of reverberatory-, and (II) that from three months' treatment of converter-copper, are as follows, exclusive of oxygen and hydrate water :—

| — | I. | II. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Ag | 53·894 [0·3064] | 55·15 [0·3076] |
| Au | 0·296 | 0·198 |
| Cu | 11·01 | 13·82 |
| Pb | 0·91 [0·0093] | 2·07 |
| Bi | 3·93 [0·0320] | 0·34 [0·0035] |
| Sb | 6·25 [0·0651] | 2·44 [0·0510] |
| As | 2·11 [0·0586] | 1·09 [0·0180] |
| Se | 0·39 } [0·0098] | 0·72 |
| Te | 1·17 } | 0·89 |
| Fe | .. | 0·80 |
| $SO_4$ | 5·27 | 10·68 |
| $H_2O$ | 2·36 | 2·60 |

The numbers in square brackets represent the percentages of the various elements in the unrefined copper, and show therefore the nature and the extent of the refining effected by electrolysis. From these numbers it may be found that the whole of the silver, gold, selenium and tellurium remain in the residue, whilst, of the other elements, there are left in the slime in the two cases, respectively :—Cu, 0·07, 0·08; Bi, 78·22, 66·71; Sb, 61·14, 29·90; and As, 22·90, 37·84 per cent.—W. G. M.

### PATENTS.

*Pipes, Tubes, and other Articles, Impts. relating to the Electric Deposition of Metal on.* [*Epicycloidal motion.*] W. Wright, Acocks Green. Eng. Pat. 190, Jan. 7, 1897.

The apparatus employed for electro-plating pipes and tubes both internally and externally at one operation consists of a vat containing a rotating shaft or spindle carried on suitable bearings, and having mounted on it three built-up discs which are adjustable on the shaft, two of which support the tubes to be coated, whilst the third carries the anode rods, each tube having one extending throughout its entire length. The tubes are loosely mounted in sockets placed concentrically on the inner surface of the supporting disc and secured by the pressure of springs at one end, or instead of such sockets and springs, hollow gun metal pins may be employed so that when the complete frame is revolved in the bath, the tubes have an independent or epicycloidal motion imparted to them.—G. H. R.

*Electro-Deposition of Metals, Certain Impts. in Apparatus for the.* [*Revolving Barrel*]. A. E. Cook, Birmingham. Eng. Pat. 1627, Jan. 21, 1897.

The apparatus which is intended for electro-plating pens and similar small articles, consists of two concentric revolving barrels, the outer of which is water-tight and contains the anodes, whilst the inner, which is perforated to allow of the circulation of the electrolyte, contains the cathodes and the articles to be plated. A water-tight door affords access to the interior of the inner barrel, and suitable means are provided for establishing electric connection between the anodes and cathodes and the source of electric supply. In a modified form of the apparatus the inner barrel is shorter than the outer, leaving a space at each end in which the anodes are placed, and the ends of the inner barrel are perforated.—G. H. R.

*Electro-Deposition of Copper and other Metals,* [*Membraneous Rubbers, &c.*]; *Impts. in and Relating to the.* W. E. Heys, Manchester. From E. Dumoulin, Paris. Eng. Pat. 2709, Feb. 2, 1897.

The claims embrace "obtaining a smooth homogeneous electro deposit of metal on a rotary cathode" . . . "by employing animal membranes [' intestines, bladders, and skins '], from which the excess of greasy and flesh matters, [' which are or can become soluble or adhesive at or below 16° C.', the 'critical' temperature, below which it is proposed to work the electrolyte] have been removed, which come into light contact with the cathode." . . . "Means for circulating, cooling, oxidising, and filtering the electrolyte" . . . purifying the electrolyte [from organic matter] by permanganates, cupric oxide, &c.; injecting air therein, and afterwards cooling, &c.

These animal membranes, "consisting of unaltered albumin, fibrin, and gelatin" the inventor terms "impregnators," which "are simply arranged in contact with the cathodes without pressure, so that the least asperity or roughness on the cathode, seizes upon some of the fibres and filaments, with which the asperities become covered or thatched [temporarily], so as to retard the further deposition of metal thereon." It is observed, with reference to the purification of the electrolyte, that—"When the organic matters come into solution, they commence to adhere to the cathode, and become imprisoned in it. Then, in the process of annealing, these matters are burnt, and thus generate gases within the metal under enormous pressures, the effect being the making of the metal short and brittle." Patent 16,360 of 1895 is referred to.—J. C. R.

## XII.—FATS, OILS, AND SOAP.

*Lard and Butter, Foreign Fats in; Detection of.* C. R. Cochran. J. Amer. Chem. Soc. 1897, **19**, 796.

*See under* XXIII., *page* 74.

*Fats and Oils, Edible; Analysing and Discriminating.* J. Suisse de Chim. et Pharm. **35**, 448; Bull. Assoc. Belge des Chim. **11**, 160.

*See under* XXIII., *page* 76.

### PATENTS.

*Soap, Improved Process for Making.* R. Gesell, Berlin. Eng. Pat. 26,396, Nov. 21, 1896.

The saponified mass is mixed with wood fibre previously impregnated with boric acid, bran, and oatmeal, and the soap salted out and moulded in the usual manner.

—C. A. M.

*Palm Oil, A New or Improved Process for the Decolorisation of.* J. L. Garle and C. C. Frye, both of Middlesex. Eng. Pat. 28,682, Dec. 15, 1896.

Ozone, ozonised oxygen, or ozonised air, which may contain a small proportion of oxides of nitrogen, is forced or drawn through the palm oil. The oil is maintained at a temperature sufficient to keep it liquid, but not exceeding 100° C., until the decolorisation is complete.—C. A. M.

*Soap, Impts. in the Manufacture of.* J. T. Potts, Bromboro' Pool, Chester. Eng. Pat. 751, Jan. 11, 1897.

Claims are made for the production of liquid soaps which will not congeal at ordinary or low temperatures, and "the fluidity of which results from the blending of two or more fatty acids, without the necessity for an excess of water." According to claims 2 and 3, "the liquid soaps are made from a mixture of two or more solid or semi-solid soaps," or "from cocoa-nut oil, resin, and one or more of the oils specified," *viz.*, "Animal, neatsfoot, lard, fish, castor, cotton, olive, ground-nut, rape, linseed," or "any vegetable fatty oil."—C. A. M.

*Oleic Acid, Impts. in the Treatment of.* J. David, Paris. Eng. Pat. 3700, Feb. 11, 1897.

Oleic acid cooled to zero is treated with 40 to 50 per cent. of its weight of conc. sulphuric acid containing about 5 per cent. of fuming acid, the temperature never being allowed to exceed 20° C. during the agitation. After standing 12 hours, the mixture is run into cold water and stirred, the temperature being kept below 30° C. After about two hours, two layers are formed—partially converted oleic acid and dilute sulphuric acid. The latter is run off, more water added and well mixed in, and after 24 hours white crystals separate; they are filtered off. The crystals melt at about 45° C., and amount to about 20 per cent. of the weight of oleic acid taken. The brown filtrate is boiled in a vessel with steam for an hour, when it separates into layers. The lower of these is run off, fresh water then added, and the whole boiled until all traces of sulphuric acid are removed from the upper layer. This is now cold-pressed in bags, the black oil runs off, and there remains in the bags a hard brittle mass which may be purified as usual in stearin works. The pressed mass melts at about 65° C., and the yield is from 30 to 35 per cent. of the oleic acid treated. By the mixture of these two products, a stearin melting at about 51° C. is obtained.

—C. A. M.

*Soaps, An Improved System for Manufacturing by the Direct Employment of Oleaginous Seeds.* J. Monterrubio, Buenos Ayres. Eng. Pat. 23,197, Oct. 9, 1897.

The seeds (pea-nut, &c.) are cleaned, crushed into a fine paste, mixed with the requisite quantity of caustic lye (hot or cold), and the mixture well stirred and beaten until saponification is complete. It is claimed that, in addition to the saving effected by the utilisation of what is usually a waste product, soaps thus produced are superior hygienically to those made from tallow, &c.—C. A. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (*A.*)—PIGMENTS, PAINTS.

### PATENTS.

*Ink or Material for Printing and Similar Purposes.* A. J. Boult, Middlesex. From E. Maître, Paris. Eng. Pat. 30,121, Dec. 31, 1896.

This is an ink termed "direct-or," consisting of an intimate admixture of gold or bronze powder with varnish and fixed oils, and specifically of a mixture of gold or bronze powder with cocoa-nut oil, palm oil, laurel oil, copal varnish, and damar in prescribed proportions, the object being to enable printing and bronzing to be performed in one operation. —C. A. M.

*Preserving Structures, Impts. in Compositions or Paints for.* J. W. Blake and S. J. Blake, both of Gosport. Eng. Pat. 9147, April 13, 1897.

Aluminium powder, turpentine, and gold size mixed in suitable proportions into a thick paste.—C. A. M.

*Black Pigments, Process for Manufacturing from Carbonaceous Materials.* R. Strobentz and J. Fried, both of Buda-Pest. Eng. Pat. 21,718, Sept. 22, 1897.

This invention is based on the fact that certain kinds of brown coal, especially lignites, contain certain organic compounds which form black compounds with metallic salts. The finely pulverised carbonaceous material is mixed, in a suitable proportion, with metallic salts (preferably the sulphates or chlorides of iron), with or without the addition of certain organic substances, such as tannic acid, gallic acid, pyrogallic acid, or similar compounds. Pigments manufactured in this way without burning or igniting, may be substituted for the lamp black and black pigments hitherto employed.—C. A. M.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Suède Leather, Bleaching, Dyeing, and Finishing.* Der Gerber, Nos. 553—559, 1897.

The skins, when tawed, are usually of a dull grey colour, unsuitable for dyeing bright or light shades, and must therefore be bleached. This is usually done by spreading the skins (grain side uppermost) upon canvas or linen, which should be tightly suspended some distance from the ground, placed with a southern aspect, so as to get as much sun as possible. Great care is necessary to avoid dirt, smoke, rain, or specks of dust, and that the skins should be spread out flat without crease or fold. The skins, which must be taken in each night, to be put back in the morning, are usually bleached white, in favourable weather, at the end of the third day; they are then washed in one or two changes of cold water, and finally in water containing a small quantity of Prussian blue, placed upon a wooden horse to drip, then dried, damped back, staked, buffed, pumiced, and redried, and are ready for dyeing any shade.

In order to produce bright and even shades, the following rules must be observed:—Absolute cleanliness of all utensils; the dye-wood extracts must be perfectly clear and freshly extracted. This is best done for these goods by pouring boiling water upon the dye-wood and allowing it to macerate for a short time, or by boiling for 15 minutes. The clear solutions must be added to the dye-bath in small successive portions; this gives greater depth, and is more economical than adding all at once. The skins must be kept in constant motion all the time, and as soon as the desired shade is obtained the skins must at once be removed and hung upon a wooden horse to drip.

The dyeing is usually carried out in circular pine-wood tubs. 20 or 30 dozen skins are put in at once and are covered with warm water. They are trodden with the feet until thoroughly soft. The dye is now added in small quantities until the required shade is produced, the operator keeping the skins in motion the whole time by treading. The skins are then taken out, allowed to drip for a few hours, then dried, and are finished by damping back, staking, pumiced if necessary, finally dried, and they are then ready for the market.—J. G. P.

*Leather, Currying of.* Der Gerber, Nos. 553—559, 1897.

The currying of leather may be divided into two heads: (1) impregnating the leather with fats in order to fill the leather and render it more pliable, to add weight, but not necessarily to combine with or tan the leather; and (2) impregnating with such fats as will "combine" with and further tan and preserve the fibres of the leather. Currying in general makes the leather more resistant, pliable, and stronger, as well as preserving the fibres and adding weight and substance. It may be accomplished by smearing the leather when damp upon the flesh side with grease, and laying away in pile, one piece upon another, flesh to flesh, for periods varying according to the thickness of the pieces or the purpose for which they are intended; or dried leather may be treated with hot grease in a drum. By the first process, 5 to 8 per cent. of grease penetrates the leather, and by drumming, 25 to 30 per cent. may penetrate. To curry leather for different purposes, the following ingredients are recommended:—

"*Strap Butts*" (and similar leathers which are curried by the table process).—A mixture of wool-fat, mineral oil, and light tallow, or cod oil and tallow.

"*French Wax Calf.*"—Dégras and wool-fat.

"*French Calf.*"—Dégras and wool-fat.

"*Common Brown Calf.*"—35 parts of dégras, 20 parts of cod oil.

"*Black Calf*" (not generally fully tanned).—40 parts of dégras, 10 parts of vaseline, 20 parts of wool-fat, and 30 parts of soft tallow.

*Satin Calf.*—Moellon and 10 per cent. of soft tallow.

*Horse Hide* (for boots).—40 parts of dégras, 10 parts of cod oil, 20 parts of wool-fat, and 30 parts of soft tallow.

*Shoe Butts.*—60 parts of dégras, 20 parts of wool-fat, and 10 parts of resin oil.

*Brown Shoe Leather* (*Cow*).—30 parts of degras, 30 parts of cod oil, 15 parts of wool-fat, and 30 parts of soft tallow.

In preparing these mixtures for use, some care is necessary to obtain a proper mixture of the fats. They should not be boiled together, but the hardest fat first put into a suitable vessel, heated to 130°—140° C., the liquid fats stirred in gradually or kept constantly stirred by some suitable appliance until almost cold; by this means a proper admixture is obtained, and the harder fats do not crystallise or separate out upon the surface of the leather, the whole mass permeating into the structure, coating and preserving the fibres and filling the cavities. Great care must be observed in the selection of the fats, that they are pure and free from mineral acids, this being especially necessary with some brands of dégras.—J. G. P.

### PATENTS.

*Treatment of Raw Hides, Impts. in or relating to.* E. A. Muskett and J. B. Scammel, trading as John Burdon and Co., London. Eng. Pat. 28,067, Dec. 8, 1896.

The invention consists in preserving hides and skins for shipment by soaking in tubs or tanks containing a 15 per cent. solution of "boronite" ("boronite" consists of 95 per cent. of boric acid and 5 per cent. of thymol) for from 1—3 hours, and then drying them to about the consistency of india-rubber. For goods that are shipped wet in casks, the process is the same, but the drying is dispensed with. An alternative method of applying the "boronite" for hides in the dry state, is to rub it well on both sides of the pelt. —J. G. P.

*Hides and Skins, Impts. in Treating.* S. Millar, 2, Broompark Terrace, Dennistoun, Glasgow, and C. E. Miller, Hillhead, Glasgow. Eng. Pat. 3180, Feb. 6, 1897.

An essential feature is the use of formalin (of a strength of about 40 per cent. formic aldehyde). In practice, details are varied, but good results are obtained in the following manner:—The hides, after unhairing and the neutralisation of the lime, are suspended in a solution of from 3—4 galls. of the formalin solution to from 1,400—1,800 galls. of water for one hundred ox hides each weighing 80 lb. For the first pack of hides more of the formalin solution is necessary. The formalin renders the gelatin in the hides insoluble, and in the after processes greatly increases the absorptive power, so that the tannin is taken up very rapidly.—J. G. P.

*Imitation Leather, Improved Manufacture of.* T. E. Bowman, Newcastle-on-Tyne, and J. Bowden, Gateshead Fell, Durham. Eng. Pat. 1587, Jan. 20, 1897.

In carrying out this invention, any suitable fabric, such as canvas, cotton, cloth, or the like, is first boiled in a solution of tannic acid and afterwards treated on one or both sides with a size of linseed or other boiled oil and the desired colouring matter. The product is dried and then treated with a mixture of linseed oil, 1 gall.; best dark Prussian blue, 2 oz.; vegetable black, 1 oz.; copal oil, 4 oz., previously boiled up together to the consistency of jelly. The product is then dried, and may be embossed or grained if desired.—J. G. P.

*Treatment of Leather Waste, and the Apparatus employed therein; Impts. in or relating to the.* A. J. Boult, London. From G. Brigalant, Barentin, France. Eng. Pat. 24,080, Oct. 14, 1897.

The patentee describes an apparatus for the conversion of leather waste into pulp. The claim is for the process of manufacturing such pulp, and for the apparatus by which the process is carried out.—J. G. P.

## XV.—MANURES, Etc.

*Nitrification in Soils.* T. Schlœsing, jun. Comptes Rend. 1897, **125**, [21], 824—827.

Artificial soils (prepared by intimately mixing quartz-sand, clay, and chalk) to which solutions of ammonium sulphate were added, were kept in large flasks for $2\frac{1}{2}$ to $3\frac{1}{2}$ months, and the nitric nitrogen determined. The following table shows the composition of the soils and the amount of nitrogen which was nitrified. Nitrifying organisms were added in each case.

| — | Sand. | Clay. | Chalk. | Water. | $(NH_4)_2SO_4$. | N. Nitrified. |
|---|---|---|---|---|---|---|
| | Grms. | Grms. | Grm. | Grms. | Mgrms. | Per Cent. |
| 1 | 100 | 0 | 0·5 | 10·0 | 50·0 | 83·0 |
| 2 | 90 | 10 | 0·5 | 10·0 | 50·0 | 91·0 |
| 3 | 80 | 20 | 0·5 | 10·0 | 50·0 | 89·0 |
| 4 | 75 | 25 | 0·5 | 10·0 | 50·0 | 56·0 |
| 5 | 70 | 30 | 0·5 | 10·0 | 50·0 | 10·0 |
| 6 | 100 | 0 | 1·0 | 9·5 | 100·0 | 63·0 |
| 7 | 90 | 10 | 1·0 | 9·5 | 100·0 | 66·0 |
| 8 | 85 | 15 | 1·0 | 9·5 | 100·0 | 94·0 |
| 9 | 80 | 20 | 1·0 | 9·5 | 100·0 | 101·0 |
| 10 | 75 | 25 | 1·0 | 9·5 | 100·0 | 21·0 |
| 11 | 70 | 30 | 1·0 | 9·5 | 100·0 | 2·7 |
| 12 | 70 | 30 | 0·5 | 10·6 | 66·6 | 80·0 |
| 13 | 70 | 30 | 0·5 | 11·5 | 66·6 | 100·0 |
| 14 | 70 | 30 | 0·5 | 13·2 | 66·6 | 100·0 |
| 15 | 70 | 30 | 0·5 | 11·0 | 66·6 | 100·0 |

The very incomplete nitrification in Nos. 4, 5, 10, and 11 could not be due to want of air, as the soils were, in every case, quite permeable; moreover, in Nos. 13–15, which contained 30 per cent. of clay, and more water, the whole of the ammonia was nitrified. The failure in the cases mentioned, must therefore be ascribed to insufficient water in presence of 25—30 per cent. of clay. With regard to the remarkable effect brought about by a slight increase in the amount of clay, or by a slight decrease in the amount of water, it is suggested that the conditions under which nitrification was diminished were due to the limit having been exceeded, below which the capillary attraction, which retains the water and dissolved substances on the soil became, nearly everywhere, superior to the osmose which caused the water to penetrate the cells of the microbes, and thus rendered them unable to obtain food from the soil. The results have an important bearing on the question of nitrification in heavy soils.—N. H. J. M.

*Black Rot [Vines], Favourable Period for the Treatment of.* A. Prunet. Comptes Rend. 1897, **125**, [22], 889—891.

Application of solutions of cupric compounds after an epidemic of black rot prevents subsequent attacks, but the greatest effect is produced by an application from two to five days after the acute period of the attack, that is, five to eight days after the first appearance of the spots. At this time the spores are liberated in great numbers.

If it should happen to be impossible to treat in time to prevent an attack, the effects may be considerably diminished by thinning the leaves and then at once treating with the cupric preparation.—N. H. J. M.

*Argon: Its Influence on Vegetation.* T. Schlœsing. Comptes Rend. **125**, [19] 719—722.

The author has made comparative experiments, lasting two months, on the growth of plants in artificial atmospheres with and without argon. Measurements of the height and weight of the plants produced from seed, and also of the carbon dioxide consumed and the oxygen produced by them during growth, lead to the conclusion that argon, in proportions similar to those in which it exists in atmospheric air, is without sensible influence on vegetation.—J. T. D.

*Basic Slag, Estimation of Citrate-soluble Phosphoric Acid in.* O. Böttcher. Chem. Zeit. 1897, **21**, [95], 993.

*See under* XXIII., *page* 71.

*Basic Slag, Estimation of Citrate-soluble Phosphoric Acid in.* M. Passon. Zeits. angew. Chem. 1897, [23], 746.

*See under* XXIII., *page* 72

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar Factories, Mutual Control of.* F. Dupont. Bull. Assoc. Chim. 1897, **15**, 116—120.

Following the lead of the sugar factories in Poland, the Association of Chemists interested in sugar works in France has organised a similar system. The results of a number of works are grouped and co-ordinated, and communicated to the works each week, two days after reception, in the form of a table in which each factory is represented by a number, names being suppressed to respect legitimate scruples. Since the start in 1891, when 14 works entered, the number has remained stationary at 13, a few works not having continued their subscription of 20 francs, and others having replaced them. A larger number would enable more useful generalisations to be made.

By comparing the results of his own works with those of his neighbours the manufacturer may see whether his work is good or bad, seize upon defects and remedy them.

| — | Density of Beetroot Juice. | Percentage of Sugar in Beetroots. | | Juice Extracted per 100 Kilos. of Beetroots. | | | Density of Diffusion Juice. | | |
|---|---|---|---|---|---|---|---|---|---|
| | | — | Mean. | Mean. | Maximum. | Minimum. | Mean. | Maximum. | Minimum. |
| 1891-2 | .. .. | .. .. | .. | 119 | 140 | 103 | 5·12 | 5·65 | 4·26 |
| 1892-3 | 5·9 to 7·0 | 10·5 to 14·0 | .. | 113 | 124 | 101 | 4·89 | 5·20 | 4·50 |
| 1893-4 | 6·6 „ 7·4 | 12·3 „ 14·7 | .. | 115 | 129 | 102 | 4·91 | 5·30 | 4·02 |
| 1894-5 | 6·3 „ 7·3 | 12·6 „ 14·3 | 13·50 | 113 | 124 | 101 | 5·12 | 5·30 | 4·90 |
| 1895-6 | 7·0 „ 8·7 | 13·0 „ 16·0 | 14·50 | 117 | 127 | 107 | 5·34 | 5·87 | 4·80 |
| 1896-7 | 6·0 „ 7·0 | 12·0 „ 13·8 | 13·00 | 110 | 123 | 102 | 4·70 | 5·10 | 4·50 |

Purity about equal to that of the beetroot juice. Sugar lost in the cossettes from 0·10 to 0·40; mean, 0·25. Indeterminate losses in diffusion, 0·02 to 1·5 per cent.; mean, 0·20, increasing towards the end of manufacture, which shows that there must be either a transformation of sugar in the diffusion, or at least an alteration of polarisation. The losses of sugar in filtration vary from 0·01 to 0·05 per cent. of beetroots. Losses of sugar in the scums vary from 0·02 to 0·54, mean, 0·1. This loss shows a diminution from 1891 to 1897, proving that the manufacturers have devoted their attention to avoiding it. Indeterminate losses 0·12 to 1·80, diminishing sensibly in later campaigns. Losses during evaporation and boiling are not given. Total loss 0·45 to 2·30, with constant diminution the three last years. The quantity of lime used, varies from 2 to 4 per cent. of the weight of the beetroots; mean, 2·50. The alkalinity of the first carbonatation juice varies about 1 grm. of CaO per litre. That of the second, between 0·40 and 0·1; mean, 0·25. Polarisation of first jet massecuite, 71 to 87, with a saline coefficient of 10 to 48, and purity of 77° to 93°. Polarisation of the second jet massecuite 60° to 73°, saline coefficient 6·50 to 14, and purity 69° to 79°. Not a single factory determined the sugar crystals in the massecuite. The returning of spun syrups was practised to a greater or less extent in all the works. The yield of first jet sugar per cent. of beetroots, varied greatly, from 6·50 to 10, according to the quantity of syrup returned, and according as mixing or crystallisation in movement were employed. One palpable fact appears from the tables, that those works using returned syrups obtain a higher yield of first jet sugar. There is, therefore, an indisputable advantage in adopting this process. The charge of cossettes per hectolitre of capacity of diffusers varies from 50 to 62 kilos. The quantity of beetroots worked in 12 hours per 100 hectolitres of available capacity of diffusers is very variable: from 25 to 55 thousand kilos. Diffusion was carried out at 75° to 85° C., and it is remarked that those works which use higher temperatures, obtain a diffusion juice of lower purity than that of the natural beetroot juice. No correlation was observed between the exhaustion of the cossettes and the temperature of diffusion, too many causes influencing the exhaustion; the number and capacity of the diffusers, the rapidity of working, the thickness and quality of the cossettes, &c. The percentage of $CO_2$ in the furnace gas is 22 to 33, mean 25. The duty of the lime furnaces which do not give a richness of 30 is evidently defective. With one or two transient exceptions, no one tested the reducing sugars. This is obviously wrong, for normal beetroot juice almost always contains a more or less considerable quantity of reducing sugars. No one took the trouble to determine the viscosity of the juices and syrups. It is to be hoped that this question will in future receive the attention it deserves.—L. J. de W.

*Cane Sugar, The Crystallisation of, in Practice.* Claassen. Zeits. für Rübenz. Ind. 1897, **47**, 799.

The author has investigated very thoroughly, and illustrates by numerical results, the following points:—The crystallisation of juices and syrups; the most favourable conditions, in respect to temperature and concentration, determining the same; and lastly, the composition of massecuites and of the adhering syrups, at various stages of the boiling process. It is shown that an important connection exists between the degree of supersaturation of the mother-syrup, the time of boiling, and the proportion of sugar that crystallises out. Supersaturation of the syrup does not in all cases accelerate the rate of crystallisation in the unit of time, but frequently brings about the reformation of small crystals—a result sought to be avoided. Depending on the purity of the juice, the coefficient of supersaturation should not exceed 1·1 to 1·2. In many sugar manufactories, the dimensions and form of the vacuum-pans are by no means calculated to satisfy the rule that boiling should be conducted slowly; and it is clear, that in such cases, it may be advantageous to put back and rework the drainings and syrups, as a means for protracting the boiling process. In respect to pure "thick juice," the limit of purity of the mother-syrup may be put down as 80°, to secure which, a definite time of boiling, and therefore a definite amount of vacuum space is required. As regards crystallisation (aided by agitation) in conjunction with "first products," two modes of procedure are to be distinguished. Firstly, assuming it be desired to reduce the purity of the mother-syrup to about 72°, the massecuite, after addition of syrup, should be boiled down until it contains 8—8½ per cent. of water; or, if highly concentrated, should be at once diluted with hot syrup, and allowed to cool in 12—18 hours to 50°—60°. Slower cooling is purposeless, as the yield of sugar is not increased thereby. If more complete separation of the sugar be desired, the water percentage should be reduced to 6—7, the mass cooled to 70°, stirred until a considerable quantity of sugar has crystallised out, and finally cooled to 50°, but not lower.—H. T. P.

*Sugar Solutions, Action of Sulphurous and Hyposulphurous Acid on.* L. Beaudet. Bull. Assoc. Chim 1897, **15**, 90—97.

The author finds that sulphurous acid has no appreciable destructive action on solutions of pure sugar below 55° C.; at higher temperatures the destruction of sugar increases rapidly with the temperature, increases likewise with the acidity and with duration of contact, but in the latter case in a decreasing progression; it decreases, finally, with the concentration, if the acidity per cent. of liquid remains constant, and increases, on the contrary, if the acidity per cent. of sugar remains constant.

The introduction of a quantity of impurities, even very small, prevents the destructive action of sulphurous acid on sugar. The author agrees with Aulard that, at the ordinary temperature, a syrup from either the cane or beetroot, whether containing glucose or not, and containing the impurities of these products, undergoes no inversion by an acidity produced by sulphurous acid. That the same syrup, evaporated in a vacuum to massecuite, that is to 85° C., undergoes no inversion or denaturing of saccharose. He is also of the opinion of Vivien, that sulphurous acid does not attack impure sugar solutions; it acts by displacing organic acids, forming alkali or alkaline earthy sulphites. The acidity of beetroot syrup set at liberty by sulphurous acid is without action on sugar.

Hyposulphurous (hydrosulphurous) acid is without destructive action on solutions of sugar, pure or impure.
—L. J. de W.

*Molasses and Osmosis Liquids, Polarisation of.* [*Clarification.*] J. Diamant. Chem. Zeit. 1897, **21**, 981—982.

The polarimetric examination of molasses and osmosis liquids, is almost universally preceded by clarification with basic acetate of lead; and various methods have been suggested for getting rid of the excess of lead, which, as has been repeatedly shown, may influence the rotation of the liquid, and besides, causes it rapidly to become turbid on exposure to air. For the purpose, the author highly recommends zinc-dust. The clarified liquid, filtered from the lead precipitate, is mixed with a little zinc-dust and well shaken. The excess of lead is almost instantly thrown down, and the liquid, when filtered, remains bright, and besides, is distinctly paler than would otherwise be the case.
—H. T. P.

*Sugar Solutions, Action of Sulphurous Acid on.* E. Urbain. Bull. Assoc. Chim. 1897, **15**, 97—106.

After the action of the sulphurous acid on the sugar solutions, the author not only determined the sugar and glucose, but the sulphurous acid by iodine, and the total sulphur acids, after oxidising with chlorine, thus obtaining the $SO_4H_2$ arising from the oxidation of sulphurous acid.

When the treatment with sulphurous acid was carried out in a vacuum of 70 cm. of mercury, only traces of sulphuric acid were found, and the glucose formed was either *nil* or much less than when the sulphitation was carried out in the open air; considerable quantities of sulphuric acid being found in the latter case. The author therefore concludes that the oxidation of sulphurous acid plays a considerable part in the reaction.—L. J. de W.

*Electro-hyposulphite* (*Hydrosulphite*) *Treatment.* [*Sugar Solutions.*] E. Urbain. Bull. Assoc. Chim. 1897, **15**, 106—115.

A complete description of the patented process of electro-hyposulphite treatment will be given in a future communication; the present paper is confined to an account of the studies made to establish the process, *viz.*, the conditions of inversion of sugar by hyposulphurous (hydrosulphurous) acid, $SO_2H_2$; on its employment, its elimination, and the degree of purification effected.

*Inversion of Sugar by Hyposulphurous Acid.*—A solution was prepared containing 32·8 per cent. of second jet sugar, and this was sulphited in a vacuum at 40° C. until it contained 1·42 grm. of sulphurous acid per litre. Eight samples of 100 c.c. each were poured into capsules containing 1 grm. of zinc in powder, and after remaining at rest for 15 minutes they were raised to temperatures of 45° to 100° C., and maintained at definite temperatures for an hour. The acid solution was then neutralised by normal soda solution and oxidised by oxygenated water. The latter precaution is indispensable, hyposulphites reducing the copper solution. Even at 90° C. no inversion was found to have taken place; at 100° C. only 0·42 grm. of glucose per 100 c.c. was found; this may have been due to the action of the zinc not having been complete, and that free sulphurous acid was still present.

*Inversion of Sugar by "Electro-hyposulphiting."*—The apparatus employed consists of a Leclanché cell, in which an alternating series of anodes of lead and cathodes of zinc are arranged, the electrodes being placed 8 mm. apart to diminish internal resistance. The vessel is heated, and a current of sulphurous anhydride, introduced by a glass tube opening below the electrodes.

The process is so arranged that the quantity of $SO_2$ introduced corresponds to the quantity of oxide of lead set at liberty by electrolysis. The source of electricity was two bichromate batteries arranged for quantity. The operation was continued for an hour, after which the liquid was filtered, the filter washed, and the solution brought to its original volume by evaporation in a vacuum, and tested for sugar and glucose, observing the same precautions as before. No inversion was found.

The decolorisation produced by hyposulphurous (hydrosulphurous) acid is remarkable and, for the same quantity of sulphur, generally much greater than with sulphurous acid, the colour of a normal syrup being 4, that of the sulphited syrup 2·9, and that of the electro-hyposulphited syrup 0·9. There seems good reason to believe that the decolorisation observed by Battut during electrolysis may have a marked influence here. Any metal convenient for purification may be used as soluble anode, but economical reasons give the preference to zinc.

The elimination of the acids of sulphur by oxidation is somewhat difficult. Hyposulphurous acid on decomposition gives thiosulphuric acid, and the hydrosulphyl which this acid contains renders its oxidation difficult; it is precisely for this reason that, as is well known, hyposulphurous acid cannot be determined by iodine. By the use of an excess of oxygenated water, however, a complete elimination may be effected, baryta being added to remove the $SO_4H_2$ formed. This elimination is much more difficult than by electrolysis, the latter alone allowing of the introduction of a metallic oxide and affording a supplementary purification as well as the elimination of the reducing agent.

By operating as described above on raw juice, and by employing anodes of lead when acting on syrups, the following comparative results were obtained:—

| — | By Double Carbonating. | By Electro-hyposulphiting of Raw Juice followed by Double Carbonating | Syrup from Double Carbonatation. | Electro-hyposulphited Syrup. |
|---|---|---|---|---|
| Sugar ......... | 10·87 | 10·92 | 45·85 | 45·85 |
| Purity ........ | 86·4 | 88·1 | 88·3 | 90·4 |
| Saline quotient | 22·2 | 23·8 | 30·8 | 31·2 |
| Alkalinity .... | 0·19 | 0·17 | Ash 1·185 | 1·17 |
| Lime salts .... | 0·025 | .. | Traces | .. |
| Colour ........ | 2·0 | 0·3 | 5·0 | 1·0 |

These results are obviously very satisfactory. Moreover, the physical condition of the syrup being absolutely changed, evaporation and boiling become very easy.—L. J. de W.

### *Rice Starch, Progress in the Manufacture of.* L. Hanemann. Chem. Zeit. 1897, 21, 982—984.

During the two last years no alterations have been made in the softening and grinding processes, and none of the suggested substitutes for caustic soda as a steeping medium have been adopted. An improved form of cylindrical sieve, however, has been introduced, built up of a series of frames bolted together, and covered separately with silk gauze. By this means a defective spot in the gauze is localised, as it were, and may be made good at comparatively small expense, smaller pieces of gauze or sound portions of old coverings being utilised for the purpose. For cleansing—an operation that is frequently too long delayed—very weak hydrochloric acid should be used, and the gauze afterwards spread out on a smooth surface and brushed with soft water.

Hundhausen, in a lecture delivered before the "Verein für Gewerbefleiss" (June 1896), has pointed out that the chief aim of the starch manufacturer must be to make his processes continuous and automatic. He attributes the fact that this object has not, in general, been realised, to the public demand for starch in pieces or rod-shaped fragments, instead of the powder form. Hundhausen further condemns the use of centrifugals as being uneconomical, and an obstacle to continuous working, and has introduced (Chem. Zeit. 1897, 21, 777) a system—almost perfectly continuous in operation—for the manufacture of wheaten starch, in which the separation of the starch from gluten, &c. is effected by means of a shallow settling gutter in combination with a suction pump. This system the author considers to be a distinct improvement, likely, in the future, to be generally introduced in the starch industry. In the case of rice starch, the gutter system would probably have to be supplemented by deep settling tanks, or centrifugals, in order to effect a complete purification of the starch.

Siemens and Halske's process (Ger. Pat. 70,012), although it has yielded excellent results in the manufacture of potato starch, has, as yet, not been applied to rice.

For moulding purposes, air pressure apparatus is now largely employed. The difficulty at first experienced, in avoiding the production of rough surfaces in the pieces, does not arise if the starch be sufficiently liquid.

Much care is required during the final drying process in order that the starch blocks may break up into well-shaped pieces. To a certain extent, success or failure may be foretold by the form of the fissures first appearing in the blocks. When the starch is dried too rapidly, the fissures formed are numerous and ragged in outline, and ragged, dull, and discoloured pieces result, which readily crumble during transit. Success is indicated by bold, well-marked cracks. If drying be conducted too slowly, the fissures, although well-formed, take a wavy course in the interior of the block, and irregular pieces result. The same thing happens when the development of the fissure is hindered by particles of gluten, fibre, &c. In practice, the blocks (planed and wrapped in paper) should be kept for 2—3 days in the drying chamber at the ordinary temperature, after which the temperature may be raised gradually, commencing at 25° C., increasing to 28° C. during the next three days, and then slowly to 32°—35° C. Starch, dried at too high a temperature during the earlier stages, loses its colour and brightness, and may become quite yellow, especially when ultramarine of a pure blue or blue-green shade has been used for staining.

Fermentation is the danger mostly to be feared at all stages, but especially when the starch is still contaminated with gluten. In the settling tanks, &c., fermentation, by reason of the bubbles of carbonic acid gas evolved, prevents the complete deposition of the starch. At a later stage (draining-boxes), when the starch is still fluid, cavities are formed in the mass. When the moisture has been reduced to 30—34 per cent., cavities are no longer formed, but the carbon dioxide, in escaping, produces fine cracks radiating in all directions, and the blocks, when dry, break up in a very irregular manner. Blocks affected in this way may be detected by the hollow sound they give out when struck. This fault, which usually may be observed in the initial stages of drying, indicates a very faulty system, *i.e.*, the starch has not been properly purified, and must be reworked

To this end the acid formed should be neutralised with a concentrated solution of $Na_2CO_3$, and the starch then stirred up with a solution of caustic soda (1·6 grms. of NaHO per litre), and again centrifugalised or otherwised purified. In warm weather it is, however, advisable to dry the starch completely, prior to such treatment, in order to kill the bacterial germs.

In practice, either sulphurous acid or caustic soda are employed to prevent fermentation. In the case of soda, the amount to be used varies according to circumstances, and in any case can be decided only by long experience.

From a financial point of view, the years 1895–96 have proved very satisfactory. The average price of rice starch during this period was 46—50 M. per 100 kilos., and the annual production 20,000 tons, of which 8,000 tons were exported.—H. T. P.

*Sorghums, A Process for the Treatment of, for White Starch and By-Products.* C. Dobrin. Zeits. für Spiritusind. 1897, **20**, 418.

The seeds of the different sorghums contain about 60 per cent. of starch, which hitherto has not been extracted as such, because, when treated by ordinary methods, a colouring matter remains on the granules. The author has devised a process (Ger. Pat. 1896, 94,954), in which the seeds, either whole or ground, are treated at a low temperature with a dilute solution (2 to 4 per cent.) of alkali peroxide, or a mixture of a caustic alkali and hydrogen peroxide. The mass is then washed with water and treated with dilute acid or an acid salt, and the starch separated from the husks in the usual manner.

By aerating the deeply coloured alkaline liquid, it becomes partially decolorised, and, on neutralisation with the acid washings (*loc. cit.*), a white gum is precipitated, serving, when washed, dried, and mixed with the decolorised husks, as a cattle food. If the brown alkaline liquid be immediately neutralised with the washings, a dark, highly nitrogenous precipitate is obtained, which can be applied as a manure.—J. L. B.

*Glutose: A New Sugar.* C. A. Lobry de Bruyn and W. Alberda van Ekenstein. Bull. Assoc. Chim. 1897, **15**, 163.

In their researches on the action of alkalis on the sugars, the authors have found, among the products of transformation of glucose, levulose, or mannose, another sugar which they name glutose. It is not fermentable, and is present in foreign molasses. Its presence will doubtless explain the indeterminate losses in cane-sugar factories and in distilleries using molasses. The publication of the results has been delayed for a year in the hope of obtaining it in a crystalline form, but so far without success.—L. J. de W.

*Caroubinose and d-Mannose.* A. van Ekenstein. Comptes Rend. **125**, [19], 719.

Effront (this Journal, 1897, 749 and 817) has described a new sugar—caroubinose. The author has prepared this according to the directions of Effront, and finds that it has all the properties of *d*-mannose.—J. T. D.

*Ammoniacum Resin, Gum from.* M. Frischmuth. Pharm. Zeits. Russl. 1897, **36**, 617.

The gum from the *Ammoniacum* resin of *Peucedanum Ammoniacum* is very much like gum arabic. Its composition is represented by the formula $2C_6H_{10}O_5.C_5H_8O_4$. When oxidised with nitric acid, it yields mucic acid and galactose, but not saccharic acid. When distilled with dilute hydrochloric acid, an amount of furfuraldehyde is produced corresponding with 16·60 per cent. of arabinose. Hydrolysis with dilute sulphuric acid yielded galactose, arabinose, a third sugar, probably mannose, and an acid.

—N. H. J. M.

*Sugar Cane and Beetroot, Direct Estimation of Sugar in.* J. Zamaron. Bull. Assoc. Chim. 1897, **15**, 74.

*See under* XXIII., *page* 75.

*Sugar in the Cane, Direct Estimation of.* H. Pellet. Bull. Assoc. Chim. 1897, **15**, 70.

*See under* XXIII., *page* 75.

*Levulose: Preparation from Mannite by Fermentation.* Vincent and Delachanal. Comptes Rend. **125**, [19], 716.

*See under* XXIV., *page* 78.

### PATENT.

*Sugar, Impts. in the Manufacture of.* A. Verley, Paris, France. Eng. Pat. 1338, Jan. 18, 1897.

The juice is rendered slightly alkaline, and submitted to the action of ozone or ozonised air until the reaction is nearly neutral. A current of sulphurous acid is passed through to completely neutralise the alkali, and the liquid is filtered.

—J. L. B.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Fermentation without Yeast Cells.* E. Buchner and R. Rapp. Ber. 1897, **30**, 2668—2678.

In order to prove that the fermentative power of yeast-juice, or zymase (this Journal, 1897, 156—157), as the authors call it, is not due to the presence of living cells (as has been suggested), the following facts are cited:—1. Yeast-juice, when passed through a Chamberland filter, retains its fermentative power unimpaired. 2. A freshly prepared yeast extract, when kept for a few days, loses its activity completely, which would not be the case if yeast cells were present. The decrease in activity is due to the action of peptonising ferments in the juice.

In comparing different yeasts, the authors find that some yield an active zymase, whilst others do not. Again, a yeast, originally active, when stored for a few days, becomes inactive, *i.e.*, yields an extract having no fermentative power whatever. From an active solution, absolute alcohol throws down a precipitate which still possesses fermentative power. Similarly, yeast-extract, evaporated *in vacuo* at 35° C., will retain its power for several months, in absence of air.

As regards the action on zymase of substances which check the action of yeast, the following observations are made:—

Zymase excites fermentation in wort containing up to 50 per cent. of solids in the shape of cane-sugar, dextrose, or glycerin; hydrocyanic acid completely checks the fermentative power of zymase, but its activity may be restored by a current of air passed through the liquid, or by addition of hydrogen peroxide. Zymase liberates oxygen from hydrogen peroxide, but loses its power of doing so if previously treated with hydrocyanic acid. The power is restored by long aeration.

A series of fermentation experiments is described, the results of which show that—

(1.) At ordinary temperatures the fermentative power of zymase (in cane-sugar solutions) diminishes rapidly after the first 24 hours, and falls to zero in 3—4 days. Compared with that of ordinary yeast, the fermentative power of zymase is very small—several hundred times less.

(2.) At higher temperatures, the action of zymase becomes at first greater, but afterwards diminishes more quickly than at low temperatures.

(3.) 1—2 per cent. of $As_2O_3$ (dissolved in $K_2CO_3$) may be added to zymase solutions, in order to destroy bacteria, &c., without sensibly affecting the fermentative capacity.

(4.) The rate of diminution in the activity of yeast-juice, when kept, depends on the temperature. At 0° C. very little change occurs in 24 hours; at 7°—8° C. the activity diminishes to less than one-half in the same period.

(5.) The activity of zymase is not influenced by some micro-organisms, *e.g.*, yeast-cells; nor is old juice (*i.e.*, inert juice) rejuvenated by the action of yeast-cells.

—H. T. P.

*Malt, The Production of Good.* W. Windisch. Wochenschr. f. Brauerei, 1897, **14**, 520—523.

The author begins by making it clear that he does not advocate the use of "forced" malts in brewing, but regards such malts as thoroughly bad, yielding, as they do, beers poor and thin in flavour, and lacking stability. Proceeding to discuss the various stages of the malting process, the author combats the prevalent opinion that a faultless malt can be produced only from the finest varieties of barley, such as Bohemian, &c. As a matter of fact, malts satisfactory in every way may be turned out from the common barleys, ordinarily available, providing they are sound.

*Cleaning and Grading.*—At this stage foreign seeds and damaged and half-corns are removed. In addition, the barley, if at all irregular, must be graded into two or even three portions, according to size of corn, each of which must be malted separately, in order that uniformity of growth may be secured. Grading is especially necessary when a mixture of small lots of barleys of different origins has to be dealt with.

*Steeping.*—The primary objects of steeping are to soften and saturate the grain with water, and to cleanse it from dust, &c. There is one point, however, generally overlooked, which the author believes to be of great importance, *i.e.*, the thorough aeration of the barley during steeping. The customary plan of changing the steep-liquor once in 24 hours is obviously inadequate; 3—4 changes daily would not be too many, and it might be advantageous, at each change, to expose the barley to the air for a short time. It is suggested, as an additional means of securing effective aeration, to force a current of air into the steep-tank, or to sprinkle water over the barley (partly exposed), by means of a rotating sparger, towards the end of the steeping process. If, in spite of all care, mould gain the upper hand on the floors, a preliminary treatment with lime will be found a cheap and effective preventive. Indeed, the author recommends its adoption in all cases. To this end, 100 grms. of unslaked lime (for each 1 cwt. of barley) should be mixed with water to a thin cream, and roused into the steep-liquor, before running in the barley. After 8—10 hours' contact, the lime-water may be drawn off and replaced by plain water. The process is stated to exert a beneficial influence on the odour of the barley (and malt), the improvement being especially noticeable in the case of bad-smelling samples.

Among the soluble constituents (carbohydrates, ash, albuminoids) of the barley—a portion of which is extracted during steeping—are included two ferments: diastase and laccase. Concerning the latter in its relation to barley, very little is known. It is possible, however—even probable—that laccase plays an important part in the respiration of the barley plant, and in the production of diastase, on the floors. It is recommended, therefore, to restrict the loss of these soluble substances to a minimum by hastening the steeping process (frequent changes of liquor, assisted by thorough aeration).

*Flooring.*—The author advocates the "slow, cool" system, in which the temperature of the malt is not allowed to exceed 19°—20° C., and is restricted during the first four days to 15°—16° C. At the low temperature, growth proceeds uniformly and healthily, and in the later stages the acrospire is obtained of the right length ($\frac{3}{4}$ to $\frac{4}{5}$ up), and a long, full rootlet development secured. Vigorous rootlet growth is of prime importance, especially in the case of barleys rich in nitrogen, in order that a sufficient proportion of those albuminoids may be withdrawn, which, if left behind in the malt, afterwards lead to "gluten" turbidity, &c., of the beer. A high flooring temperature generally results in the production of long rootlets, but they remain thin and unsound, whilst the acrospire lags behind, insufficiently developed. At the same time much sugar is produced in the interior of the corn, the malting loss becomes greater on account of increased respiration, and the albuminoids suffer far-reaching degradation. The final result is a "forced" malt. On the other hand, an "undergrown" malt is equally objectionable. Maltsters, on account of interested motives in desiring to turn out heavy malt, are particularly prone to err in this direction. Undergrown malts, although as a rule sufficiently diastatic, are in other respects very unsatisfactory, and may give rise to serious trouble in the brewery. The practice of some maltsters, of cutting down the flooring process to 7 days, or even 4 days (in America), is altogether wrong. Good modification cannot be obtained in so short a time, but requires slow, cool growth for at least 10 days. During the whole of this period the malt must, of course, contain sufficient moisture, and in order that this may be the case, sprinkling, as a rule, will be found necessary. If so, the operation should not be deferred until the malt appears withered, but be applied so soon as it appears surface-dry. In addition, it is better to sprinkle repeatedly with small quantities of water, than to apply a large quantity in one operation. In the later stages of growth the malt should furnish the necessary moisture by its own respiration. Any indication to the contrary implies the existence of a grave fault, hardly to be rectified by sprinkling, but, on the other hand, likely to be intensified thereby.

*Withering.*—That the withering process is of value, is generally conceded. Ripening, especially in the case of stubborn barleys, is carried a step further, and the malt is, to a certain extent, fore-dried.

*Kilning.*—Apart from the chemical changes taking place, the character of the malt in respect to colour, is decided on the kiln. For dark (*i.e.*, high-dried) malt, care must be taken to insure the presence of sufficient moisture at definite higher temperatures. If the malt is to be pale, it must, before curing, be dried at a low temperature, by the aid of a brisk current of air, and as rapidly as possible. In any case, it is dangerous to expose the malt, whilst still decidedly moist, for too long a period, to a temperature of 25°—37·5° C., for at this stage, the conditions obtaining on the kiln constitute an exalted "forcing" process, which, if unduly prolonged, will end in the complete ruin of the malt.

In conclusion, the author expresses the opinion that—given a good malt produced on the above lines—as good (or even better) beers could be brewed on a rational single or double mash system, as by the three-mash process at present in use (*i.e.*, in lager-beer breweries).—H. T. P.

### *Beetroot Juice, Difficulty in the Fermentation of.* G. Arachequesne. Bull. Assoc. Chim. 1897, **15**, 137—139.

The cause of the difficulty of fermentation of beet juice from abnormal beetroots, at the commencement of a campaign, remains still unsolved, as during the last season no trouble was experienced, and therefore no observations could be made. In the case of juice from normal beetroots, a similar difficulty was noted by Thomassin. The heavy rains of August and September 1896, having soaked the ferruginous subsoil of Puiseux, caused a temporary contamination with iron in the water of a well from which a distillery was supplied. The difficulty in starting the fermentation was traced to this cause, the yeast employed taking several days to become "acclimatised" to the excess of iron.—L. J. de W.

### *Cellulose, A Ferment of.* V. Omélianski. Comptes Rend. 1897, **125**, 970—973.

The author has isolated a bacillus with the power of fermenting cellulose. To initiate the fermentation, Swedish paper, cut into strips and mixed with chalk, is put into a mineral solution, and a little soil rich in vegetable *débris* added. Fermentation commences after 5—6 days at 35° C. At the end of a month a new mixture is sown with one of the strips. When the paper has reached an advanced stage of decomposition, the bacillus can be found under the microscope. The bacillus could not be cultivated satisfactorily in any of the usual media. The products of the fermentation are carbonic acid, hydrogen, acetic and normal butyric acids, with traces of valeric acid and a higher alcohol.—A. C. W.

### *Malting.* C. Hauter-Speyer. Zeits. für Spiritusind. 1897, 401.

At the commencement of the malting season, difficulties often arise owing to bad and uneven germination. The author has made some experiments with the object of overcoming this.

A fine sample of barley was taken, which, however, had a slight mouldy smell, due to being sacked too soon; it was steeped for 100 hours in clean cold water, the water being frequently changed. After eight days at 55° F., about

33 per cent. of the corns had germinated; there was an unpleasant smell, and the malt was quite unfit for use.

Three experiments were then made, in which, instead of water for the first steep, lime water was used; for the first, saturated lime water; the second, 50 per cent. lime water; and the third, 25 per cent. lime water.

The percentages of germinated corns in these three experiments were, respectively, 90·7, 94·7, and 96·6.

The author consequently recommends 25 per cent. lime water for the first steep at the commencement of the malting season.—A. L. S.

*Wort [Brewing], Composition of, during the Different Stages of Mashing; with Particular Reference to the Modification of the Albuminoids.* W. Loé. Allgem. Anzeiger für Brauereien, **13**, [42], 1249—1250.

The conclusions drawn from the experiments made by the author are summarised as follows:—

(1.) Extraction is chiefly effected during the mashing at 37°·5 C. Then follow:

(2.) Continuous formation of maltose at the expense of dextrin, and most actively between 37°·5 and 50° C.

(3.) Apparent regeneration of dextrin at the expense of non-nitrogenous extractive matter. Optimum temperature, 65°—77°·5 C.

(4.) The bulk of the albumin is dissolved at 37°·5 C., and gradually decreases up to 65° C., whereupon it remains constant.—C. S.

*Beer and Wort: Relation of Gravity to Dry Extract.* C. N. Riiber. Videnskabsselskabets Skrifter. I. Mathem. naturw. Klasse 1897; Zeits. für d. ges. Brauw. **20**, [43], 552—557.

The author describes his new drying apparatus, working with a partial vacuum, and by means of which extracts can be dried in two days at 80° C. for scientific purposes, or in 1½ hours at 100° C. for practical brewery work. He also gives details of the various sources of error in the determination of gravities, and describes a simplified pyknometer for obtaining accurate results in practice. Finally, he has compiled several extensive tables showing the true ratio between the gravity and dry extract of beers of various kinds, and prepared according to different systems of mashing.—C. S.

*Beer, A Departure in the Production of.* O. Reinke. Wochenschr. f. Brauerei, 1897, **14**, 524—525.

Referring to the increased production, during late years, of light ales, non-alcoholic herb-beers and the like, and commenting on the facilities afforded by the use of compressed carbonic acid in the bottling of such beverages, the author states that the latest departure in this direction is the introduction of carbonated wort, *i.e.*, sterilised, plain or hopped, wort, bottled with "gas" at about ¼ atmosphere pressure.—H. T. P.

## PATENTS.

*Brewing, Improved Process for.* C. Zimmer, Breslau, Germany. Eng. Pat. 26,655, Nov. 24, 1896.

The malt is divided into flour and bran, which are mashed separately, the first being boiled, whereby extracts poor and rich in diastase respectively are obtained. The flour, after further mashing with the extract from the bran, is boiled and cleared. The spent grains of this mash and the lixiviated bran are then mixed to form a third mash, and the wort extracted from this is mixed with the first obtained wort.

Combination of the worts from the first and third mashes is effected, the subsequent treatment being conducted in the usual manner.—J. L. B.

*Yeast, Manufacture of Nutritious Products from.* J. Goodfellow, Leyton, Essex. Eng. Pat. 13,722, June 3, 1897.

*See under* XVIII. A., *page* 82.

*Malt Liquors, such as Ale, Beer, Stout, Porter, and other like Saccharine Liquids, Impts. in Compositions, or Mixtures to be Used for the Preservation of, and in the Methods of Using and Applying such Compositions or Mixtures.* E. E. M. Payne, T. O. Kent, E. E. Pullman, of Aylesbury, Brixton, and Godalming respectively. Eng. Pat. 2287, Jan. 28, 1897.

The patentees employ a mixture of hydrate, sulphate, and carbonate of calcium, of which the first and last constituents may be omitted according to the nature of the liquid to be preserved. These substances are moistened, pressed into cakes, and attached to pieces of wood, cork, or other devices so that they may float in the liquid. The mixture may also be used to coat the interior of the barrel. It is stated that the salts remain practically unaltered until required through increased acidity in the liquid to which they have been added.—J. L. B.

*Ethyl Alcohol, Impts. in the Manufacture of.* E. Zdarek, Vienna, Austria. Eng. Pat. 21,878, Sept. 24, 1897.

"A method of manufacturing ethyl alcohol from cellulose or wood, consisting chiefly in boiling the wood mashed in water with sulphuric acid or sulphuric acid anhydride in quantity equal to one-tenth to 3 per cent. of the weight of the mash, or with a mixture of both these acids, from ¼ to 3 hours at a temperature of about 130° to 170° C., the pressure in the boiler being kept at about ½ to 2 atmospheres above that which corresponds with the temperature in the boiler, by forcing in or injecting an inactive gas, and after having effected the inversion by boiling, removing the acids contained in the mash and empyreumatic substances either":—(*a.*) By neutralising the free sulphuric acid with lime, and heating the mass to 118°—130° C. to remove acetic acid and empyreumatic substances. Any levulinic acid in the mash is decomposed before boiling by the addition of nitric acid. (*b.*) By converting the organic and inorganic acids into calcium salts, and decomposing these salts together with empyreumatic substances by electrolysis into carbon dioxide and ethane. The mash freed from mineral and organic acids is concentrated and fermented with yeast, the alcohol being obtained in the usual manner by distillation.—J. L. B.

*Improving or Oxidising Alcoholic Beverages, An Improved Method of.* Y. D. Möller, Wedel i/H., Germany. Eng. Pat. 23,249, Oct. 9, 1897.

By adding hydrogen peroxide solution to the alcoholic liquid it is claimed that the process of ageing is very considerably accelerated.—J. L. B.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Black Tea from Green Tea Leaf, Impts. in the Manufacture of.* S. C. Davidson, Belfast. Eng. Pat. 1124, Jan. 15, 1897.

The process claimed consists in first withering, and at the same time more or less oxidising ("fermenting") the green leaf by submitting the unrolled leaf to the action of a current of warm, moist air at a temperature of 135° to 145° F. During this operation the tea is continually lifted up and allowed to fall, and fresh air is continually admitted to replace the air from which the oxygen has been absorbed. The leaf is then rolled and dried, or the juices may be first "thickened" (with or without an intervening second rolling), and the leaf finally dried. It is optional but not preferable to also submit the leaf to more or less of the ordinary fermentation process before the final drying.
—W. P. S.

*Lard and Butter, Foreign Fats in; Detection of.* C. B. Cochran. J. Amer. Chem. Soc. 1897, **19**, 796.

*See under* XXIII., *page* 74.

*Margarin, Sesamé Oil cannot be used for the Recognition of.* Raumer. Zeits. angew. Chem. 1897, [23], 749.

*See under* XXIII., *page* 74.

*Fats and Oils, Edible, Analysing and Judging.* J. Suisse de Chim. et Pharm. **35**, 148; Bull. Assoc. Belge des Chim. **11**, 100.

*See under* XXIII., *page* 76.

*Sugar in Cocoa Preparations, Estimation of.* L. de Koningh. Zeits. f. angew. Chem. 1897, 22.

*See under* XXIII., *page* 76.

*Butter, Estimation of all the Volatile Fatty Acids in.* F. Wrampelmeyer. Landw. Versuchs-stat. **49**, 215; Chem. Centr. 1897, **2**, [19], 986.

*See under* XXIII., *page* 76.

## PATENTS.

*Preserving Alimentary Substances, A Process for.* J. Imray, London. From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis and D. A. Rosenstiehl, Paris. Eng. Pat. 1447, Feb. 18, 1897.

The process is one for preserving fruits and meat. For preserving fresh meat, it is first slightly roasted and soaked in a solution containing from 1 to 3 parts per thousand of citric, tartaric, or malic acid. It is then put in cases which are filled with carbonic acid and hermetically sealed. The closed cases are finally heated 5 or 6 times to a temperature of 45° to 55° C.

Fruits may be put in the cases raw or slightly cooked, the cases being filled up with either water, or with sweetened, acidulated, or aerated water, or with carbonic acid. After the cases are hermetically sealed, they are heated three times to 50° C., or six times to 45° C.—W. P. S.

*Yeast, Manufacture of Nutritious Products from.* J. Goodfellow, Leyton, Essex. Eng. Pat. 13,722, June 3, 1897.

Yeast is submitted to the following three processes:—1. Digestion for about 24 hours at 120°—140° F. with an equal weight of a dilute solution (0·1 per cent.) of lactic acid, or hydrochloric acid, or caustic soda or potash, the product consisting of albuminates, &c. 2. Treatment for six hours at 100° F. with an aqueous solution of 0·2 per cent. hydrochloric acid, and 2 per cent. of pepsin; the albumoses, peptones and parapeptone, and other soluble matters that are formed, are filtered off. 3. The yeast is made alkaline by the addition of an equal weight of a dilute solution of sodium carbonate, and digested for six hours at 100° F. with a glycerin extract of ox or sheep pancreas. The peptones, albumoses, and other extracts in solution are then filtered off. The residue from the third treatment may be submitted to the second process, and that from the second process to the third treatment.

The three products may be concentrated *in vacuó* and used separately or mixed as foods.—J. L. B.

*Bread, Impts. in the Manufacture of.* W. G. Smith, London. Eng. Pat. 11,913, June 19, 1897.

The inventor malts carefully selected wheat and mixes the resulting wheat malt with three to four times its weight of unmalted flour. This mixture is then converted into dough by the addition of a specially prepared ferment and sponge, the method of making which is claimed.—W. P. S.

*Butter, Impts. in the Making or Production of.* B. Willcox, London. From The Bearden Butter Company, Knoxville, Tennessee, U.S.A. Eng. Pat. 22,2[illegible]5, Sept. 28, 1897.

The improvements claimed consist in mixing equal quantities of sweet cream and sour cream, adding 1 lb. of butter to each pint of this mixture, and churning all together in an open vessel at a temperature of about 70° F. The entire contents of the churn become a solid mass, which contains all the mineral and nitrogenous constituents of the cream.

—W. P. S.

*Centrifugal Machines for Examining and Testing Milk, Sediments of Urine and other Fluid Substances; Impts. in or relating to.* F. Hugershoff, Leipzig. Eng. Pat. 24,321, Oct. 11, 1897.

*See under* XXIII., *page* 79.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Refuse from the City of Glasgow, The Treatment and Utilisation of.* Ex-Bailie Murdoch. Jour. of State Medicine, 1897, **5**, 501.

Previous to 1878, the city refuse of Glasgow was disposed of as in many other large cities, *viz.*:—collected and stored in depôts, where it accumulated, sometimes to the extent of upwards of 30,000 tons, until an outlet could be found for it. In the year mentioned, the first "refuse despatch works" was erected at St. Rollox, in which all material collected during the night was either burnt or despatched from the city in the morning.

The success was so great that a second similar works was erected in 1883, and a third in 1890. A fourth is now being erected, and a fifth is in contemplation. When all five works are in operation it is expected that the entire refuse of the city will be dealt with. The process at each works is as follows:—

Carts with refuse from ashpits and bins, on arriving, pass over a weighing machine, the weight and time of arrival being recorded, to the tipping floor, where there are various shoots to the flat beneath for the different classes of material. The ashpit refuse falls into a horizontally revolving screen. The finer portions pass through the screen into a mixing machine, which also receives a regulated quantity of excrementitious matter from a tank conveniently situated, and also a proportion of street sweepings. The whole is mixed by revolving blades, and falls into railway waggons on the siding underneath. This product forms a manure which finds a ready sale among farmers. That portion of the refuse which cannot pass through the screen is forced by the revolving process on to an endless carrier, and whilst passing along this carrier any articles of value are picked off and set aside. The remainder falls on to a range of furnaces at a lower level, in which it is consumed. The sweepings of the paved streets in wet weather (largely horse droppings) are shot into tanks with sloping bottoms, which allow the water to drain off. The settlings of these tanks are mixed with the prepared manure. Stable manure is dropped from the tipping floor direct into waggons and is sold. Mud from macadamised roads, and rubbish from the quays and wharves, are conveyed to tips on the farms of the department. The works are ventilated by powerful fans, which exhaust from the mixing chamber and force the vitiated air through pipes into the cremating furnaces.

1,259 tons of refuse per day are thus disposed of, of which about 20 per cent. is combustible matter consumed in the furnaces. Of the remainder, amounting to over 1,000 tons per day, about 52 per cent. is sold to farmers scattered over 15 counties in Scotland, and the rest is sent to tips on the Corporation farms. There is a good demand for the prepared manure, except during seed or harvest time, when the surplus is sent to the farms of the department. The manure is carried in railway waggons (of which there are 890 owned by the Corporation), at special rates fixed by Act of Parliament, and is delivered carriage free to the consumers; and, although the price has gradually diminished for some years, the revenue last year amounted to 14,000*l*. The boilers at the works are fired exclusively by cinders found in the refuse. Articles such as bones, glass, rags, iron, and other metals are separately collected and sold. Soldered tins are passed through a furnace; the solder melted off is sold, and the tins are disposed of for making box-clips. The clinkers from the furnaces are now broken up by a machine, and disposed of as road-metal and for making concrete. The waste heat from the furnaces is estimated to be equivalent to 8,960 horse-power per day of 10 hours, in addition to the requirements of the works, and arrangements are being made to utilise this heat for electric lighting or other purposes. There are four farms owned by the Corporation, situated from 10—11 miles from Glasgow, and comprising a total of 768 acres. One of these farms (Fullwood Moss) 17 years ago was a useless swamp, but by draining and the application of the refuse it has been made first class agricultural land, and has yielded for the last 10 years a profit of nearly 300*l*. per annum.

The total cost of the operations of the cleansing department for the past year was about 64,500*l.*, which is less than 4*d.* per £ on the rental of the city.—L. A.

*Deep-Well Waters derived from the Chalk.* T. Dunlop, M.B. Jour. of State Medicine, 1897, **5**, 512.

As the author thinks it is not generally known that in some districts waters derived from the chalk contain considerable quantities of sodium chloride and sodium carbonate, and sometimes but little calcium carbonate, he places on record the following analyses which he has made in Dr. Thresh's laboratory. He divides them into the following groups:—

1. *Waters containing a large Amount of Calcium Carbonate.*

| Locality. | Depth of Well. Ft. | Grains per Gallon. | | | |
|---|---|---|---|---|---|
| | | Total Solids. | Calcium Carbonate. | Sodium Carbonate. | Sodium Chloride. |
| Sudbury | 200 | 31·0 | 21·5 | .. | 5·2 |
| Bishop's Stortford | .. | 25·6 | 20·2 | .. | 2·8 |
| Saffron Walden | 50 | 46·0 | 23·0 | .. | 4·3 |
| Norwich | 340 | 37·0 | 22·0 | .. | 4·6 |
| Chatham | .. | 23·5 | 17·5 | .. | 2·9 |
| Rochester | 135 | 35·0 | 16·0 | .. | 8·5 |

2. *Waters containing a moderate Amount of Calcium Carbonate.*

| Locality. | Depth of Well. Ft. | Grains per Gallon. | | | |
|---|---|---|---|---|---|
| | | Total Solids. | Calcium Carbonate. | Sodium Carbonate. | Sodium Chloride. |
| East Donnyland | 305 | 81·5 | 8·5 | 17·5 | 41·6 |
| Colchester (1) | .. | 69·5 | 10·0 | 6·4 | 40·0 |
| Colchester (2) | .. | 72·1 | 9·8* | .. | 35·1 |
| Mistley | 400 | 55·0 | 14·0* | .. | 27·0 |
| Gosport | 222 | 67·0 | 15·5* | .. | 37·0 |

* Includes a little magnesium carbonate.

3. *Waters containing very small Amounts of Calcium Carbonate.*

| Locality. | Depth of Well. Ft. | Grains per Gallon. | | | |
|---|---|---|---|---|---|
| | | Total Solids. | Calcium Carbonate. | Sodium Carbonate. | Sodium Chloride. |
| Leigh (Essex) | 520 | 66·0 | 2·5 | 13·6 | 32·00 |
| Tiptree | 562 | 133·0 | 2·5 | 28·0 | 71·00 |
| Felsted | 404 | 67·0 | 4·5 | 18·7 | 31·50 |
| Southend | 900 | 66·5 | 3·0 | 15·5 | 38·27 |
| Colchester | 218 | 83·0 | 5·0 | .. | 40·90 |
| Maldon | 234 | 87·0 | 4·0 | 23·5 | 40·90 |
| Ilford | 292 | 34·5 | 3·5 | 13·5 | 5·10 |

In these waters, when the proportion of sodium chloride is considerable, it is usually found that the chalk is not covered by an impervious stratum. Such waters, therefore, though derived from wells of considerable depth, are really subsoil waters, or there is some direct communication with the sea or a tidal river. In the county of Essex, the majority of the deep wells derive their water from the tertiary sands lying above the chalk, and these waters are very similar in character to those obtained from the chalk in the same locality.—L. A.

### PATENT.

*Sewage and like Liquids, Impts. in and connected with the Treatment of.* J. Pratt, York, W. Prest, Ilkley, and G. B. Waugh, Bradford. Eng. Pat. 1172, Jan. 16, 1897.

This invention has for its object the automatic addition of the necessary quantities of chemicals to sewage, for the purpose of precipitation. The plant consists of a rocking-box placed over a tank containing the chemical agent in solution, and worked by a crank connected to a water-wheel placed in the current of the sewage. Each revolution of this wheel causes the rocking-box to dip into the liquid in the tank and take up a certain quantity, which then runs off into the sewage through an opening at the bottom of the box. In order to regulate the amount of solution taken up by the rocking-box, it is fitted at each end with a number of detachable tubes, curved upwards, which, when the box dips, allow the solution to run in. All the parts which come in contact with the chemical agent used, should be constructed of vulcanite. The solution runs into the above tank from a large cistern, and the flow is regulated by a ball tap in the former.—W. P. S.

## (*C.*)—DISINFECTANTS.

*Formaldehyde, Detection of.* Lebbin. Zeits. Allgem. Oesterr. Apoth. Ver. **51**, 92.

*See under* XXIII., *page* 74.

### PATENTS.

*Apparatus for Sterilising Liquids, and for Use as a Receptacle for Containing the Same, An Improved.* L. Fremy, Chalonnes-sur-Loire, France. Eng. Pat. 18,816 (under Internat. Convent.), Aug 13, 1897. In France, March 18, 1897.

The apparatus claimed consists of a tube of unoxidisable metal fitted at each end with tightly-fitting screw caps. The cap at the lower end is fitted with a tap for drawing off the liquid, whilst the upper is also provided with a tap for admitting air when the liquid is drawn off. This air is purified by passing through an antiseptic plug placed in a tube above the upper tap. The taps may be secured by lead seals after the sterilisation is completed.—W. P. S.

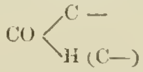
*Formaldehyde Gas, Impts. in Method and, Apparatus for Generating.* R. E. Parraga, New York, U.S.A. Eng. Pat. 25,632, Nov. 1, 1897.

The process claimed for regenerating formaldehyde gas from its solutions, consists in heating the solution and then superheating the vapour without subjecting it to pressure. In the apparatus described and claimed, the reservoir with a bottom of non-conducting material is connected by means of a valve with a coil arranged underneath, which may be heated by means of a lamp. The solution, on entering the coil, is at once vaporised; during its passage through the coil, the vapour is heated to a high temperature, thus decomposing the polymers.—A. C. W.

## XIX.—PAPER, PASTEBOARD, Etc.

*Sulphite-Cellulose Process, Theory of the.* P. Klason. Svensk kemisk tidskrift, 1897, **9**, No. 6.

The author's researches respecting the constitution of lignin show that: 1. Lignin is a glucoside, one of the constituents of which is an aromatic compound—*lignyl glycide*—containing two nuclei. 2. Lignyl glycide contains 18—22 carbon atoms in the molecule, and includes the following atomic groups—methoxyl, hydroxyl, the glycide residue $C_3H_5O$, and active carbonyl,



$$CO\begin{cases} C- \\ H\ (C-) \end{cases}$$

Lignin is decomposed by calcium bisulphite, with formation of sugar, and the calcium salt of the dibasic lignyl-sulphonic acid. Coniferin is decomposed by calcium bisulphite in an analogous manner, yielding coniferyl-sulphonic acid. The author concludes, therefore, that coniferin contains a coniferyl glycide.

If, on account of defective circulation in the cellulose boiler, sufficient lime be not present to neutralise the lignyl-sulphonic acid formed, the lignyl glycide suffers polymerisation, and is converted into a dark-brown resin, which is insoluble in sulphurous acid.—H. T. P.

### *Soda-Sulphate Cellulose.* Papier Zeit. 22, [94], 3360.

Ungerer's method of boiling wood for the manufacture of cellulose has been the subject of discussion, and a correspondent expresses surprise at Knosel's statement to the effect that 100 to 120 digesters are required to produce 20,000 kilos. of cellulose by this method. According to this correspondent, 7,000 kilos. of dry pulp have been produced daily with 10 digesters, 270 to 280 kilos. being produced from each digester. In using sodium sulphate lye, it was found useless to circulate the lye through 7 or 8 digesters as proposed by Ungerer, the lye, after leaving the fourth digester, being practically exhausted, besides being saturated with resinous matter. At the works where the writer was engaged, two batteries of five digesters each were used and only four in each battery were actually employed, the fifth remaining as a reserve; with these eight digesters, 11,000 to 12,000 kilos. could be produced, but owing to the absence of sufficient beating-engine power, only 8,000 kilos. were daily produced. With adequate lye heaters, 10 digesters are quite sufficient to produce 20,000 kilos. of dry pulp daily. The cause of this system not being more universally adopted, is probably due to the difficulty in keeping the lye-heaters in good repair. At first these heaters had to be replaced by new ones after about 10 to 15 months working. By the use of welded lye-heaters instead of riveted ones, and with an arrangement whereby the liquor entered the heater at a high temperature, better results were obtained, whereby no repairs were necessary for a period of two years. The valves employed were of iron, with sharp valve seats and rings of lead in the cones, which proved to be almost indestructible. The writer states that with the Ungerer method of working, he was able to produce a stronger, more regular, and more easily bleached pulp than he has since been able to make in mills, where horizontal, vertical, or revolving digesters are used. By Ungerer's method, the writer produced 100 kilos. of air-dry cellulose from 0·85 cb. m. of piled wood, using 160 kilos. of inferior coal, 15 to 16 kilos. of sulphate, and 35 kilos. of lime.—S. P. E.

### *Soda-Sulphate Cellulose.* Papier Zeit. 22, [97], 3470.

Referring to the previous article, a correspondent says the writer appears to overlook the fact that the digesters referred to were the small old-fashioned pots of 50 to 60 kilos. capacity. With regard to the opinion that the lye is practically exhausted after passing through four digesters, the following analyses of the lye, taken from each of the digesters of a battery of seven, is of interest.

| — | Total $Na_2O$ | Combined with Organic Matter. | Caustic Soda. | As Sodium Carbonate. |
|---|---|---|---|---|
| | Grms. | Grms. | Grms. | Grms. |
| Digester No. 2... | 874 | 251 | 298 | 325 |
| " " 3... | 729 | 543 | 31 | 155 |
| " " 4... | 657 | 579 | 22 | 56 |
| " " 5... | 611 | 552 | 9 | 50 |
| " " 6... | 605 | 558 | 3 | 44 |
| " " 7... | 598 | 582 | 0 | 16 |

The above table shows that the solution of the incrusting matter of the fibres takes place mainly in the second and third digester. The lye in the fourth digester was somewhat muddy and the bulk of the soda combined.—S. P. E.

### *Paper, Use of Talc in the Manufacture of.* Papier Zeit. 1897, 22, [87], 3099.

During the past few years the use of talc as a loading material, and for producing a satin surfaced paper, has greatly increased. Talc is a translucent mineral having a hardness of 1·0 to 1·5 and a specific gravity of 2·5 to 2·8. It is a hydrated silicate of magnesia having a formula $3MgO.4SiO_2.H_2O$. Pure talc, when ground, gives a white powder having a characteristic greasy touch. The presence of iron or other impurities gives it a greyish or yellowish hue. Clay, lime, and iron are the most frequent impurities to be found in talc, and their presence in quantities greatly interferes with the commercial value of the mineral. In Rammelsberg's handbook of mineral chemistry, the analyses are given of nine samples of talc from different parts of Europe and America, two of which, by Scheerer, are given in the following table:—

| Origin | Tyrol | St. Gothard, Switzerland. |
|---|---|---|
| Appearance | .. | Large flakes. |
| Specific gravity | 2·79 | 2·79 |
| Silicic acid | 62·12 | 60·85 |
| Clay | .. | 1·71 |
| Magnesia | 31·15 | 32·08 |
| Oxide of iron (FeO) | 1·82 | 0·00 |
| Water | 4·73 | 1·95 |

Talc, according to Rammelsberg, is also found in Russia, Ireland, North Carolina, U.S.A., Hungary, and Canada. The same author says some varieties of talc contain more than 5 per cent. of clay and iron oxide. Talc exists in large quantities in the Eastern Alps, the valleys of the Murz, the Liesing, and the Paltenbach separating the northern calcareous Alps from the Central Alps and Styria, the latter place being noted for a superior quality which is now being largely exported.

The following analyses were made by Prof. Kludy, of the Vienna Technological Museum:—

| —— | Austrian Talc. | | French Talc. | | Italian Talc. | |
|---|---|---|---|---|---|---|
| Silicic acid, $SiO_2$ | 59·59 | 92·51 | 50·91 | 75·77 | 51·23 | 83·55 |
| Magnesia, MgO | 32·92 | | 24·86 | | 33·32 | |
| Oxide of iron, FeO | 0·79 | | 2·58 | | 1·89 | |
| Lime, CaO | 0·59 | | 1·82 | | 1·80 | |
| Loss by ignition: water, $CO_2$, and organic matter. | 3·79 | | 6·64 | | 5·46 | |
| Clay, $Al_2O_3$ | 1·76 | | 13·19 | | 7·08 | |
| Alkali and difference | 0·56 | | .. | | 0·22 | |
| | 100·00 | | 100·00 | | 100·00 | |

Although the price of talc is higher than that of kaolin, it nevertheless pays to use it for high grades of paper where a better finish is required. The surface produced by talc is superior to that obtained by kaolin, and it will absorb printing- as well as writing-inks.—S. P. E.

### *Paper, Blue, White Spots in.* Papier Zeit. 1897, 22, [81], 2990.

Blue paper very frequently contains white spots. Some samples showed them irregularly dispersed upon one side of the paper, whilst the other (upper) side only showed traces of them. With the point of a knife blade the grains may be removed and examined. They may be grains of the loading material (china clay, &c.), which has been imperfectly incorporated with the fibre. Sometimes grains of calcium monosulphite are present, when imperfect sulphite pulp has been employed. Grains of sand are occasionally present when ground wood pulp has been used. This sand comes from the grinding stones, or the mineral loading may contain a large quantity. There is no difficulty in ascertaining the nature of the grains and in tracing their origin. They appear as white spots owing to the fact that they will not take the colouring matter employed for dyeing the paper.—S. P. E.

## PATENTS.

### *Paper, Impts. in Coating and Drying.* A. Masson, Wandsworth. Eng. Pat. 21,978, Oct. 5, 1896.

By the improved arrangement, one or both sides of the paper may be coated and dried in a continuous manner. The coating arrangement may be of the ordinary construction. The paper, after receiving a coat on one side, passes on a travelling web to the drying apparatus, being dried by means of jets of hot air impinging upon the coated surface. If both sides of the paper are to be coated, a duplicate set of coating and drying apparatus is provided, and the web of paper is conveyed in the usual way from the first drying apparatus to the second coating arrangement, and thence over the second drying apparatus, and finally reeled. Accompanying the specification are two drawings representing a plan and section of the arrangement adopted.

—S. P. E.

*Paper, Cardboard, &c., Treating of, with a Composition to render Waterproof, and the Markings of Writing Inks Indelible.* D. C. Simpson, Edinburgh. Eng. Pat. 24,010, Oct. 28, 1896.

The inventor describes the preparation of the composition as follows:—I mix and dissolve in one gallon of water, 6 oz. of alum, $1\frac{1}{2}$ oz. of potassium chlorate, and 1 oz. of caustic soda. Of this solution I take $\frac{1}{6}$ gall., or as much as will dissolve the following, *viz.*:—9 oz. of china clay, 6 oz. of white lead or flake white, to the consistency of cream. Of this solution I take $\frac{1}{6}$ gall. and mix with it $\frac{2}{3}$ gall. of the following solution, *viz.*:—$2\frac{1}{2}$ lb. of shellac dissolved by 12 oz. of ammonia, and the mixture diluted by a gallon of water. The solution is applied by immersion or by means of a sponge or brush, or it may be incorporated with the pulp before making the paper or board, in the proportion of $\frac{1}{2}$ gall. of the solution to 1 gall. of pulp.—S P. E.

*Plastic, Flexible, Elastic Articles, Manufacture of.* A de Pont and S. de Pont Bare. Eng. Pat. 24,790, Nov. 5, 1896.

Among a large variety of articles that may be made from this material, the inventors give the following as being suitable for ivory substitute:—

| | Per Cent. |
|---|---|
| Soluble cellulose (prepared from wood or cotton, &c.) | 30 |
| Palm oil, castor oil, or glycerin | 5 |
| Phosphate of lime | 15 |
| Bone dust, sawdust, or other powdered material | 30 |
| Sundry pigments as magnesia, alumina, &c. | 15 |
| Gum | 5 |

Aniline or other dyes in alcoholic solution may be added; the bone dust and pigments are preferably added after solution. The mass is subjected to heat and great pressure in moulds, when solid articles are to be made. When flexible materials are required, the compound is spread over the surface of a woven fabric. The inventors give the following as being suitable for such materials:—

| | Per Cent. |
|---|---|
| Soluble cellulose | 60 |
| Solvent | 25 |
| Palm, castor oil, or glycerin | 5 |
| Pigments or aniline dyes | 5 |
| Gums | 5 |

The fabric, when coated, may be dried at 220° F., which renders the material insoluble and non-inflammable. —S. P. E.

*Artificial Ivory, Impts. in.* C. Marter, London. Eng. Pat. 26,015, Nov. 18, 1896.

By this invention the bones of cattle, &c. are first cleaned and bleached with chloride of lime, and then treated with a solvent, to remove grease, &c., and afterwards sorted according to colour. By means of machinery similar to that used for working wood, the bones are reduced to shavings or thin strips, in which condition they may be further bleached if necessary. In this condition the shavings are boiled in a closed vessel until slightly softened, and the gelatin rendered to a certain extent soluble. The mass is then placed in powerful presses and compressed to the desired shape in moulds or dies, which may or may not be heated.—S. P. E.

*Pearl Hardening or Satin Spar [Finishing Paper], Impts. in the Manufacture of.* W. Brothers and W. M. Brothers, Rawtenstall, Lancaster. Eng. Pat. 1577, Jan. 20, 1897.

Natural or waste calcium sulphate is boiled with hydrochloric acid, the gypsum being added by portions as it assumes a crystalline condition in the liquid, until the contents of the vessel "become thickish." When the change is completed, the mass is expressed in a filter press, and the clear liquid is cooled in shallow vessels, to obtain an additional portion of the crystallised sulphate. The remaining acid liquor is re-used. According to a modified process, the boiling acid, diluted or otherwise, is simply saturated with gypsum, and the clear liquor set to crystallise; but this process is less productive than the former.—E. S.

*Cellulose Material, Manufacture of Hard, Dense, Horny.* C. D. Abel. Communicated by Vereinigte Köln-Rottweiler Pulverfabriken, Cologne. Eng. Pat. 18,930, Aug. 16, 1897.

The pulp is beaten in the usual way, until the fibres, as such, are destroyed, and the cellulose is reduced to an impalpable condition. In this condition it is dried in the open air, or at a gentle heat of about 40° C., forming a hard, tough mass, having a specific gravity of 1·4 to 1·5. This material the inventor terms *cellulith*, and says it can be worked with tools like wood, horn, ivory, &c.

Before drying the material, the bulk of the water may be removed by centrifugal machines or other suitable means. The hardness and density may be varied by the addition of suitable minerals to the pulp, to which also colours may be added. Dissolved shellac or caoutchouc may also be added to render the cellulith waterproof.—S. P. E.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Didymium, Contributions to the Chemistry of.* L. M. Dennis and E. M. Chamot. J. Amer. Chem. Soc. 1897. **19**, [10], 799—809.

The compound character of didymium has been fully recognised, but the attempts to separate its constituents have been very unsuccessful. The authors have experimented in this direction, using earths obtained in the usual way from monazite sand. The cerium was separated by Mosander's chlorine method: three tall 20-litre bottles were so connected in series that a current of chlorine could be passed through them all; dilute solutions of the nitrates were placed in the bottles, and when the first was saturated with chlorine (a fact which could be ascertained by shaking), it was removed, and a similar bottle filled with fresh solution was placed at the other end of the series. By continuing the process in this way, no loss of chlorine occurred, and a large amount of material could be treated in a short space of time. After the cerium hydroxide had settled, the clear solution was siphoned off and filtered, and was then free from ceria. This solution, containing the earths of the didymium and yttrium group, was then transferred to tall cylinders, oxalic acid in excess being slowly run in whilst the liquid was violently agitated. The precipitate was thoroughly washed with fresh water by decantation, whilst any didymium contained in the ceria precipitate was, after the thorough washing of the latter, recovered by re-dissolving the precipitate in hydrochloric acid, adding potassium hydroxide, and re-chlorinating. The thoroughly washed oxalates of the earths should be perfectly free from ceria; they should be dried in an air-bath, ignited in platinum dishes, and dissolved in dilute nitric acid. Earths of the yttria group were removed by stirring the dilute solution of the nitrates with a good excess of potassium sulphate crystals by means of a turbine. The resulting precipitate of the double sulphates of the didymium group, after washing and drying, were dissolved in dilute ammonium acetate solution and filtered, sufficient hydrochloric acid was added to the filtrate to decompose the ammonium acetate, and the liquid was precipitated with ammonia. The hydroxides were washed until the washings were free from sulphates, and were then dissolved in nitric acid and used for the separation experiments. In one case the yttria group was removed after a series of fractional crystallisations, and it was then found that the double sulphates of the earths at the neodidymium end were much more easily soluble in ammonium acetate than were the praseodidymium fractions.

In fractionating the earths of this group, Welsbach's process was first tried; but his account is vague, and the results were unsatisfactory. Bettendorff's method for material rich in lanthanum and poor in didymium was also unsatisfactory in this case, inasmuch as there was a large excess of didymium present; it rapidly removed samarium, however, although the spectrum absorption bands of didymium showed but little change. An accidental observation led to the abandonment of concentration by heat and rapid crystallisation in the Welsbach process, in favour of spontaneous evaporation and slow crystallisation. About a kilogram of the earths, free from ceria, was converted into the double ammonium nitrates, a little nitric acid was added, and the whole was so far evaporated that partial crystallisation took place only after 48 hours. The mother-liquor was poured off and set aside to crystallise spontaneously; the crystals were dissolved in warm dilute nitric acid, and partial crystallisation was again effected, the crystals and mother-liquor being again and again treated in the same way. After 20 fractions had been formed, all those of the same colour, shade, and intensity were united, and each such portion was fractioned separately, similar fractions being united from time to time. In this way, 105 fractions of the double nitrates were obtained. Most of the lanthanum was separated in the first few portions, but much was carried through, depositing gradually with the other earths, until, apparently, a certain relative concentration of the different earths was reached, when white crystals again separated, subsequent fractions becoming more and more pink, until the rose-red neodidymium and samarium earth salts were formed; then light-coloured crystals would again appear, followed by the same succession of colours. Thus, fractions 1 to 10 were almost pure lanthanum ammonium nitrate, 11 to 16 showed a growing intensity of the greenish prasecdidymium components, 17 was whitish, with a tinge of brownish-pink, increasing in intensity up to 24; 25 was like 17; but the next fractions showed a rapidly darkening pink colour; 30, 38, 48, 57, 72, 78, 80, and 88 were light again; but after 88 the pink colour increased to the end of the series. In all cases the light-coloured crystals showed the bands of prasecdidymium much more strongly than those of the neodidymium earths, whilst the samarium bands were almost invisible. It is evident that the amount of free nitric acid has a powerful influence upon the character of the crystals.

In practice, the nitrates of the earths are mixed with ammonium nitrate in sufficient quantity to yield 4 mols. of $NH_4NO_3$ to 1 mol. of $R_2O_3$, where R has a mean atomic mass of 142 (that of the colourless fractions was found to be 139). Sufficient nitric acid is added to form 1 part of acid to 4 of water by volume. After stirring, the solution is evaporated so that nearly all the salts will crystallise out in 24—36 hours. The small amount of mother-liquor is poured off and set aside undisturbed until a good crop of crystals is obtained; the liquid is again poured off and crystallised as before. The first large crop of crystals is dissolved in so much warm (1:4) nitric acid that crystallisation will commence after 5—10 hours; a fairly large crop is again obtained, and the mother-liquor is again fractionated. The same treatment of crystals and liquid is effected in all cases; the crystals from time to time are spectroscoped, and all those fractions giving the same absorption bands are united, irrespective of their positions in the series. A pocket spectroscope applied to the crystals lying in the dish suffices for the purpose, but success largely depends upon the judgment brought to bear upon the examination. It should be remembered, however, that the spectrum of the crystal is very different from that of its solution. As the neodidymium end of the series is approached, lanthanum ammonium nitrate is added to the fractions, with a little (1:4) nitric acid, the amount depending upon the volume of the solution and the proportion of didymium earths present.

The chief difficulty in this process lies in the deliquescence of the double ammonium nitrate compounds of the didymium earths, which prevents crystallisation in a moist atmosphere. The work is therefore best done in the winter in dry steam-heated rooms. It is best to use ammonium nitrate all through, and not to substitute sodium nitrate at the end, as Welsbach suggested. In one month, a sample of prasecdidymium almost free from neodidymium was thus obtained from 800 grms. of the mixed oxides. The presence of lanthanum at the end is most desirable. The bulk of the samarium concentrates in the last mother liquor.

—W. G. M.

### *Thoria from Ceria, Separation of.* E. Hintz and H. Weber. Zeits. Anal. Chem. 1897, **36**, 676—685. (Compare this Journal, 1896, 702.)

The authors have made an extended series of experiments to determine whether thoria can be completely separated from ceria by means of the thiosulphate method. They find that an absolute separation is impossible, since minute quantities of cerium are carried down in the thiosulphate precipitate; whilst, on the other hand, thorium in very small quantities remains behind in solution. The errors, however, are not very large, and partially neutralise each other. In any case, this method of separation is preferable to that based on the solubility of thorium oxalate in ammonium oxalate, with or without the addition of ammonium acetate.

For the separation of thoria from ceria in the analysis of incandescent mantles, which contain approximately 99 per cent. of the former and 1 per cent. of the latter, the following method gave the most satisfactory results on a small scale. A solution of nearly the above composition was made by mixing 100 c.c. of a thorium nitrate solution containing 1·5139 grm. of thoria with 100 c.c. of a cerium ammonium nitrate solution containing 0·0141 grm. of cerium dioxide. The solution was diluted to about 400 c.c. and, after the addition of 10 grms. of sodium thiosulphate, was boiled. The precipitate was filtered off and washed, dissolved in hydrochloric acid, whilst the residue of sulphur was ignited, and the ash fused with potassium bisulphate. The fused mass was then dissolved in water and some hydrochloric acid, and the solution precipitated with ammonia. The precipitate formed in this way was dissolved in hydrochloric acid, and the solution added to the main solution. After evaporation, the residue was dissolved in water and a few drops of hydrochloric acid, and the solution precipitated with sodium thiosulphate as before.

The filtrates from the first and second thiosulphate precipitates were treated with ammonia, the precipitates filtered off, washed, dissolved, and the united solutions evaporated to remove the free acid. After the addition of one drop of hydrochloric acid, the solution was again precipitated with thiosulphate. The precipitate obtained by adding ammonia to the filtrate, was dissolved in nitric acid, evaporated to dryness, and the residue dissolved in water and precipitated with oxalic acid. The weight of the washed and ignited precipitate was 0·0150 grm. of cerium dioxide. This cerium dioxide, however, still contained a small quantity of thoria, as was proved by conversion into the oxalate and treatment with ammonium oxalate. 0·0011 grm. of thoria containing a trace of ceria was dissolved. By subtracting this amount, the quantity of cerium dioxide obtained becomes 0·0139 grm.

The third small thiosulphate precipitate was found, on redissolving and reprecipitation, to contain a minute quantity —*viz.*, 0·006 grm.—of cerium dioxide.—J. S.

### *Phosphoglycerate of Lime.* Adrian and Trillat. J. Pharm. Chim. 1897, **6**, 433—438.

Commercial phosphoglycerate of lime differs widely in composition, solubility, and reaction towards litmus. The residues insoluble in water are chiefly composed of calcium phosphate and sulphate. The salt is almost insoluble in boiling water; if a cold saturated solution be warmed, precipitation begins at 32° C., is abundant at 40° C., and almost complete at the boiling point. This property affords a method of purification which serves to eliminate free glycerin, phosphoric acid, and glycerophosphoric acid.

—A. C. W.

### *Glycerophosphoric Acid, Neutralisation of, by Alkalis in the Presence of Helianthin A. and of Phenolphthalein.* H. Imbert and A. Astruc. Comptes Rend. 1897, **125**, [24], 1039.

*See under* XXIII., *page* 73.

### *Glycerophosphoric Acid, Heat of Neutralisation of.* H. Imbert and G. Belugou. Comptes Rend. 1897, **125**, [24], 1040.

*See under* XXIV., *page* 78.

### *Iodoform, Electrolytic Preparation of.* F. Foerster and W. Meves. Zeits. f. Elektrochem. **2**, 1897, 268—272.

The first description of a method of preparing iodoform electrolytically, was in Schering's patent of 1884. It was carried out by electrolysing an alcoholic solution of potassium iodide through which a current of carbonic acid was passed. About a year ago, A. Neubert investigated the method, and found that it was best to start by adding a large amount of potassium carbonate, and warming the solution to 60°—67°. He used a current-density of 3 ampères per sq. dcm. Since then Elbs and Herz have further examined the process. They work at 60° with a solution containing 6 grms. of soda, 10 grms. of potassium iodide, and 20 c.c. of alcohol, to 100 c.c. of water. They use a current-density not exceeding 1 ampère per sq. dcm., and state that the current of carbonic acid is unnecessary. It is chiefly with a view to clearing up the latter point that the present authors have repeated and extended the previous experiments. They use an electrolyte containing 60 grms. of potassium iodide, 20 grms. of soda, and 80 c.c. of alcohol in 400 c.c., at a temperature of 60°—65°. The cathodes are enveloped in parchment paper. This is the chief point in which they differ from Elbs and Herz, who used a porous cell. Some kind of partition is necessary; otherwise the iodine set free at the anode travels to the cathode, and reacts upon the caustic potash there, before it has time to make iodoform. Thus, in one experiment, in which the cathode was not enveloped, the yield of iodoform was only 49·8 per cent., whereas it rose to 97·1 per cent. when the cathode was enveloped. The parchment paper was found to last well, and the resistance which it introduces is very much smaller than that of a porous cell. The difference affects the process and the composition of the electrolyte in another way. Two equations have been proposed as representing the reaction which goes on—

$$(1)\ C_2H_6O + I_8 + H_2O = CHI_3 + H_2CO_2 + 5HI,$$
$$(2)\ C_2H_6O + I_{10} + H_2O = CHI_3 + CO_2 + 7HI,$$

of which the latter is the most probable, inasmuch as no formic acid has been detected in the electrolysed solution. During electrolysis with a porous cell, soda has to be continually added to the anode-fluid, in order to neutralise the acid formed, and to keep the solution slightly alkaline; under these circumstances, there is no object in passing in carbonic acid (which is what Elbs and Herz assert). Now, when parchment paper is used, there is far less to hinder the caustic alkali, which is formed at the cathode, from mixing with the anode-fluid and neutralising the free acid; and equation (2) shows that for 10 equivalents of iodine, we get only 9 equivalents of acid, whereas 10 equivalents of alkali appear at the cathode. It is in order to neutralise this increasing excess of alkali, that the current of carbonic acid is required. Thus there is no essential contradiction between Schering's patent and the results obtained by Elbs and Herz when using a porous cell. The presence of an alkaline carbonate favours a good yield of iodoform, whereas caustic alkali interferes with it. The amount of alkaline carbonate increases as the process continues, but it does not interfere with the yield unless it is present in such excess as to prevent the iodoform from crystallising out and collecting at the bottom of the cell.

It appears possible to convert into iodoform about 80 per cent. of the potassium iodide used. The amount of iodide present should be kept constant for a certain time, and then any further addition should be stopped, and as much as possible of the remaining salt converted. Attempts to convert the whole of the iodide present in a solution into iodoform, show that this is only possible with a great expenditure of electrical energy.—D. E. J.

*Styrolene [Cinnamene] and Metastyrolene: Their Reciprocal Transformation under the Influence of Heat.* G. Lemoine. Comptes Rend. 1897, **125**, [15], 530—533.

The same limit is reached whether styrolene or metastyrolene is heated in a sealed tube; the proportion of styrolene in the ultimate product increases with the size of the vessel, indicating that the phenomenon is similar to dissociation, and that equilibrium is reached at a definite vapour-pressure of styrolene (temperature being constant). The conversion of styrolene into metastyrolene is ultimately (after a week) complete, or nearly so, at 97° C.; at higher temperatures more styrolene remains.—J. T. D.

*Antipyrine, The Compounds of, with Aldehydes.* G. Patein. Comptes Rend. 1897, **125**, 956—959.

Antipyrine (2 mols.) forms compounds with aldehydes (1 mol.), the union taking place between carbon atoms, and not by means of nitrogen. In these compounds, which may be regarded as substituted methanes, the nitrogen atom united to the methyl group has lost its power of combining with chloral and phenols. The condensation is brought about by the action of hydrochloric acid. Formopyrine (this Journal, 1896, 372) is a compound of 2 mols. of antipyrine with 1 mol. of formaldehyde, and contains water of crystallisation. Chloral reacts with antipyrine molecule for molecule, the union taking place through nitrogen.

—A. C. W.

*Cananga Oil.* Report of Schimmel and Co., Oct. 1897, 7.

Much of the oil of cananga exported from Java, appears to be largely adulterated. A sample reputed to be pure, was found to contain not less than 25 per cent. of cocoa-nut oil, so that when placed in a freezing mixture, a butter-like mass resulted. On distillation with steam, over 30 per cent. of non-volatile residue was obtained. The following figures were obtained with pure cananga oil:—Residue on distillation with steam, about 5 per cent. (the original oil was non-congealing in a freezing mixture); specific gravity, 0·916; optical rotation, − 21; saponification figure, 17·8; solubility in 90 per cent. alcohol, soluble in ½ to 2 volumes.

—J. O. B.

*Vetivert Oil.* Report of Schimmel and Co., Oct. 1897, 57.

The following comparison between a sample of vetivert oil distilled in Réunion (presumably from fresh roots) and one distilled in Leipzig (from dried roots) shows many points of divergence:—

| —— | German Distilled Oil. | Réunion Distilled Oil. |
|---|---|---|
| Specific gravity ..... | 1·019 to 1·027 | 0·9995 |
| Rotation............ | +25° to +26° | +29° |
| Saponification factor | 70 to 80 | 22·6 |
| Solubility in 80 per cent. alcohol. | 1 in 1½ to 2 | 1 in 1½ to 2 |

It was further noted that when the oils were fractioned at 23 mm. pressure, and the first two fractions collected between 144°—164° C. and 164°—170° C., in the case of the German oil these were distinctly levogyrate, while the Réunion fractions were strongly dextrogyrate. The odour of the German oil was stronger and more persistent than that of the Réunion product.—J. O. B.

*Guaiacum Wood Oil.* Report of Schimmel and Co., Oct. 1897, 29.

This oil is obtained from the wood of an undetermined species of South American guaiacum. It is sometimes erroneously termed "Champaca" oil, although it has nothing in common with the true champaca. Guaiacum oil, which is solid at ordinary temperatures, has an extremely delicate tea-like odour, which has been found of great value in perfumery. The solid crystalline alcohol, "guaiaol," which causes the oil to have the relatively high melting point of 45° to 50° C., is practically odourless, and when partially removed, leaves the other constituent in a fluid condition, in which state it is more suitable for use in perfumery.

—J. O. B.

*Savory Oils.* Report of Schimmel and Co., Oct. 1897.

*Oil of Summer Savory (Satureja hortensis).*—The fresh flowering herb, cultivated in Saxony, gave 0·097 per cent. of oil. The specific gravity of this was 0·904, and the rotation + 0° 4′. It required nine volumes of 80 per cent. alcohol for its solution. It contained 38 per cent. of carvacrol. The odour was very aromatic; but, on account of the small yield, the oil cannot be produced at a price to permit of commercial use.

*Oil of Mountain Savory.*—This was distilled from fresh plants of *Satureja montana* cultivated in Saxony. The yield of oil was 0·18 per cent., its sp. gr. 0·939, and its rotation + 2° 35′. It gave a clear solution with 4·5 and more volumes of 70 per cent. alcohol, and with 1½ or more parts of 80 per cent. alcohol. It contained 65 per cent. of a phenol; its odour closely resembles that of common savory oil, and according to Haller, it consists mainly of carvacrol, another phenol having a higher boiling point, and a terpene.—J. O. B.

*Balsam Tansy Oil.* Report of Schimmel and Co., Oct. 1897, 60.

This oil was distilled from cultivated flowering plants of *Tanacetum balsamita*, a yield of 0·064 per cent. being obtained. The odour was balsamic, and recalled that of the common tansy (*Tanacetum vulgare*). The specific gravity was 0·943, and the rotation − 53° 18′ at 16° C., and its total acid figure 21; on standing in a cool place, paraffin-like crystals separated on the surface. The oil was not soluble in 80 per cent. alcohol, but gave a clear solution with one to two parts of 90 per cent. alcohol; on addition of more of the solvent, however, the liquid again became turbid. The oil distilled between 207° and 283° C. in the following fractions:—

From 207° to 220°, 10·4 per cent.; from 220° to 230°, 16·8 per cent.; from 230° to 240°, 18 per cent.; from 240° to 250, 12·4 per cent.; from 250° to 283° C., 21·2 per cent.; residue, 21·2 per cent.—J. O. B.

*Sicilian and Calabrian Essential Oils [Lemon, Orange, and Bergamot], Standards for.* Report of Schimmel and Co., Oct. 1897, 22.

The following characters are suggested as standards for determining the purity of the aurantiaceous essences:—

*Oil of Lemon.*—Sp. gr. 0·857 to 0·862 at 15° C. Opt. rot. (100-mm. tube) + 57° to + 67° at 20° C.

When 50 c.c. of the oil are slowly distilled in a fractionator of prescribed size, the first 5 c.c. of the distillate should have a rotation not lower by 5° than that of the original oil. Or the test of Soldaini and Berte may be employed. One-half of a sample of 25 c.c. of oil is distilled; the optical rotation of the distillate should be higher than that of both the original oil and the residue in the fractionator.

*Oil of Sweet Orange.*—Sp. gr. 0·847 to 0·853 at 15° C. Opt. rot. (in 100-mm. tube) between + 96 and + 98 at 20° C.

*Oil of Bergamot.*—Sp. gr. between 0·882 and 0·886 at 15°. Opt. rot. (in 100-mm. tube) between + 8 and + 20 at 20° C.

The amount of linalyl acetate determined by saponification with alcoholic soda, in an open flask, fitted with a reflux condenser, should not be less than 30 per cent.

The residue on evaporation on the water-bath should not exceed 5 per cent. The oil should give a clear solution with half a volume of 90 per cent. alcohol, remaining clear on the addition of more of the solvent.—J. O. B.

*Orris Root, Cultivation of, in Italy.* Report of Schimmel and Co., Oct. 1897, 38.

The following details concerning this little-known and important branch of Italian agricultural industry may prove of interest. Although the culture has been conducted in Italy for more than 200 years, and the export of the dried root is an important feature in the trade of the country, no official statistics are available with regard to it.

Orris is planted on hills and their declivities, never in valleys, mostly in sunny clearings, or lengthwise between rows of vines in vineyards, and is seldom grown in extensive fields. It grows only in dry stony ground. When set, the plants require no further care, and are left undisturbed for two or three years. Then the gathering of the rhizomes commences. The cutting, cleaning, and preparation for the market require patient and tiresome labour; generally, the root is harvested in the third year, but when prices are high and profitable, it is frequently cut in the second year of growth. When this is not the case it is preferable to leave the root until the third year, as the growth is then larger, fuller, and firmer. On the other hand, 100 kilos. of second year's roots give about 40 kilos. of dried product, while the same amount of third year's growth only gives from 30 to 35 kilos. of dry root.

The age of the rhizome may be recognised by the number of joints on the root stock. Half of the last joint remains on the living plant, which is reset after removing the portion required for drying. This is done in new ground, within 14 days of the first lifting. The old ground is left at least one year, for recovery, or may be used for raising cereals.

The fresh cut roots are first placed in water to facilitate peeling, they are then peeled and dried in the sun. This is generally accomplished within 14 days.

Statements made as to the extensive cultivation of orris in Calabria, in Southern Italy, in the neighbourhood of Reggi, prove to be unfounded. *Iris florentina* grows wild in this district, but not in sufficient quantity to make the collection of the root commercially profitable.

Orris roots from Morocco and East India have lately been brought into commerce, but are quite unfit for distillation or for perfumery.—J. O. B.

*Cerium, Determination of, in Presence of the Rare Earths.* G. von Knorre. Zeits. f. angew. Chem. 1897, [21], 685.

*See under* XXIII., *page* 72.

*Mercuric Salts, Estimation of.* L. Vanino and F. Freubert. Ber. 1897, **30**, [18], 2808.

*See under* XXIII., *page* 72.

*Chloroform, Decomposition of, in the Organism* A. Desgres and M. Nicloux. Comptes Rend. 1897, **125**, [23], 973.

*See under* XXIV., *page* 78.

*The Bergamot Crop.* Report of Schimmel and Co., Oct. 1897, 25.

*See under* Trade Rep., *page* 84.

*Rose Oil, Bulgarian.* [*Adulteration.*] Report of Schimmel and Co., Oct. 1897, 18.

*See under* Trade Rep., *page* 84.

*Theobromine, Some Derivatives of.* H. Brunner and H. Leins. Ber. **30**, [17], 2581.

*See under* XXIV., *page* 78.

*Santonin, New Reaction of.* A. Jaworowsky. Pharm. Zeits. Russl. 1897, **36**, 559; through Chem. Zeit. Rep. 1897, **21**, [91], 269.

*See under* XXIII., *page* 75.

*Opium, Quantitative Estimation of Starch in.* L. F. Kebler and C. H. Lawall. Amer. J. Pharm. 1897, 234; Chem. Centr. 1897, **2**, [19], 985.

*See under* XXIII., *page* 77.

*Oil of Roses, Estimation of.* F. Dietze. Süddeutsch. Apoth. Zeit. 1897, [89]; Chem Zeit. **21**, [95]; Chem. Rep. 288.

*See under* XXIII., *page* 75.

## PATENTS.

*Piperazine [Salts], Impts. in the Treatment of.* W. B. Bishop and F. W. Passmore, London. Eng. Pat. 26,078, Nov. 19, 1896.

Piperazine tartrate and citrate are constant in composition, non-hygroscopic, and may be dried at a high temperature without loss. The preparation of these salts by mixing the solutions of acid and base, or by melting them together, is claimed; 3 mols. of piperazine to 2 mols. of citric acid or 1 mol. each of base and tartaric acid are employed.

—A. C. W.

*Alkylated Uric Acid, Impts. in the Preparation of.* C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 656, Jan. 9, 1897.

The preparation of alkyluric acids by the action of halogen alkyls upon salts of uric acid or alkyl uric acids suspended in water, or upon these substances suspended in water in the presence of salts of feeble organic or inorganic acids (acetic, carbonic, boric), is claimed.—A. C. W.

*Hydroxycaffeïne, Impts. in the Manufacture of, by the Direct Methylation of Uric Acid or of* 1.3-*Dimethyluric Acid.* J. Y. Johnson, London. From C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 2472, Jan. 29, 1897.

Hydroxycaffeïne has been shown to be identical with 1.2.3-trimethyluric acid (E. Fischer, Annalen, **215**, 268, and this Journal, 1882, 200). The patentees claim the processes for preparing it, which consist in treating an alkaline solution of uric acid or of 1.3-dimethyluric acid with a methylating agent at a low temperature, in the first case at least 4 mols. of alkali (caustic potash) being present to 1 mol. of uric acid, in the second at least 2 mols. to 1 mol. of the dimethyluric acid.—A. C. W.

*Nitroalphylpyruvic Acids, Manufacture of.* Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 2,556, January 30, 1897.

The manufacture of *o*- and *p*-nitrophenylpyruvic acids by the condensation of nitrotoluenes or their homologues in the *o*- and *p*-positions, with the esters of oxalic acid in the presence of sodium ethylate or methylate is claimed, and the compounds mentioned as new products.—A. C. W.

*Iodine in Mineral Oils, A Method for Dissolving.* F. W. Klever, Cologne, Germany. Eng. Pat. 5256, Feb. 26, 1897.

"A process for the manufacture of a solution of iodine in mineral oils characterised by the fact that heavy mineral oils of the specific gravity of 0·89 to 0·90, if desired, with the addition of fatty oils or oleic acid mixed with alkalis, under pressure if necessary, are oxidised, and then in the resultant mass the iodine is dissolved." The solution of iodine so prepared is also claimed.—A. C. W.

*Guaiacol Compounds* [*Alkaloid Salts of Sulphonic Acid*], *A New or Improved Manufacture of.* G. L. Schaeffer, Brooklyn, U.S.A. Eng. Pat. 8227, March 30, 1897.

The patentee claims compounds of alkaloids (quinine) and guaiacolsulphonic acid—

$$C_6H_3(OH)(OCH_3)SO_3H,$$

these salts do not possess the odour and caustic properties of guaiacol itself. The normal quinine salt is—

$$(C_6H_4O_2.CH_3.HSO_3).C_{20}H_{24}N_2O_2.$$

The acid salt contains 2 mols. of guaiacol.—A. C. W.

*Ozone, the Manufacture or Production of* [*"Open Ozonisers"*], *Impts. in and connected with, and in Apparatus therefor.* E. Andreoli, London. Eng. Pat. 15,813, July 2, 1897.

*See under* XI. A., *page* 53.

## XXI.—PHOTOGRAPHY.

*Phenols, Poly-substitution, Developing Power of; Influence of the Ketone Group on.* A. and L. Lumière and Seyewetz. Bull. soc. franc. de Phot. et Photogr. 1897, **7**, 166; through Chem. Zeit. Rep. 1897, **21**, [91], 276.

It is well-known that the entrance of the acid group CO.OH into an aromatic compound will lessen or even destroy the developing power of the body. The authors have now examined into the influence of the ketone group in the same direction. They find that the entrance of a ketone group into the structure of an aromatic compound which on account of its phenol-like character, is capable of acting as a developer, does not lessen its developing power if the ketone group is united with an aliphatic residue, or is connected with another aromatic group containing no phenol group; but the developing power ceases if there be a hydroxyl in both.—W. G. M.

### PATENT.

*Photographic Printing Paper, Manufacture of an Improved.* Drs. M. Jolles and L. Lilienfeld, Vienna. Eng. Pat. 30,161, Dec. 31, 1896.

Instead of albumins, gelatins, &c., hitherto used, the inventors employ proteids containing phosphorus, known by the various names of nucleo-proteids, nucleo-albumins, nucleo-globulins, nucleins, para-nucleo-albumins, para-nucleo-proteids, &c., which compounds contain the phosphorus in organic combination. A suitable proteid or a mixture of several, is dissolved in an alkali, alkaline earth, or salt thereof either in a warm or cold state. For ordinary purposes a 10—12 per cent. solution of the proteids in a 3—6 per cent. solution of the salts will be found suitable. A small quantity of alcohol is advisable under certain circumstances. The proteid employed may be tanned. The solution so prepared is salted in the ordinary way with a haloid and may or may not be coloured, and filtered and applied to the paper in a hot, warm, or cold state. The inventors further describe a variety of ways of applying the proteids to the paper as well as a number of combinations in which the material can be applied and used.—S. P. E.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

*Explosives or Explosive Compounds* [*Nitrated Cellulose*], *Impts. in, and in the Manufacture of.* A. E. Pike and W. H. Thew, both of London. Eng. Pat. 25,704, Nov. 14, 1896.

This patent is for an explosive produced by nitrating cellulose, obtained from the potato refuse resulting from the manufacture of potato flour. The refuse is washed, ground, and sifted and may be treated with hot dilute sulphuric or hydrochloric acid to remove the starch and other foreign matters.—W. M.

*Explosives* [*Chlorinated Dinitrobenzene*] *especially applicable for Use in Coal Mines, Impts. in the Manufacture of.* W. J. Orsman, Gathurst, Wigan. Eng. Pat. 29,598, Dec. 23, 1896.

This explosive is made by adding an equal weight of melted chlorinated dinitrobenzene to dry powdered cellulose and incorporating together at a steam heat. When cold, 3 to 5 times the weight of ammonium nitrate is added to this mixture, the whole forming a bulky explosive stated to be safe to use in coal mines and those which are fiery.—W. M.

*Matches, Impts. in, and in the Method of Manufacturing same.* B. Baron, London. Eng. Pat. 29,670, Dec. 24, 1896.

Fine wood fibres obtained by shredding, are twisted, spun, or woven into a rod or cord, which is coated with spermaceti, cut into lengths, and tipped in the usual way.—W. M.

*Explosives, Improved Manufacture of.* [*Ammonium Nitrate, &c.*] M. Bielefeldt, Wittenberg, Saxony. Eng. Pat. 2557, Jan. 30, 1897.

This explosive consists of ammonium nitrate mixed with 4 or 5 per cent. of resin and 4 or 5 per cent. of nitrates of sodium, barium, calcium, or strontium, or mixtures of their nitrates. It is claimed that the addition of the last-named ingredients increases the safety of the explosive.—W. M.

*Blasting Compositions* [*Bichromates*], *Impts. in.* H. Ritter von Dahmen, Breslau, Silesia. Eng. Pat. 3135, Feb. 5, 1897.

The use of chromates and bichromates in the production of nitroglycerin explosives is claimed. The following mixture is said to give good results:—Nitroglycerin, 30 parts; sawdust, 35 parts; ammonium nitrate, 30 parts; potassium bichromate, 5 parts.—W. M.

*Gunpowder, An Improved Manufacture of.* J. C. Chapman, London. From C. W. Volney, New York. Eng. Pat. 25,204, Oct. 30, 1897.

The process described is for providing grains of gunpowder containing trinitrocellulose with a surface coating of dinitrocellulose, by reducing the trinitrocellulose on such surface to dinitrocellulose, by means of reducing agents, such as alkaline sulphites. The object is to form a slow-burning coating over a quick-burning core.—W. M.

*Gunpowder, An Improved.* J. C. Chapman, London. Communicated from C. W. Volney, New York. Eng. Pat. 25,413. Nov. 2, 1897.

The explosive consists of nitrocellulose, basic Rosaniline or its homologues (as Chrysaniline or Leucaniline), and trinitrobenzene, or a trinitro compound of the benzene series. Forty parts of trinitrobenzene are dissolved under pressure, and then mixed with a solution of 55 parts of nitrocellulose and 5 parts of basic rosaniline, and after removal of excess of solvent, pressed into shape in the usual manner. – W. M.

# XXIII.—ANALYTICAL CHEMISTRY.

## *APPARATUS, ETC.*

*Filter Paper, The Reaction of.* L. Magnier de la Source. J. Pharm. Chim. 1897, **6**, 438—440.

R. Gans observed that when certain organic acid solutions were warmed with filter paper, a portion of the acid disappeared. The author, having noticed the opposite result, has investigated the reaction of various filter papers. Ordinary filter-paper (12 cm. diameter) saturates 0·0015 grm. of sulphuric acid. French Berzelius paper appears to be entirely neutral. Schleicher and Schüll's paper, washed with hydrochloric and hydrofluoric acids, has an acidity equivalent to 0·0014 grm. of sulphuric acid. A Swedish Berzelius paper made more than 50 years ago has no acidity equal to 0·0003 grm. of the same acid. Thus, in determinations of tartar, a very small quantity of acid in the paper leads to an appreciably augmented result. For such use, every trace of acid should be eliminated from the paper by long washing with boiling water.—A. C. W.

*Stove (Electrical) for Laboratory.* M. D. Sohon. J. Amer. Chem. Soc. 1897, **19**, 790—792.

The copper stove (Fig. 1) is 3 in. in height, exclusive of the legs, and is 7½ in. in diameter. The top is fitted with the usual concentric rings, while the bottom is left open. About 2 in. from the top is a heating plate attached to the legs independently of the body, and readily detached, and having a clearance of ¼-in. all round between it and the wall of the stove. The heat is derived from the coils B (Fig. 2) of rheostat wire, imbedded in silicate insulation and resting on a ½-in. asbestos board C. This is covered with a copper protecting plate D. The coil is in two unequal parts, so that three different temperatures (preferably 90°, 110°, and 125° C.) may be obtained, according to the manner of applying the current by means of the switch E. By using suitable coils or by interposing a resistance, the stove may be used with liquids more volatile than water. It is especially recommended for the distillation of, or for extractions with inflammable liquids. A group of such stoves independently connected, is highly preferable to the large water- or steam-baths generally employed.—W. G. M.

Fig. 1.

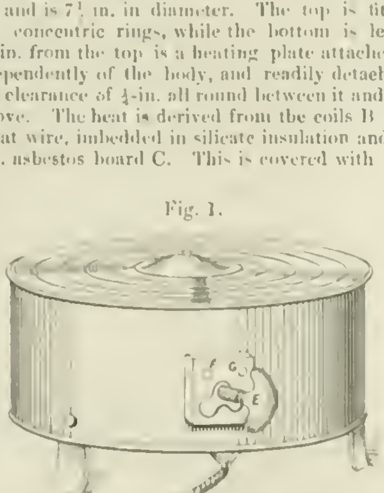
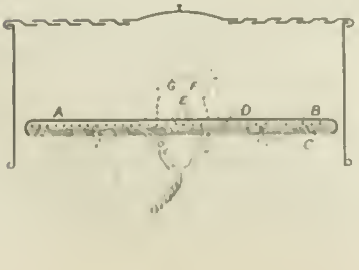


Fig. 2.



### PATENTS.

*Centrifugal Machines for Examining and Testing Milk, Sediments of Urine, and other Fluid Substances; Impts. in or relating to.* F. Hugershoff, Leipzig. Eng. Pat. 23,321, Oct. 11, 1897.

The pan or tray of the machine is fixed on a spindle provided with a pin, which is adapted to be engaged by a clutch or tooth on a driving drum loosely mounted on the spindle. The power is applied to the drum by a band or cord, with a slight downward tendency, which moves the drum downwards, against the action of a spiral spring, until it engages with the pin on the spindle. "By means of this arrangement a reciprocating action of the drum imparted, may be caused to convey a continuous rotary motion to the spindle and pan or dish."—R. A.

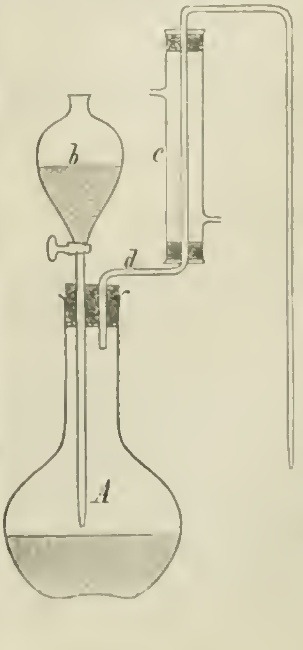
## *INORGANIC CHEMISTRY.—QUALITATIVE.*

*Sulphurous Acid, Reduction of, to Sulphuretted Hydrogen; also Arsenic and Antimony, Detection of, in Presence of much Tin, and of Arsenic in Commercial Sulphuric Acid.* E. Donath. Zeits. Anal. Chem. 1897, **36**, 663—665.

Hot stannous chloride solution containing free hydrochloric acid, reduces sulphurous acid to sulphuretted hydrogen, probably in accordance with the equation—

$$3SnCl_2 + SO_2 + 6HCl = 3SnCl_4 + 2H_2O + H_2S.$$

When the solution does not contain too much free acid, stannic sulphide, or a mixture of this with stannous sulphide, is precipitated. In order that this reaction shall take place, certain precautions must be observed; otherwise sulphur will be produced by the action of the sulphurous acid on the sulphuretted hydrogen. A continuous current of $H_2S$ may be obtained in the following way. A flask A contains a solution of fresh tin-salt in a mixture of one volume of concentrated hydrochloric acid and one and a half volumes of water. The strongly acid solution is heated to near the boiling point, and a solution of sodium sulphite or bisulphite slowly dropped in from the tap funnel *b*.



*Detection of Arsenic and Antimony in presence of large Quantities of Tin.*—The solution to be examined is mixed with a strongly acid solution of stannous chloride, brought nearly to the boiling point, and sodium sulphite or an aqueous solution of sulphurous acid slowly added. Arsenic and antimony sulphides are precipitated, whilst the tin remains in solution.

*Detection of Arsenic in Commercial Sulphuric Acid.*—10—15 c.c. of the acid are mixed with an equal volume of water, added to a strong hydrochloric acid solution of stannous chloride, and treated as above. If arsenic be present, a distinct separation of finely-divided yellow arsenic sulphide takes place after heating some time, which is not to be confused with the opalescence due to sulphur which may possibly be precipitated.—J. S.

*Copper, Phenol as a Delicate Reagent for.* A. Jaworowsky. Pharm. Zeits. Russl. 1897, **36**, 529.

The following observations prove an ammoniacal solution of phenol to be a very delicate test for copper. A solution of copper sulphate (1 in 10,000) yields a pale blue coloration in 20 minutes, changing to dirty blue in 1 hour, to dark violet-blue in 3 hours; 1 in 100,000, distinctly blue in 20 minutes when viewed in column, greenish in 1 hour, brownish-blue in 3 hours, dark green in 18 hours; 1 in 1,000,000, pale blue after 18 hours. With a still weaker solution, 1 in 10,000,000, a very faint reaction was obtained in 24 hours. Potassium ferrocyanide produced no coloration in either of the two last-mentioned solutions.

—H. T. P.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Permanganate Solutions, Stability of, when preserved under Vaseline Oil.* Meineke and K. Schröder. Zeits. f. öffentliche Chem. **3**, 5.

The evaporation of permanganate solutions may be prevented by covering with a layer of vaseline oil. A minute amount of peroxide separates at the interface, but after standing two months, this was found to have no appreciable effect on the strength of the solutions. The authors find that commercial vaseline oil is preferable to the light petroleum spirit recommended by Topf, since the latter absorbs oxygen and carbonic acid. Solutions of potassium cyanide, sodium thiosulphate, &c. may be preserved in the same way.—J. S.

*Silicates, Analysis of.* A. Leclère. Comptes Rend. 1897, **125**, [22], 893–894.

The silicate is mixed with three parts of lead oxide or less, in presence of alkali, and heated in a muffle, in a small covered platinum capsule, for about half an hour at an orange-red heat. If the silicate be not very finely powdered, it may be necessary to add more lead oxide and to prolong the heating. On cooling the capsule quickly, the contents solidify and at the same time become detached from the capsule. It is then treated, either cold or, better, at about 40°, with at least 10 parts of a mixture of equal amounts of ordinary and fuming nitric acid. If the substance is first powdered, the action is complete in an hour, otherwise a day or two may be required. The acid is then diluted with boiling water to dissolve the lead nitrate, filtered, and washed. The silica is ignited at a very high temperature, and weighed.

The filtrate is concentrated to get rid of the acid, and treated, first with alcohol, and then with slightly more hydrochloric acid than is necessary to precipitate the lead. It is then filtered, evaporated to dryness, and the bases determined by the methods of Sainte-Claire Deville and Schloesing.

*Preparation of Lead Oxide.*—In order to avoid the platinum employed in the above method being acted on, pure lead oxide must be used. This is prepared as follows: —A solution of pure commercial lead nitrate (15 per cent.) is treated with a saturated solution of oxalic acid containing 3 per cent. of nitric acid. The dense precipitate of lead oxalo-nitrate is separated, dried, and ignited below a red heat in a porcelain dish. A quarter of it is soaked with nitric acid, mixed with the rest, and the whole is re-ignited. The product is a very fine powder, suitable for attacking silicates.—N. H. J. M.

*Nitrous Acid, A New Gas-Volumetric Method for the Estimation of, and other Substances which can be Oxidised by Hydrogen Peroxide.* E. Riegler. Zeits. anal. Chem. 1897, **36**, 665—668.

The method depends on the fact that nitrous acid is rapidly and completely oxidised to nitric acid by hydrogen peroxide, thus:—$HNO_2 + H_2O_2 = H_2O + HNO_3$. One molecule of nitrous acid requires one atom of oxygen. The quantity of nitrous acid can therefore be calculated from the volume of oxygen used. One c.c. of oxygen, measured at 0° C., and 760 mm., corresponds to 0·0034 grm. of nitrous acid in the form $N_2O_3$. In order to determine the quantity of oxygen used up, two experiments are necessary. A blank experiment is first made to ascertain the volume of oxygen given off on treating a definite measured quantity of the hydrogen peroxide with $H_2SO_4$ and $KMnO_4$. For this purpose the ordinary form of Knop-Wagner azotometer or the modification described by the author, may be used.

5 c.c. of hydrogen peroxide, containing not more than 1—1·2 per cent. of $H_2O_2$, 30 c.c. of distilled water, and 10 c.c. of pure concentrated sulphuric acid are placed in the generating vessel, whilst the inner receptacle contains a few crystals (0·1 grm.) of $KMnO_4$. The vessel is then shaken until a permanent red coloration is produced, and the liberated oxygen measured under the existing conditions of temperature and pressure. Let this volume of oxygen reduced to 0° C. and 760 mm. be $V_0$.

The apparatus is then washed out and 30 c.c. of the solution of nitrous acid to be examined (which must not contain more than 0·15 per cent. of $N_2O_3$) is introduced 5 c.c. of the same hydrogen peroxide solution are added and 1—2 drops of sulphuric acid. After shaking and waiting about 5 minutes, 10 c.c. of concentrated sulphuric acid are added in small portions at a time. A few crystals of potassium permanganate are now placed in the inner vessel and the operation conducted as before. If the reduced volume of oxygen given off in this case, is $v_0$, then—

$$\left(\frac{V_0 - v_0}{2}\right) \times 0{\cdot}0034 = (V_0 - v_0) \times 0{\cdot}0017$$

represents the quantity of $N_2O_3$ in the solution examined.

The author gives test analyses. The same method may be used to determine the amount of ferrous iron present in a solution. Silver oxide may also be substituted for the potassium permanganate, in which case the procedure is as follows:—10 c.c. of the 1 per cent. $H_2O_2$ solution, 40 c.c. of water, and 15 drops of a 15 per cent. solution of caustic potash are mixed in the generating vessel. The inner receptacle contains 5 c.c. of a 3 per cent. solution of silver nitrate. The rest of the operation is then conducted in the same way as before, except that the $N_2O_3$ is represented by $(V_0 - v_0) \times 0{\cdot}0034$, since, in this case, the whole of the oxygen is derived from the hydrogen peroxide.

—J. S.

*Basic Slag, Estimation of Citrate-soluble Phosphoric Acid in.* O. Böttcher. Chem. Zeit. 1897, **21**, [95], 993–995.

The method, which was already described (*ibid.*, p. 168), consists in the preparation of a citrate extract (Wagner), (this Journal, 1897, 1044), treatment of 50 c.c. of the extract as soon as possible (the same day, at the latest) with 50 c.c. of Maercker's citrate solution and 25 c.c. of magnesia mixture (Maercker). The whole is then at once shaken for 30 minutes, and filtered, as soon as possible, through a Gooch crucible. The following table shows that concordant results are obtained when the precipitation takes place within six hours, but not after 24 hours:—

| —— | 1. | | 2. | |
|---|---|---|---|---|
| | $Mg_2P_2O_7$ | $P_2O_5$ | $Mg_2P_2O_7$ | $P_2O_5$ |
| | Grm. | P.c. | Grm. | P.c. |
| Molybdate method .... | 0·1061 | 13·58 | 0·1186 | 15·18 |
| Citrate method : | | | | |
| Fresh extract .... | 0·1056 | 13·52 | 0·1190 | 15·23 |
| After 2 hours .... | 0·1058 | 13·54 | 0·1194 | 15·28 |
| „ 4 „ .... | 0·1062 | 13·59 | 0·1200 | 15·36 |
| „ 6 „ .... | 0·1075 | 13·76 | 0·1208 | 15·46 |
| „ 24 „ .... | 0·1392 | 17·82 | 0·1421 | 18·19 |

With regard to the separation of silica, in the case of slags unusually rich in silica, Wagner (this Journal, 1897, 1044) states that when the fresh citrate extract is treated with alkaline citrate and magnesia-mixture, the precipitate is quite free from silica, and that it is immaterial whether filtration is at once proceeded with, or after two hours. The author, however, maintains that it is important in such cases, to filter immediately, and gives some results showing a considerable increase in the weight of the precipitate after two hours.—N. H. J. M.

*Mercuric Salts, Estimation of.* L. Vanino and F. Treubert. Ber. 1897, **30**, [18], 2808—2809. (This Journal, 1897, 1013.)

Phosphorous acid, after 12 hours' standing in the cold, completely reduces mercuric chloride to calomel. Warming would cause the formation of metallic mercury. In the presence of hydrogen peroxide, the precipitated calomel remains unchanged, even on the water-bath, and the reaction is complete in a few minutes. In an estimation, excess of hydrogen peroxide is added, then phosphorous acid, and the mixture warmed on the water-bath until the precipitate is flocculent, when it is collected on a filter, and dried at 105° C. The use of other oxidising agents, *e.g.*, nitric acid and chlorine water, is not practicable, since these added in excess would dissolve the precipitated calomel.
—A. C. W.

*Basic Slag, Estimation of Citrate-soluble Phosphoric Acid in.* M. Passon. Zeits. angew. Chem. 1897, 746—749.

The proposed method is based on the fact that, in estimating phosphoric acid in slags, results identical with those obtained by Wagner's method can be obtained by employing citric acid solutions of different composition, provided that the acidity of Wagner's solution is maintained. The amount of alkali citrate present in the solution, is of less importance.

It was previously established (Chem. Zeit. 1896, No. 11) that in most cases, a solution of citric acid (14 grms. per litre) gave results agreeing with those obtained with Wagner's solution. In the few cases in which too high results were obtained, the addition of a little ammonium citrate (about one-tenth the amount used by Wagner) caused the results to correspond with the Wagner results.

Further experiments showed that the addition of tripotassium citrate had an effect similar to that of ammonium citrate, in lowering the results, which came out too high according to the Wagner method; and, finally, a solution was adopted which contained 140 grms. of crystallised citric acid and 30 grms. of tripotassium citrate per litre. In 20 determinations made with this solution, the results agreed well with those obtained with Wagner's solution: all were within 0·3 per cent.; in 12 cases there was less than 0·1 per cent. difference, and in 3 cases 0·25 per cent. difference. When kept for a short time the phosphate solution frequently gives a precipitate of calcium citrate, but it was found that this does not affect the results in the least.

Tripotassium citrate was prepared by adding a slight excess of caustic potash to a solution of citric acid, and evaporating on the water-bath until crystals begin to separate. The liquid was then allowed to cool, being stirred all the time, filtered, washed with as little water as possible, then with alcohol, and finally with ether. The crystals contain 1 mol. of $H_2O$.

On the whole, the author considers that the general employment of citric acid (1·4 per cent.) would be the most satisfactory, as the results obtained with it nearly always agree with the Wagner results; or, since the Wagner solution is said to give slightly low results, as compared with those of vegetation experiments, a solution containing 150 grms. of free citric acid per litre (using 50 c.c. for each determination) might be suitable.—N. H. J. M.

*Silver Assay, A Modified Method of.* A. E. Knorr. J. Amer. Chem. Soc. 1897, **19**, 814—816.

To obviate the tediousness of the last stage in the Gay-Lussac silver test, the author employs Volhard's thiocyanate process at the end. 1 grm. (about) of the silver is dissolved in an 8-oz. bottle with a good stopper, using chlorine-free nitric acid diluted with two volumes of water; after heating until all nitrous fumes have been expelled, enough water is added to prevent crystallisation of silver nitrate, and precisely 100 c.c. of the standard salt solution are run in. The stopper is then inserted, and the solution is shaken until the silver is collected; it is then filtered off, 5 c.c. of ferric ammonia alum are added as an indicator, and the standard thiocyanate solution (of which 1 c.c. = exactly 1 mgrm. of pure silver) is run in from a burette in the cold until the characteristic colour of the ferric thiocyanate is permanent. Proof tests with fine silver are conducted simultaneously. The titre of the salt solution is found by deducting the number of c.c. of the thiocyanate used for a proof test from the number of mgrms. of the fine silver initially weighed out. The fineness of the assays is then calculated by adding the number of c.c of thiocyanate used in any assay to the standard of the salt solution thus found. Copper and other impurities do not interfere; filtration from silver chloride is necessary, and the wash water must be free from chlorine. The limit of accuracy of the assay is 0·2 to 0·3 mgrms.—W. G. M.

*Cerium, Determination of, in presence of the Rare Earths.* G. von Knorre. Zeits. f. angew. Chem. 1897, [21], 685—688.

The reduction of ceric to cerous salts by hydrogen peroxide takes place in a previously acidified solution with rapidity and completeness, according to the equation—

$$2Ce(SO_4)_2 + H_2O_2 = Ce_2(SO_4)_3 + H_2SO_4 + O_2.$$

Accordingly, to the acidified solution of a weighed quantity of the ceric salt is added a measured volume of dilute hydrogen peroxide solution of known strength, at the ordinary temperature, and the excess of peroxide is determined by titration with potassium permanganate solution. The end point of the reaction is quite sharply seen, though Rose has stated that an acid solution of a cerous salt slowly decolorises permanganate. A freshly prepared ceric solution is reduced by hydrogen peroxide almost instantaneously, but one which has been prepared for some time requires perhaps a quarter of an hour for complete reduction (yielding, however, then the same result as a portion of the same solution titrated fresh). The reaction may be hastened with such solutions, by heating them to boiling after the addition of acid; but if the acid be added only after the heating, the results are always too high. Either sulphuric or nitric acid may be used to acidify, and the results are not affected at all by the presence of filter-paper in the solution, so that filtration exerts no reducing action on the ceric salt. The solution of permanganate should not contain more than 2 grms. of $KMnO_4$ per litre; and the hydrogen peroxide solution used by the author is of such a strength that 20 c.c. correspond to 25 c.c. of the permanganate solution. A number of test analyses are given, which show the accuracy of the method as applied to pure salts of cerium. The author promises a further communication.—J. T. D.

*Sodium Oxalate, Use of, in Volumetric Analysis.* S. P. L. Sörensen. Zeits. Anal. Chem. 1897, **36**, 639—643.

The author recommends the use of normal sodium oxalate as a source of sodium carbonate for determining or adjusting the strengths of normal acids employed in volumetric analysis.

The pure substance is easily obtained in the following way:—One part of commercial sodium oxalate is dissolved in about 32 parts of water (this is a saturated solution at about 15° C.), and made slightly alkaline with caustic soda. The solution is allowed to stand in a tall glass cylinder until it has become quite clear, when it is filtered from any impurities, such as calcium oxalate, which may have separated out. After evaporating the filtrate on a water-bath to one-tenth of its original volume, sodium oxalate crystallises out, leaving the more easily soluble impurities, such as potassium oxalate, caustic soda, chlorides and sulphates, &c., in the mother-liquor. The crystals of sodium oxalate are then filtered off, pulverised, and washed several times with cold water with the aid of a filter-pump. A single recrystallisation is usually sufficient to prepare the salt in a state of purity. If the mother-liquor, however, should not be quite clear, or if it contain traces of alkalis or sulphates, this operation must be repeated as often as is necessary. Sodium oxalate contains no water of crystallisation, and can safely be dried at 125°—150° C. It can be easily and accurately weighed out, and since it is not hygroscopic, the amount of available sodium can be exactly ascertained. The weighed quantity for analysis is converted into sodium carbonate by careful ignition in a platinum crucible, provided with a well-fitting lid in order to prevent loss by decrepitation.

This source of error may be removed by filtering a warm saturated solution of the substance into alcohol, filtering off the precipitate, and drying at 125°—150° C. as before. When carbon is formed during the ignition, it must be removed by igniting with the lid partially on. After cooling, the crucible, with its contents, is placed in a tall beaker, and water and a slight excess of normal acid added. The analysis is then completed in the usual manner.

Test analyses, which are given, show the accuracy of the method. The author also prefers sodium oxalate to oxalic acid or ammonium oxalate for adjusting the strength of potassium permanganate solutions, since these substances are more liable to effloresce on keeping.—J. S.

*Phosphorus, Estimation of, in Steel, Iron, and Iron Ores.* J. Ohly. Chem. Zeit. 1897, **21**, 139—140.

The following modification of the molybdate method gives results equal to those attainable gravimetrically, but enables them to be obtained in about five minutes. 2 grms. of the steel are dissolved in an 8-oz. Erlenmeyer flask, in 45 c.c. of warm nitric acid (sp. gr. 1·16). When dissolved completely, 5 c.c. of a saturated solution of potassium permanganate are added to the hot iron solution, and the mixture is boiled until the violet colour of the liquid has disappeared. Then five or six drops of saturated solution of permanganate are added, with a few drops more if necessary, just to dissolve the precipitated oxide completely, any excess being avoided. The solution is now cooled to 60° C., 5 c.c. of ammonia solution (sp. gr. 0·9) are added, the flask is shaken carefully until solution is effected, 30 to 40 c.c. of molybdate solution are added, the flask is again shaken thoroughly, and the resulting precipitate is allowed to subside for a few seconds. The precipitate is now rapidly filtered, and washed six times with 2 per cent. nitric acid, the flask being rinsed out with acid of the same strength. The precipitate and flask are next washed with a 2 per cent. potassium nitrate solution, and the precipitate, with the filter paper, is transferred from the funnel to the original flask. 25 c.c. of normal sodium hydroxide solution are added, the flask is shaken until the yellow precipitate is dissolved, the inner surface of the flask is rinsed with water, three or four drops of phenolphthaleïn are added, and the liquid is titrated with nitric acid of such strength that 1 c.c. = 0·01 per cent. P (*viz.*, 100 c.c. of $HNO_3$ of 1·42 sp. gr. to 11 litres of water). The titre of the acid is determined by means of a steel in which the phosphorus has been estimated gravimetrically. The soda solution is, by preference, of such strength that 25 c.c. = 16 c.c. of the acid; it should be standardised daily. The molybdate solution is made by dissolving 1 lb. of $MoO_3$ in 1,200 c.c. of water and 700 c.c. of $NH_3$ (sp. gr. 0·9), filtering the solution, and adding to it 300 c.c. of nitric acid (sp. gr. 1·42), and then adding 575 c.c. of this solution to a mixture of 1,200 c.c. of water and 475 c.c. of nitric acid (sp. gr. 1·42). In treating pig-iron, the carbon and insoluble matter should be filtered off after dissolving the iron. In the case of iron ores, it is necessary that all the phosphorus shall be in a form soluble in nitric acid.—W. G. M.

*Bromine: Separation from Mixed Alkaline Chlorides and Bromides.* H. Baubigny and P. Rivals. Comptes Rend. 1897, **125**, [15], 527—530.

The authors' method (this Journal, 1897, 635) does not allow the direct determination of the bromine, which attacks the grease of the air-pump jar, and can thus only be estimated by difference. Nor is it feasible to boil away and collect the bromine, instead of operating *in vacuo*. But by passing a current of air through the solution, the bromine is completely removed, and can be completely collected.

Two flasks are arranged in the same water-bath—one containing water to saturate the air used, the other containing the mixture for analysis, with the requisite amounts of copper sulphate and potassium permanganate, and provided with two tubes, one reaching to the bottom and expanded at the end, for the entrance of air, the other an exit tube, which is connected with the condenser, a straight tube with several bulbs, filled with dilute caustic potash solution. The bath is heated to the requisite temperature, and the air current set up; the humidity of the air is regulated by dipping the washing flask more or less deeply, so that the level of liquid in the distillation flask remains nearly constant. At the end of the operation the liquid is rinsed out from the condenser, a little sulphurous acid added, and then a mixture of nitric acid and silver nitrate to acid reaction, heated to boiling, and the silver bromide, after cooling, filtered off and weighed. The distillation flask containing the chlorine is treated exactly as directed in the earlier paper (*loc. cit.*).—J. T. D.

*Chlorine and Bromine: Separation and Determination in a Mixture of Alkaline Salts.* H. Baubigny and P. Rivals. Comptes Rend. 1897, **125**, [17], 607—610.

Experimenting by their method (see previous abstract) with chlorides and bromides separately, and determining in the one case the amount of chlorine carried over, and in the other the amount of bromine remaining in the distillation flask, the authors find that for the complete decomposition of the bromide a certain minimum concentration of the copper salt is necessary, but that the volume of the liquid (*i.e.*, the concentration of the bromide solution) is not, within wide limits, a determining factor; on the other hand, while the partial decomposition of the chloride depends to some extent on the proportion of copper sulphate and of potassium permanganate present, it can be altogether prevented by keeping below a certain maximum concentration of the chloride solution. If, in 100 c.c. of a solution containing 15 to 16 per cent. of $CuSO_4.5H_2O$ and 0·7 to 0·8 per cent. of permanganate, the amount of chlorine present does not exceed that corresponding to 0·25 grm. of sodium chloride, a complete separation of chlorine from bromine is possible in 1¼ to 1½ hours. The results of experiments quoted in the paper show the process to be very accurate.—J. T. D.

*Glycerophosphoric Acid, Neutralisation of, by Alkalis, in the presence of Helianthin A and of Phenolphthaleïn.* H. Imbert and A. Astruc. Comptes Rend. 1897, **125**, [24], 1039—1040.

Glycerophosphoric acid behaves towards Helianthin A as a monobasic acid; towards phenolphthaleïn as a dibasic acid. This is also the case with phosphoric acid, the combination of which with glycerin has not diminished its acid properties. By ordinary acidimetry the strength of solutions of glycerophosphoric acid can be determined with sufficient accuracy.—A. C. W.

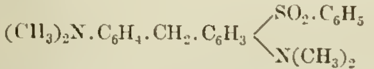
## ORGANIC CHEMISTRY.—QUALITATIVE.

*Benzenesulphinic Acid as a Reagent* [*for presence of Quinone and Quinonoid Groups*]. O. Hinsberg. Ber. 1897, **30**, [18], 2803—2805.

Benzenesulphinic acid unites with compounds of quinone-like structure with formation of sulphones (this Journal, 1896, 922). Tetramethyldiaminodiphenylcarbinol in acid solution assumes the quinone structure, so that, for example, its hydrochloride has the formula—

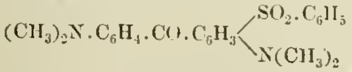
$$(CH_3)_2N.C_6H_4.CH : C_6H_4 : N(CH_3)_2Cl.$$

The author finds that this compound in acid solution unites with 1 mol. of benzenesulphinic acid to produce the very stable substance—



$$(CH_3)_2N.C_6H_4.CH_2.C_6H_3\begin{cases}SO_2.C_6H_5\\N(CH_3)_2\end{cases}$$

The stability of the compound towards acids and alkalis shows that it is not merely a salt of the sulphinic acid, and when oxidised by the bichromate mixture, it forms a ketone, probably of the constitution—



$$(CH_3)_2N.C_6H_4.CO.C_6H_3\begin{cases}SO_2.C_6H_5\\N(CH_3)_2\end{cases}$$

—A. C. W.

*Picric Acid, Detection of.* H. Swoboda. Zeits. Allgem. Oesterr. Apoth. Ver. **50**, 617; through Zeits. Anal. Chem. 1897, **36**, 513.

On treating a cold aqueous solution of picric acid with Methylene Blue, a violet flocculent precipitate, soluble in ether, chloroform, or hot water, is obtained. The colour of the solution varies from blue to green. On evaporating the solution in chloroform to dryness, a violet residue is obtained. —D. B.

*Pyruvic Acid, Two Colour Reactions of.* L. Simon. Comptes Rend. 1897, **125**, [15], 531—536.

To 1 c.c. of pyruvic acid solution is added an equal bulk of caustic potash solution of density 1·15 to 1·20, and then a few drops of sodium nitroprusside solution. An orange-red colour is struck, which, with strong solutions, is more nearly violet. A slight addition of acetic acid turns the red colour to a deep violet. This reaction, however, is produced by a variety of substances other than pyruvic acid (aldehydes, ketones, ethyl acetate, &c.), and can hence only be used with caution. With ammonia instead of potash, however, a violet-blue colour is struck, which becomes more and more greenish as the solution is more dilute, and the appearance of which is hastened by a trace of acetic acid and by very gentle heat. The colour is fairly stable, but slowly fades to a reddish-orange. No other substance has been found by the author to give this reaction, except acetophenone, which is easily distinguished from pyruvic acid by Legal's reaction (in which a stable blue coloration is given). Derivatives of pyruvic acid give the same reaction as the acid itself; the reaction appears to be characteristic of these derivatives, and to be connected with the presence in the molecule, of the group $- CH_2.CO.CO_2H$.

All the fatty amines, added to a mixture of pyruvic acid and a few drops of sodium nitroprusside, give at once a deep violet colour, changing on addition of acetic acid to blue, and then rapidly disappearing. This reaction appears to be characteristic of amines of the fatty series.—J. T. D.

*Margarin, Sesamé Oil cannot be used for the Recognition of.* Raumer. Zeits. angew. Chem. 1897, [23], 749—751.

In 1896 it was shown by Spampani and Daddi (Staz. sperim. agrar. Ital. **29**) that the milk of goats, fed with sesamé oil, showed the Baudouin reaction (salmon-red coloration) with hydrochloric acid and furfural. Recently Hanmux (Rep. Pharm. 1897, 208) found that butter coloured with turmeric showed the same reaction as fats containing this sesamé oil, or as sesamé oil itself and alone.

Experiments were made by the author with a number of vegetable dyes—*Daucus carota*, safflower, saffron, *Calendula*, *Bixa orellana*, and turmeric, but only the last named gave exactly the same colour reaction as sesamé oil. The colour was produced with hydrochloric acid only. The following dyes also gave the reaction:—Ponceau, Orange II., β-Naphthol Orange, Tropaeolin O O O No. 2, Acid Yellow G, Dimethylaniline Orange, and Metanil Yellow S. The colour reaction was produced, in each case, with hydrochloric acid alone.

The above results, especially those of Spampani and Daddi, show that sesamé oil, which manufacturers are now compelled to mix to the extent of 10 per cent. with the margarin, is a substance unsuitable for the purpose in view. For although the dyestuffs named give the colour with hydrochloric acid alone, and sesamé oil only after addition of furfural, yet if the colour were obtained after addition of the acid alone, any further and possible reaction with furfural would not be discernible.—N. H. J. M.

*Lard and Butter, Foreign Fats in; Detection of.* C. B. Cochran. J. Amer. Chem. Soc. 1897, **19**, 796—799.

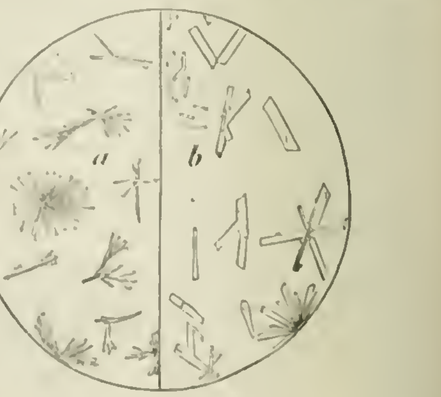
The presence of beef fat or mutton tallow in lard, may be detected by adding 22 c.c. of fusel oil to 2 c.c. of the melted fat, warming to a "blood heat" to obtain a perfect solution, cooling slowly to 16° or 17° C. and keeping it at this temperature for two or three hours. A crystalline deposit is thus obtained, the amount and character of which vary with the fat under examination. The deposit is thrown on a filter paper and drained thoroughly, and is then entirely or in part, dissolved in ether in a test tube the mouth of which is plugged with cotton wool. The crystals, which form on standing, are mounted in a drop of cotton seed oil, and examined microscopically. Lard containing 10 per cent. of beef or mutton fat, shows abundant crystals of typical beef fat, usually much more numerous than lard crystals; but with 20 per cent. of the beef fat, no lard crystals are seen. With lard containing 5 per cent. of oleo-stearin, the crystals, though somewhat modified, gave distinct evidence of beef fat being present; but with 2 per cent. of oleo-stearin, the indications were uncertain. The amount of the deposit from the fusel oil at 17° C. varies with the fat tested; thus, beef suet treated as above, gave a deposit of 16 c.c., and that from oleo-stearin filled the tube to the level of the liquid, while the maximum from pure lard was 1 c.c. By combining these indications with readings of the melting point of the fat, using standards of pure lard (one with high, and the other with low melting point), it may be possible to estimate the amount of the adulterant in lard. But before reading off the volume of the deposit, it is well to shake the cylinder, after which the deposit will settle evenly and more compactly. The melting points of pure lard samples were found to range from 34° to 45° C.

In examining butter or oleomargarin, 8 c.c. of the fusel oil are added to 2 c.c. of the filtered fat, and the mixture is warmed and cooled as before. Oleomargarin gives the more copious deposit; but the butter and oleomargarin crystals from the ether, although large and well formed, are very similar, yet apparently sufficiently unlike to allow of the two materials being thus distinguished apart. Perfect crystals characteristic of lard and of beef fat have been obtained from oleomargarin. The fusel oil used had the sp. gr. 0·8159 and the boiling point 129° C.—W. G. M.

*Formaldehyde, Detection of.* Leblin. Zeits. Allgem. Oesterr. Apoth. Ver. **51**, 92; through Zeits. Anal. Chem. 1897, **36**, 518.

Boil a few c.c. of the liquid to be tested, with 0·05 grm. of resorcinol, to which half or an equal volume of a 50 per cent. solution of caustic soda is added. If formaldehyde be present, the yellow solution changes to a fine red colour. Analogous compounds showing the usual reactions characteristic of aldehydes, fail to give this coloration. The reaction is said to be sufficiently delicate to detect 1 part of formaldehyde in 10 million parts of water.—D. B.

*Atropine and Hyoscyamine, New Method for the Distinction of.* S. Vreven. Chem. Zeit. Rep. 1897, 308.



A drop of aqueous solution of the alkaloid, acidified with sulphuric acid, is placed on an object glass, a trace of Marmé's reagent (5 grms. of potassium iodide, and 2·5 grms. of cadmium iodide in 30 c.c.) added, and the mixture stirred until it becomes clear. Under the microscope, well-defined crystals are at once seen to form: in the figure, *a* represents the appearance with hyoscyamine, *b* with atropine. The method is well adapted for the recognition of the alkaloids in urine. The alkaloid is obtained for examination by extracting with chloroform, the fluid made alkaline with ammonia, adding alcohol to remove the emulsion, filtering, extracting with dilute acid, repeating the treatment with ammonia and chloroform, washing the chloroform solution, and evaporating. Urine, to which no alkaloid had been added, gave on similar treatment only a yellowish precipitate with Marmé's reagent, due to leukomaïnes and ptomaïnes.—A. C. W.

*Santonin, New Reaction of.* A. Jaworowsky. Pharm. Zeits. Russl. 1897, **36**, 559; through Chem. Zeit. Rep. 1897, **21**, [91]. 269.

If 0·01—0·02 grm. of santonin be dissolved by warming it carefully in 2 c.c. of strong sulphuric acid, and an equal volume of a 1 per cent. solution of cerium sulphate acidified with sulphuric acid be added, the original straw-yellow of the santonin solution will be changed to cherry-red, and on the addition of about 8 c.c. of water, a fine violet precipitate will be produced.—W. G. M.

*Oil of Roses, Examination of.* F. Dietze. Süddeutsch. Apoth. Zeit, 1897, [89]; Chem. Zeit. **21**, [95], Chem. Rep. 288.

The most trustworthy method for detecting the presence of geranium oil, which is commonly added to oil of roses, depends on the determination of the saponification number. A weighed amount of the substance is boiled for half an hour with alcoholic $\frac{1}{2}$N-potash, and titrated with $\frac{1}{2}$N sulphuric acid, with phenolphthaleïn as indicator. The following numbers were obtained with oil of roses of different origin: German, 8·6; Bulgarian, 9·2; Turkish, 8·9; whilst oil of geranium gave:—French, 59·1; African, 53·1; Spanish, 75·3; Indian, 32·5. Adulteration of oil of roses with oil of geranium would, therefore, give rise to a higher saponification number.

Pure oil of roses should have a sp. gr. not above 0·870 at 15°. The solidifying point should not be below 15°—20°, the rotatory power in a 20 mm. tube should not be more than −1° 30′ at 20°, and the saponification number should not exceed 9·5—10.—N. H. J. M.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

*Fehling's Solution.* J. E. Gerock. Ber. 1897, **30**, [18], 2865—2867. (See also this Journal, 1897, 1046.)

It has been long known that the tartaric acid in Fehling's solution might be replaced by other compounds containing the hydroxyl group, *e.g.*, glycerin, mannite, carbonic acid. Divalent copper is able to replace the hydrogen in the hydroxyl group in compounds of a certain constitution and in the presence of alkalis, compounds of the type R.O.CuOM (M = alkali metal) are formed. If the radicle R is susceptible of oxidation under the influence of the divalent copper and excess of alkali, the copper is converted into the monovalent state, in which it is not capable of forming compounds of the above type, and so is precipitated. In other cases no oxidation occurs, and such substances can form a Fehling's solution, which when brought into reaction with a body of the former type, is reduced.

—A. C. W.

*Sugar in the Cane, Direct Estimation of.* H. Pellet. Bull. Assoc. Chim. 1897, **15**, 70—74.

The preparation of an average sample of cane is very difficult, from the great variation in composition in different parts of the same stalk as well as in different canes from the same stool. The lower part of the stalk may show 17 to 20 of sugar per 100 c.c. of juice and 0·2 to 0·5 of reducing sugars, and the upper part, 6 to 8 of sugar per 100 c.c. and 0·6 to 2 per cent. of reducing sugars. This difficulty is further increased owing to the hardness of the rind and the resistance of the inner fibres, so that a fine division, similar to beetroot pulp, is unattainable and the coarse filaments are not exhausted with certainty by alcohol which has been used in the hope of eliminating foreign polarising substances, the so-called plus sugar. Hot aqueous digestion affords a simple and rapid method for the direct estimation of the sugar. At least twice the normal weight of cane should be used and the mean of several tests taken. To allow for the volume of the marc, an average is adopted of 10 per cent. of insoluble residue and a volume of 90 per cent. of the weight of the residue; thus, if twice 16·20 or 32·40 grms. are weighed out, this corresponds to 3·24 of liquor or 3·24 × 0·9 = 2·916 of marc, and if the liquor is made up to 200 c.c., the result must be multiplied by 0·986 to get the percentage of sugar. As the woody fibre filled with air tends to rise, it is kept down by a perforated disc of lead maintaining the whole of the insoluble matter below the level of the liquid. Basic acetate of lead is added before heating. The cane is divided by a cane cutter into discs 2 to 3 cm. thick. A kilo. of these is rapidly ground in a mortar to furnish the sample. In factories where diffusion is practised, samples of the fresh slices are taken just as in the case of beetroots. The cane-cutter of Gallois and Dupont may be used. It is in the form of a disc with four variable knives, and may be worked by hand or power.

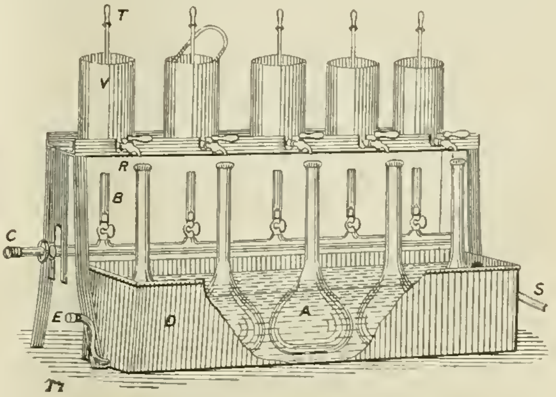
Other methods of determining sugar in the cane directly, are lixiviation with hot water containing lead acetate by Castel's arrangement, or intermittent extraction by Zamaron's apparatus. But, however obtained, the result represents the sample only, and not the richness of 100 kilos. of canes as weighed, owing to changes taking place after delivery at the factory, and from the varying amount of dust, soil, and *débris*, and damaged and gnawed canes. Altogether the loss of sugar from these causes may amount to from 1 to 2·5 per cent.

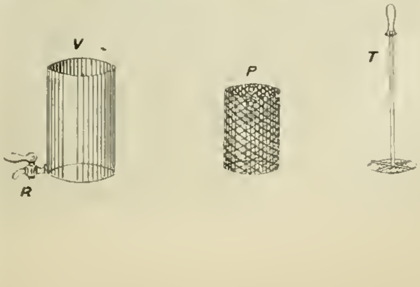
Having regard to the difficulty of sampling, the indirect method of analysis is preferred, that is, the analysis of the juice obtained by milling, of course, without the addition of water. Gallois and Dupont have a laboratory mill and a double screw press which give an extraction of about 65 per cent The sugar per 100 c.c. of juice is calculated to 100 grms. of cane, the factor employed being determined by experience. For a cane containing 10 per cent. of ligneous matter, the factor 0·90 would be too high, as the juice first extracted is purer than what remains, that found in practice is usually discovered to be 0·87 to 0·885, depending on the cane crushed, the hardness of the rind, state of maturity, &c. To take account of the loss of weight of the cane, the result must be multiplied by 0·99 to 0·975, which reduces the coefficient to 0·85 or 0·87. In Mauritius the manufacturers and the chemists have agreed to adopt 0·84 as mean coefficient. This figure could not be adopted for other countries, or during the whole period of crushing, but should be determined weekly. Young canes of 8 to 8·5 per cent. of ligneous matter have a coefficient of 0·86 to 0·87, while canes, more or less dry and resistant, with 12 per cent., would give a coefficient as low as 0·84 or 0·83.

—L. J. de W.

*Sugar Cane and Beetroot, Direct Estimation of Sugar in.* J. Zamaron. Bull. Assoc. Chim. 1897, **15**, 74—78.

The apparatus consists of a series of cylindrical receptacles of copper resting on a metal table, each furnished with a tap,





R. for drawing off the sugar liquor into a 1,000 c.c. flask A; a cylindrical basket, P, to receive the pulp is placed in each receptacle, a projection at the bottom of the latter preventing the bottom of the basket from coming into contact with that of the receptacle. Each basket is provided with a perforated metal disc with a handle T, to press down the pulp. A series of Bunsen burners, C, B, are arranged under the receptacles, and the flasks stand in a tank D (with in- and out-lets E and S), through which cold water flows, so that the volume may be made up to 1,000 c.c. without delay.

100 grms. of pulp are introduced into each basket, and exhausted with boiling water. 200 c.c. are first poured in, and this is boiled for 10 to 12 minutes, and the liquor drawn off into the flask. In all, six successive exhaustions are made, each with 200 c.c. except the last, when only 150 c.c. are used. Owing to loss by evaporation during the boiling, about 960 c.c. will be obtained. Before drawing off the first liquor, 10 to 15 c.c. of basic acetate of lead at 28° Baumé is added, to precipitate organic matters and prevent alteration of the liquor. The liquor is finally cooled to 20° C., made up to the mark, and polarised in a 400 mm. tube. The reading, multiplied by 1·3024, gives the sugar in 100 grms. of cane or beetroot.

The pulp is pressed and dried at 100°—110°, to obtain the ligneous matter.

A little lime or baryta should be added, to neutralise the strong acidity of unsound canes so as to prevent inversion, as also in the case of beetroots.—L. J. de W.

*Sugar in Cocoa Preparations, Estimation of.* L. de Koningh. Zeits. f. angew. Chem. 1897, [22].

16·35 grms. of the sample are accurately weighed out. 100 c.c. of distilled water added, and, after shaking well for about 10 mins., the solution is filtered into a 50 c.c. flask, which has also a mark at 55 c.c. 50 c.c. of filtrate are collected, concentrated lead acetate added to the higher mark, the mixture shaken well and filtered, and the filtrate examined in a 22 cm. tube, with the polariscope. The polarisation is, say, 17, and this is taken, for the time being, as the percentage of raw sugar in the sample. If 16·35 grms. of pure raw sugar be mixed with 100 c.c. of water, the volume increases by just about 10·2 c.c.; the weighed-out sample (16·35 grms.), containing 47 per cent., or 7·68 grms., of sugar, would therefore cause an increase of volume of 4·8 c.c. In order, therefore, to obtain the actual percentage of sugar in the sample, the result obtained by polarisation must be multiplied by $\frac{100 + 4·8}{100}$. This correction is, of course, not mathematically correct, but is sufficient for the purpose in question. The other soluble constituents of the cocoa which might cause an increase of volume are precipitated by the addition of the lead acetate.—A. S.

*Phenylhydrazine, Volumetric Determination of.* H. Causse. Comptes Rend. 125, [19], 712—714.

PHENYLHYDRAZINE in acetic acid solution reduces arsenic acid according to the equation—

$$As_2O_5 + C_6H_8N_2 = N_2 + H_2O + C_6H_6O + As_2O_3$$

The following solutions are needed for the author's method:—

1. 125 grms. of arsenic acid, dissolved on the water-bath in 650 c.c. of water and 125 c.c. of strong hydrochloric acid; the cooled solution is filtered and made up to a litre with glacial acetic acid. 2. Decinormal iodine solution. 3. 200 grms. of caustic soda (free from sulphides) dissolved in a litre of water. 4. Cold saturated solution of sodium bicarbonate. 5. Starch solution and phenolphthalein as indicators. The sample of phenylhydrazine or its hydrochloride is weighed (about 0·2 grm.), placed in a 500 c.c. flask, 60 c.c. of the arsenic acid added, and the liquid boiled under a reflux condenser, using a spiral of platinum wire to prevent bumping. When action has ceased (about 40 minutes), the liquid is cooled, 200 c.c. of water and then soda to alkaline reaction, added, and finally a drop or two of hydrochloric acid; 60 c.c. of the sodium bicarbonate solution are then run in, and the arsenious acid determined by iodine solution and starch. The method is accurate, and can be applied also to the compounds of phenylhydrazine with aromatic aldehydes. In the case of its compounds with fatty aldehydes, the aldehyde should be removed before titration, on account of its action on arsenic acid.—T. T. D.

*Butter, Estimation of all the Volatile Fatty Acids in.* E. Wrampelmeyer. Landw. Versuchsstat. 49, 215—218; Chem. Centr. 1897, 2, [19], 986.

IT is stated that in the following method, the two defects of Wollny's modification of Reichert's process are overcome, *viz.*, the estimation of only part of the volatile fatty acids and the impossibility of completely separating the alcohol used in the saponification. About 5 grms. of the filtered fat are warmed in a flask of 700—800 c.c. capacity, with 20 c.c. of an alkaline glycerin lye (100 grms. of NaOH dissolved in 100 c.c. of water; 20 c.c. of this solution to 180 c.c. of concentrated glycerin) over the direct flame, till no more frothing occurs and a clear soap is formed. 250 c.c. of hot, recently boiled, distilled water are cautiously added, then a drop of litmus solution and 50 c.c. of sulphuric acid (20 c.c. of $H_2SO_4$, of sp. gr. 1·84, per litre). The mixture is then steam-distilled, the tube connecting the flask to the condenser being provided with a safety tube. The rate of distillation should preferably be about 1 litre per hour, and care must be taken to prevent spirting over. The distillate, which is collected in two portions of 1 litre and ½ litre respectively, is filtered, and 500 c.c. of the filtrate from the litre and 250 c.c. from the ½ litre, titrated. A blank estimation should always be made, and the results corrected, if necessary. The results obtained with glycerin-potash lye were unsatisfactory.

—A. S.

*Fats and Oils, Edible; Analysing and Discriminating.* J. Suisse de Chim. et Pharm. 35, 448; Bull. Assoc. Belge des Chim. 11, 160—163.

THE following schedule of analyses has been ratified by the Société Suisse des Chimistes Analystes:—

### A.—METHODS OF ANALYSIS.

1. *Baudouin's Reaction* (*Villavecchia and Fabris' Modification*). *Reagent.*—2 grms. of furfuraldehyde in 100 c.c. of alcohol.

*Application.*—10 c.c. of the oil are agitated thoroughly for half a minute with 0·1 c.c. of the reagent and 10 c.c. of hydrochloric acid (sp. gr. 1·19). A red coloration indicates sesamé oil, but Bari olive oil will frequently give a faint tinge. Any other coloration may be disregarded.

2. *Becchi Reaction.* *Reagent.*—1 grm. of silver nitrate is dissolved in 5 c.c. of water and mixed with:—Alcohol, 200 c.c.; ether, 20 c.c.; and nitric acid (1 : 10), 1 c.c.

*Application.*—10 c.c. of fat and 3 c.c. of reagent are heated in a test tube on the boiling water-bath for 10 minutes. Cotton-seed oil is indicated by a brown or black coloration.

3. *Wellmann's Reaction.* *Reagent.*—Molybdic acid, 5 grms., saturated with sodium carbonate solution; and evaporated to dryness together with 1 grm. of sodium phosphate, then fused and dissolved in boiling water, concentrated nitric acid (5—7 c.c.) being added till the yellow shade of the liquid is permanent, whereupon the solution is made up with water to 100 c.c. Commercial sodium phosphomolybdate may also be used, 5 grms. being dissolved and made up to 100 c.c. after adding nitric acid as prescribed.

*Application.*—2 c.c. of reagent shaken up for 1 minute with 1 c.c. of fat, dissolved in 5 c.c. of chloroform. A green coloration (changing to blue on addition of ammonia) is formed in the aqueous layer when vegetable oils (except cocoanut oil) are present.

4. *Allen's Reaction.*—Equal volumes of fat and concentrated nitric acid (sp. gr. 1·4) agitated for half a minute and left to stand for about 15 minutes.

Cotton-seed oil gives a coffee-brown coloration.

5. *Maumené Thermal Test.*—50 grms. of melted fat are placed in a 100 c.c. beaker (diameter 5 c.m.) and kept in an oven at 10°—12° C.; then mixed with 10 c.c. of 95·6 per cent. sulphuric acid of the same temperature and stirred with a thermometer; the rise in temperature is then read off.

6. *Hübl Iodine Number. Reagents.*—(1) Solution of 25 grms. of iodine in 500 c.c. of alcohol. (2) 30 grms. of mercury bichloride dissolved in 500 c.c. of alcohol. (3) 25 grms. of sodium thiosulphate dissolved in 1 litre of water. (4) Aqueous solution of potassium iodide (10 grms. per 500 c.c.). (5) Aqueous solution of potassium bichromate (3·874 grms. per litre). (6) ½ litre of starch solution.

*Titrating the Thiosulphate Solution (Volhard).*—10 c.c. of the above potassium iodide solution and 20 c.c. of the bichromate solution are mixed with 5 c.c. of concentrated hydrochloric acid and titrated back with the thiosulphate. 200 c.c. of bichromate solution = 0·2 grm. of iodine.

*Application.*—0·15—0·20 grm. of a drying oil, or 0·30—0·50 grm. of a non-drying oil or a solid fat are dissolved in 10 c.c. of chloroform and mixed with 20 c.c. of iodine solution and 20 c.c. of mercury bichloride, a further 10 c.c. of each being added if decoloration immediately ensues. The titre of the iodine solution is found by a blank experiment.

After leaving the liquid at rest for four hours in the dark, water is added, with sufficient potassium iodide to re-dissolve the mercuric iodide formed, the excess of iodine being then titrated with the thiosulphate solution in presence of a little starch.

7. *Saponification Value. Reagent.*—A solution of 30 grms. of potassium hydroxide in 100 c.c. of water, made up with absolute alcohol to 1 litre.

*Application.*—2 grms. of fat are saponified by heating in a porcelain capsule on the water-bath with 25 c.c. of the above reagent, the excess of the latter being then titrated back with $\frac{1}{2}N$ hydrochloric acid, phenolphthaleïn being the indicator. A blank experiment is made with another 25 c.c. of the reagent.

8. *Rancidity.*—10 grms. of fat are dissolved in 50 c.c. of a mixture (equal vols.) of ether and alcohol, and titrated with $\frac{1}{20}N$ sodium hydroxide, phenolphthaleïn being the indicator. 1 c.c. of alkali solution per 100 grms. of fat used, denotes one degree of rancidity (Köttstörfer).

9. *Water and Foreign Substances. Water, Qualitative Test.*—A little of the fat is heated in a test tube to a temperature of 120° C., maximum. *Quantitative.*—Same method as in the case of butter.

*Non-Fatty Matters.*—10 grms. of the fat are saponified with the quantity of alcoholic KHO indicated in the saponification test. The residue, after driving off the alcohol, should be entirely soluble to a clear solution in water.

*Physical Examination.*—(1) Specific gravity in the Ambühl instrument at the temperature of boiling water. (2) Refractive index, at 40°, determined by the Zeiss instrument. (3) Melting point in capillary tubes. (4) Setting point, Dalican method.

### B.—Judging Hog Fat and Olive Oil.

*Essential Tests.*—(1) Determination of water and non-fatty substances. (2) Qualitative tests for foreign fats. (3) Specific gravity. 4. Refractive index.

*Contingent Tests.*—(1) Iodine number. (2) Saponification value. (3) Maumené thermal test. (4) Melting and setting points.

Fundamental characteristics :—

| —— | Hog Fat. | Olive Oil. |
|---|---|---|
| Specific gravity ............... | 0·860—0·862 | 0·8618—0·863 |
| Refractive index.............. | 49—53 | 53—55 |
| Iodine number................ | 55—63 | 78—84 |
| Thermal test (Maumené) ..... | 24°—18° | 39°—42° |

Hog fat rarely exhibits a greater rancidity than 5°. In judging this point the flavour and odour of the product must be chiefly considered.—C. S.

### *Opium, Quantitative Estimation of Starch in.* L. F. Kebler and C. H. Lawall. Amer. J. Pharm. 1897, 234; Chem. Centr. 1897, **2**, [19], 985.

The author recommends the following process for the estimation of starch in opium :—10 grms. of opium are mixed with cold water, alcoholic potash added, the mixture heated for a short time on the water-bath, and filtered hot. The alcohol is then driven off, the residue taken up with water and a few drops of hydrochloric acid, and heated for three hours in connection with a reflux condenser. The liquid is then neutralised with soda, filtered, and made up to a certain volume. The sugar content is estimated volumetrically or gravimetrically in the filtrate by means of Fehling's solution. Other carbohydrates are estimated conjointly with starch by this method.—A. S.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

### *Reduction of Carbon Dioxide at the Ordinary Temperature.* A. Lieben. Monatsh. f. Chem. 1897, **18**, 582—588.

In continuation of his previous work on this subject (Monatsh. 1895, 211), the author has investigated the action of nascent hydrogen from different sources on aqueous solutions containing carbon dioxide. When platinised magnesium was allowed to act on a solution containing disodium hydrogen phosphate and ammonium carbonate, through which a current of carbon dioxide was continuously passed, after the lapse of 11 weeks it was found that of the disodium hydrogen phosphate originally present, one-third had been transformed in such a way that the phosphoric acid was contained in the precipitate, whilst the sodium was converted into sodium bicarbonate. Only traces of formic acid were obtained, whilst a similar experiment with aluminium amalgam gave considerable quantities.

In the same way, a solution of potassium sulphate, into which baryta water was slowly but continuously introduced, and through which carbon dioxide was simultaneously led, when acted upon by platinised magnesium, produced only traces of formic acid, whilst aluminium amalgam produced considerable quantities.

The author ascribes this difference in the behaviour of nascent hydrogen to unequal quantities of energy accompanying the liberation of the hydrogen.—J. S.

### *Wood-Smoke, Relation between the Antiseptic Properties of, and the Aliphatic Aldehydes.* G. Pasqualis. Atti. del Instit. Venet. d. science, lettere ed arti, **8**, [7], 713—730; Chem. Centr. 1897, **2**, [20], 1012.

The author shows, by bacteriological experiments, that the antiseptic properties of wood-smoke depend not only on the aromatic constituents, but also on the volatile constituents. These properties greatly resemble those of formalin. It was shown, by means of Hehner's reaction, that besides acetaldehyde, furfurol, and dimethylacetaldehyde, which are well known as constituents of wood-smoke, the latter also contains formaldehyde in considerable quantities. It was found, by means of Schiff's reaction, that the smoke from 100 grms. of shavings from different kinds of wood contains about 0·5 grm. of formaldehyde. For disinfecting purposes, the author prefers the use of formaldehyde vapour, or spraying with a 0·5 per cent. formaldehyde solution, rather than fumigating with wood-smoke. Acetaldehyde and dimethylacetaldehyde were found to possess antiseptic properties.—A. S.

### *Fire-Damp Explosions: Ammonium Salts as Preventives, in Blasting Operations.* Oesterr. Zeits. Berg- u. Hüttenw. 1897, **45**, 568.

A non-explosive salt of ammonium, if introduced into the bore-hole along with the charge, is volatilised during detonation, and cools the gases to such a degree that ignition of the "fire-damp" is rendered impossible. In order to prevent the production of oxides of nitrogen, an admixture of some inert carbonaceous substance is recommended. About 1 part of ammonium salt may be used to 2 parts of blasting powder, the materials to be charged separately into the bore-hole. It is immaterial whether the explosive be introduced first or last; but the ammonium salt should be contained in a waterproof case and surrounded with melted paraffin.—H. T. P.

### *Chromium Tetroxide and Salts of Perchromic Acid.*
O. F. Wiede. Ber. **30**, 2178—2189.

To 10 grms. of chromic anhydride, dissolved in 500 c.c. of ice-cold water, are added 500 grms. of pure ether, cooled in ice and salt, and 100 c.c. of a pure 10 per cent. solution of hydrogen peroxide. The resulting perchromic acid is dissolved in the ether by shaking, and the aqueous solution run off in a separating funnel from the ether, which is well washed with ice-cold water. By cautiously adding cooled ammonia to the blue ethereal solution, and allowing to stand, the blue colour disappears, while the ammonia solution turns first deep brown, and then becomes brighter in colour, while an amorphous greenish-brown substance is deposited. This, collected on a filter, redissolved in 10 per cent. ammonia at 50° or 60°, and allowed to cool, deposits dark brown crystalline needles, which are washed with absolute alcohol and ether, and dried over caustic potash in an atmosphere of ammonia. 500 c.c. of the ethereal solution yield 0·5 to 1 grm. of the substance. Analysis gave figures corresponding with $CrO_4.3NH_3$.

The crystals are soluble in dilute ammonia and (with partial decomposition) in water. The dry substance readily explodes on heating, producing clouds of flocky chromium oxide. Both acids and fixed alkalis decompose it, evolving oxygen, and producing, in the case of acids, a chromic salt; in the case of alkalis, a chromate. If the substance be covered with ether, and dilute sulphuric acid be carefully added, the blue coloration of perchromic acid is produced.

Treating the blue ethereal solution with pyridine instead of ammonia, and evaporating the ether rapidly by a current of air, a blue residue is left; and when a portion of this is sown in another portion of the pyridine-containing ethereal solution, it induces the formation of blue crystals, which, collected, dried, and analysed, appear to have the composition $CrO_4.OH.C_5H_5N$. The dry crystals do not spontaneously decompose, but a temperature below 100° C. at once determines their explosion, and when moist, they rapidly decompose into chromic acid and a brownish resin. They do not dissolve in water, but do in most neutral organic solvents.

Using aniline in place of pyridine, the blue ethereal solution becomes violet, and on adding an equal volume of cooled petroleum spirit, crystallisation sets in, and a crop of dark violet, almost black, crystals is obtained, which explode more readily than even the pyridine compound, so that the desiccator needs to be kept cool with ice. The formula appears to be $CrO_4.OH.C_6H_7N$. The crystals dissolve in ether, but not in benzene nor in petroleum spirit. Water rapidly decomposes them, while acids and alkalis evolve oxygen with explosive violence.

The aniline and pyridine compounds seem to be salts of a true perchromic acid ($CrO_4.OH$), the anhydride of which ($Cr_2O_9$), is possibly the blue substance in the ethereal solution worked with; while the ammonium compound seems to be one of $NH_3$ with a hitherto unrecognised chromium oxide, $CrO_4$.—J. T. D.

### *Magnesium, Basic Salts of.*
Tassilly. Comptes Rend. 1897, **125**, 605—607.

A SOLUTION of 145 grms. of magnesium bromide in 380 c.c. of water is heated to boiling, and 5 grms. of magnesia, calcined at a low temperature, gradually added. The solution is re heated till the magnesia dissolves, and the liquid filtered hot into flasks, which are well corked and allowed to stand. After a fortnight, small groups of acicular crystals separate, the analysis of which agrees with the formula—

$$MgBr_2.3MgO.12H_2O.$$

At 120° in a current of dry air the crystals lose water, and become $MgBr_2.3MgO.6H_2O$. Atmospheric carbon dioxide rapidly acts on this hydroxybromide, which is also decomposed by water and by alcohol. All attempts to prepare a hydroxyiodide failed.—J. T. D.

### *Hypophosphorous Oxide [$P_2O$].*
A. Besson. Comptes Rend. 1897, **125**, [24], 1032—1033. (See also this Journal, 1897, 612.)

IF phosphorus trichloride and phosphorous acid be heated on the water-bath in a flask provided with an inverted condenser, hydrochloric acid is evolved, and a reddish-yellow solid is formed in the liquid. After addition of water, filtering, and washing with cold water, a substance is left having the composition represented by $P_2O$. The author considers its formation to be explained by the reactions: $PCl_3 + H_3PO_3 = 3HCl + P_2O_3$; $2P_2O_3 = P_2O + P_2O_5$. The same oxide is obtained by passing a slow current of well-dried air through a cold or lukewarm solution of phosphorus in carbon tetrachloride.—A. C. W.

### *Glycerophosphoric Acid, Heat of Neutralisation of.*
H. Imbert and G. Belugou. Comptes Rend. 1897, **125**, [21], 1040—1042.

THE addition of the first molecule of alkali to glycerophosphoric acid produces as much heat as if it acted upon phosphoric acid; the second molecule of alkali produces more heat than in the case of phosphoric acid, but less than the first molecule; whilst the third molecule gives rise to no appreciable heat production. The authors regard this as a proof that the glycerin has only caused the alcoholic function of phosphoric acid to disappear.—A. C. W.

### *Levulose: Preparation from Mannite by Fermentation.*
Vincent and Delachanal. Comptes Rend. **125**, [19], 716—717.

THE ferment of sorbose was allowed to act on a 3 per cent. solution of mannite, with an appropriate peptone nutrient solution. The ferment developed rapidly at 30° C., and the liquid, after fermentation had ceased, was treated with lead acetate. After removing the lead, evaporating, removing unused mannite by means of alcohol, and distilling off the latter, the solution contained a reducing sugar, the osazone of which had the melting point of that of dextrose and levulose. The rotatory power of the solution was near that of levulose, and on preparing and decomposing the lime compound a solution was obtained giving the same figure for rotation as levulose. The mannite, then, is oxidised by the action of the ferment, according to the equation—

$$C_6H_{14}O_6 + O = C_6H_{12}O_6 + H_2O.$$

—J. T. D.

### *Chloroform, Decomposition of, in the Organism.*
A. Desgrez and M. Nicloux. Comptes Rend. 1897, **125**, 973—975.

It is generally considered that chloroform undergoes no change in the body. Desgrez (this Journal, 1897, 1035) has shown that it is decomposed by aqueous alkaline solutions with production of carbon monoxide. The analysis of the gases of the blood of a dog, under ordinary conditions and under chloroform anæsthesia, showed that in the latter case more carbon monoxide was present, apparently produced by the decomposition of the chloroform by the alkalis of the blood. The quantity of carbon monoxide so formed, though very small, is yet sufficient to have an appreciable effect on the respiratory exchange.—A. C. W.

### *Theobromine, Some Derivatives of.*
H. Brunner and H. Leins. Ber. **30**, [17], 2584—2587.

BY heating silver theobromine with alkyl iodides in a sealed tube at 100° for 24 hours, the authors have prepared propyl-, isopropyl-, butyl-, and amyl-theobromine. These homologues of theobromine are crystalline powders, little soluble in cold alcohol and water, more soluble in hot, somewhat soluble in ether and chloroform. With chlorine water and ammonia all give the theobromine and caffeine reaction; silver nitrate added to their aqueous solutions produces a flocculent precipitate soluble in ammonia. The melting points lie above 270° C.

Nitrotheobromine was obtained by the action of nitric acid on theobromine as a light yellow microcrystalline powder, soluble with difficulty in cold water, alcohol, and ether; more readily in hot water and alcohol.—A. C. W.

### *Phenylhydrazine, Certain Combinations of, with Metallic Iodides.*
J. Moitessier. Comptes Rend. 1897, **124**, [26], 1529—1531.

PHENYLHYDRAZINE was found to combine with the iodides of metals of the magnesium group, giving compounds analogous to those obtained with the corresponding bromides (Comptes Rend. 1897, **124**, 1242) and chlorides (*ibid.* **124**, 1306). The organic base was determined in these bodies by the method of E. von Meyer, which is based on the formation of one molecule of iodobenzene and three molecules of hydriodic acid, through the action of an excess of iodine on one molecule of phenylhydrazine. The phenylhydrazine iodides of zinc, cadmium, manganese, and nickel are described.—J. O. B.

*Phenylhydrazine, Certain Combinations of, with Metallic Nitrates* [*of Co, Zn, Cd, and Ni*]. J. Moitessier. Comptes Rend. 1897, **125**, [3], 183—185.

The nitrates of the metals of the magnesium group combine directly with phenylhydrazine, forming compounds analogous to those formed with the haloid salts of the same metals. The phenylhydrazine nitrates of cobalt, zinc, cadmium, and nickel are described.—J. O. B.

*Phenylhydrazine, Combination of, with Certain Metallic Acetates.* J. Moitessier. Comptes Rend. 1897, **125**, [17], 611—612.

Phenylhydrazine forms, with the acetates of metals of the manganese group, compounds analogous to those of the other salts already investigated by the author. A mixture of phenylhydrazine in alcoholic solution and the metallic acetate in powder was boiled under a reflux condenser on the water-bath for about 15 minutes. A clear solution was thus obtained, from which, on cooling, the new compound crystallised out. All these compounds were soluble in water, alcohol, and chloroform, especially when warm, but were insoluble in ether. When heated to 100° C. they gradually lost their phenylhydrazine. The phenylhydrazine acetates of zinc, cadmium, manganese, cobalt, and nickel are described.—J. O. B.

*Chloride of Nitrogen, Preparation of.* W. Hentschel. Ber. 1897, **30**, [17], 2642. (See also this Journal, 1897, 698.)

Chloride of nitrogen may be prepared by the action of sodium hypochlorite on ammonium chloride. The author finds that bleaching powder may be used instead of the hypochlorite if a certain quantity of acid be added to its solution. The formation of the explosive compound in any quantity by this method necessitates adherence to the proportions given:—3 litres of a chloride of lime solution, containing 22·5 grms. of available chlorine per litre, are placed in a stoppered flask of 5 litres capacity, 10 per cent. hydrochloric acid is added through a funnel reaching to the middle of the liquid, which is kept in gentle motion, until a portion tested with excess of 20 per cent. ammonium chloride gives no evolution of gas. Generally 300 c.c. of acid are required. Then 300 c.c. of the ammonium chloride solution are added, and after gentle shaking, 300 c.c. of benzene; the flask is immediately well shaken for half a minute. If the light be strong, the flask is now protected by a black cloth, the aqueous solution siphoned off, and the benzene solution poured through a folded filter containing 20 grms. of powdered calcium chloride. About 290 grms. of 10 per cent. nitrogen chloride solution are obtained; this should be kept in the dark or, better, used freshly prepared.—A. C. W.

*Chloride of Nitrogen, Action of, upon Aniline, and also Methyl- and Ethylaniline.* W. Hentschel. Ber. 1897, **30**, [17], 2643—2649.

When a 5—10 per cent. nitrogen chloride solution in benzene is allowed to drop into an aniline solution of the same strength, the mixture becomes warm, and a dirty green coloration appears, turning to a reddish-brown. At the same time solidification occurs, owing to the separation of ammonium chloride and aniline hydrochloride. On the addition of more nitrogen chloride, the latter salt disappears, and when about 1 mol. of the chloride has been added to 1 mol. of aniline, the reaction is finished when the liquid again becomes clear. On evaporation, the solution solidifies to a crystalline cake of ordinary trichloraniline. Among the intermediate products of the reaction were found, in addition to aniline hydrochloride, azobenzene and Aniline Black. Chloride of nitrogen converts aniline hydrochloride into trichloraniline without noticeable evolution of gas or production of colour. It was found that aniline hydrochloride suspended in benzene is converted by chlorine into trichloraniline hydrochloride. This unstable salt is at once decomposed by water or alcohol, but may be made by passing hydrochloric acid gas into a benzene solution of trichloraniline, which substance is thus shown to possess basic properties. Nitrogen chloride reacts with acetanilide to produce *p*-chloracetanilide.

The actions of chlorine and chloride of nitrogen on methylaniline, are similar to those observed with aniline: trichlormethylaniline, melting at 28·5° and boiling at 256°, is obtained. The hydrochloride of this base is converted by oxidation with chromic acid into trichloraniline.

Dimethylaniline enters into no simple reaction with nitrogen chloride: a colouring matter is formed, together with a crystalline substance, $C_{24}H_{11}N_2Cl_{13}$, which, on heating, evolves 5HCl, and is possibly a derivative of tetraphenylhydrazine, $C_{24}H_{20}N_2$.—A. C. W.

## PATENT.

*Extinguishing Fires, Improved Compound for Use in.* A. J. Boult, London. From A. O. Richter, Dresden, Germany. Eng. Pat. 23,126, Oct. 8, 1897.

A mixture of 5 parts by weight of alum with 1 part of "natron" is made, of which mixture 1 litre by volume is dissolved in a hectolitre of water to constitute a fire-extinguishing liquid.—E. S.

# New Books.

Deuxième Congrès International de Chimie Appliquée. Organisé sous le Patronage du Gouvernement Français, par L'Association des Chimistes de Sucrerie et de Distillerie de France et des Colonies (M. L. Lindet, President) Réuni à Paris, du 27 juillet au 5 aout, 1896, sous la Présidence de M. Berthelot.

Comptes Rendus. Par François Dupont, Secrétaire Général du Congrès, Secrétaire Général de L'Association des Chemistes de Sucrerie et de Distillerie. Siège de L'Association des Chimistes de Sucrerie et de Distillerie, 156, Boulevard de Magenta. 1897. Price of volumes 1 to 5, Fr. 22.80; each of the first four vols., Fr. 7.50; vol. 5, Fr. 5.

Five 8vo volumes, in paper covers, containing the Reports of Proceedings in 1896 of the Second International Congress of Applied Chemistry, organised by the Association des Chimistes de Sucrerie et de Distillerie.

Vol. I. contains the Proceedings of Section I., Sugar Industry; and Section II., Alcohol Industries and Fermentation. The matter fills 768 pages.

Vol. II. is devoted to the Reports of Section III., Agricultural Industries; Section IV., Agricultural Chemistry; and Section V., Official Analyses and Scientific Apparatus. 540 pages.

Vol. III. to the Reports of Section VI., Chemical Industry; Section VII., Photography; and Section VIII., Metallurgy, Mines, and Explosives. 487 pages.

Vol. IV. to the Reports of Section IX., Chemistry applied to Medicine, to Pharmacy, Hygiene, and Foods; Section X., Electro-chemistry and Electro-metallurgy; Section XI., Waste Waters and Liquors (Residuals). 654 pages.

Vol. V., List of Members; Composition of Committees, Sections; The Excursions and Visits to Factories, &c. 257 pages.

Introduction to the Study of Organic Chemistry. A text-book for students in the Universities and Technical Schools. By John Wade, B.Sc., Senior Demonstrator of Chemistry and Physics at Guy's Hospital. Swan, Sonnenschein, and Co., Ltd., Paternoster Square, London. 1898. Price 7*s.* 6*d.*

This book contains frontispiece, preface (in which it is stated that the object of the work is to be a guide to students commencing Organic Chemistry), table of contents, list of charts and of illustrations. The subject-matter fills 442 pages, and at the end are alphabetical name and subject indexes.

The text is sub-divided as follows:—PART I. TYPICAL COMPOUNDS OF SIMPLE CONSTITUTION. Section I. The Alcohols and Acids. Organic Radicles. Section II. Synthesis and Molecular Structure. Section III. The Theory of Structure developed. Section IV. The Hydrocarbons and the Theory of Substitution. PART II. ALIPHATIC OR OPEN-CHAIN COMPOUNDS. Section V. Substitution Products of Methane and Ethane. Section VI. Three-carbon Derivatives. Section VII. Four-carbon Derivatives. Section VIII. Six-carbon Derivatives. Section IX. Aliphatic Derivatives of Nitrogen, &c. PART III. AROMATIC OR RING COMPOUNDS. Section X. Benzene and the Phenyl Radicle. Section XI. The Benzyl-Benzoic Group. Section XII. The Poly-Substitution Products. Benzene. Section XIII. The Methyl-Benzenes and their Derivatives. Section XIV. The Higher Alkyl-Benzene Derivatives. Section XV. Complex Ring Compounds. Section XVI. Ring Compounds of Nitrogen and other Elements. In Appendix I., pages 411—442, there are a series of Laboratory Notes, arranged as exercises, on the various chapters in the book. In Appendix II. is a Scheme of Qualitative Analysis. There are 10 illustrations in the text.

# Trade Report.

## TARIFF CHANGES AND CUSTOMS REGULATIONS.

### DUTY ON ETHYL BROMIDE AND CHLORIDE.

*Chem. and Druggist*, *Jan.* 1898, 58.

The customs tariff of the United Kingdom provides for the payment of a duty of 13*s.* 7*d.* per gal. on imported ethyl iodide, but the revenue from that source is practically nil, and was so in 1896–97. Ethyl bromide and chloride, which are much more used, were not mentioned in the tariff until Saturday last, when a general order was issued from the Custom House, London, imposing a duty of 1*s.* per lb. on the importation of ethyl bromide, and a duty of 15*s.* 8*d.* per liquid gal. (equivalent to 1*s.* 10*d.* per lb.) on the importation of ethyl chloride. Similar duties were previously obtained by other means.

### NEW BULGARIAN CUSTOMS TARIFF.

*Bd. of Trade J.*, *Jan.* 1898, 59.

| Classification of Articles. | Conventional Rates of Duty under Treaties with | | | |
|---|---|---|---|---|
| | Austria-Hungary. | France. | United Kingdom. | Russia. |
| Hydraulic lime and cement | .. | 10 *ad val.* | .. | 10 *ad val.* |
| Soap, common | 18 *ad val.* | 18 " | .. | 18 " |
| " toilet | .. | 12 " | .. | 12 " |
| Sole leather, and other kinds not specially mentioned | 16 *ad val.* | 16 " | .. | 16 " |
| Leather wares | " " | " " | .. | " " |
| Patent leather and kid | .. | 12 " | .. | 12 " |
| Perfumery | .. | " " | .. | " " |
| Candles | 18 *ad val.* | 18 " | .. | 18 " |
| Window glass | 20 " | 20 " | .. | 20 " |
| Sugar, all kinds | " " | " " | .. | " " |
| Alcohol | 18 " | Hecto., 12·60 fr. | .. | Hecto., 12·60 fr. |
| Rocksalt | .. | 100 kilos. net, 3·08 fr. | .. | 100 kilos. net, 3·08 fr. |
| Gunpowder | .. | 100 kilos. net, 112 fr. | .. | 100 kilos. net, 112 fr. |
| Fireworks and other explosives | .. | 100 kilos. net, 140 fr. | .. | 100 kilos. net, 140 fr. |
| Mineral waters | 10 *ad val.* | .. | .. | 10 *ad val.* |
| Soda, alum, ammonium salts, and blue and green vitriol | .. | .. | 12 *ad val.* | .. |
| Copper | .. | .. | 10 " | .. |
| Tinplates | .. | .. | 12 " | .. |
| Naphtha and kerosene | .. | .. | .. | 100 kilos., 1 fr. |

### GENERAL ORDER 1/1898.

Custom House, London,
1st January 1898.

Explosives Act, 1875.

Order in Council prohibiting the Importation of Acetylene in certain Cases.

The accompanying copy of an Order in Council, dated the 26th ultimo, is herewith transmitted for the information and guidance of the officers concerned with the reference to the prohibition therein contained of the importation of acetylene into this country in certain cases.

By order of the Board,
JOHN COLBROUX.

At the Court at Windsor, the 26th day of November 1897.

Present.

The Queen's Most Excellent Majesty in Council.

Whereas by section 104 of the Explosives Act, 1875, it is enacted that Her Majesty may, by Order in Council, declare that any substance which appears to Her Majesty to be specially dangerous to life or property, by reason either of its explosive properties or of any process in the manufacture thereof being liable to explosion, shall be deemed to be an explosive within the meaning of the said Act, and the provisions of the said Act (subject to such exceptions, limitations, and restrictions as may be specified in the Order) shall accordingly extend to any such substance in like manner as if it were included in the term explosive in the said Act:

And whereas acetylene, when liquid or subject to a certain degree of compression, is specially dangerous to life or property by reason of its explosive properties:

Now, therefore, Her Majesty is pleased, by and with the advice of Her Privy Council, to order and declare, and be it ordered and declared, as follows:—

Acetylene, when liquid, or when subject to a pressure above that of the atmosphere capable of supporting a column of water exceeding 100 ins. in height, and whether or not in admixture with other substances, shall be deemed to be an explosive within the meaning of the said Act, subject to the following exception: that if it be shown to the satisfaction of the Secretary of State that acetylene declared to be explosive by this Order when in admixture with any substance, or in any form or condition, is not possessed of explosive properties, the Secretary of State may by Order exempt such acetylene from being deemed to be an explosive within the said Act.

And whereas by section 43 of the Explosives Act, 1875, it is provided that Her Majesty from time to time by Order in Council may prohibit either absolutely or except in pursuance of a licence of the Secretary of State under the said Act, or may subject to conditions or restrictions the manufacture, keeping, importation from any place out of the United Kingdom, conveyance, and sale, or any of them, of any explosive which is of so dangerous a character that, in the judgment of Her Majesty, it is expedient for the safety of the public to make such Order:

And whereas it is, in the judgment of Her Majesty, expedient for the public safety that acetylene, when an explosive within the meaning of this Order, shall be prohibited:

Now, therefore, in pursuance of the above-mentioned provision of this Act, Her Majesty is pleased, by and with the advice of Her Privy Council, to order and prescribe that acetylene declared to be an explosive by this Order shall be prohibited from being manufactured, imported, kept, conveyed, or sold.

C. L. Peel.

## *EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.*

### Trade of Uruguay.

*Foreign Office Annual Series*, 1897, 2020.

According to the official figures now published, it appears that the total imports for the year 1896 were 5,431,931*l*. Comparing these with those given for the year 1895, 5,391,295*l*., there is a small increase of 40,636*l*. in favour of 1896.

The total exports for 1896 are given as amounting to 6,468,710*l*., against 6,924,173*l*. for the year 1895, or a decrease of 455,463*l*.

It is the practice now of the custom-house in Uruguay to put an official valuation, generally a very high one, on all articles imported into the country, for the purpose of applying the tariffs.

This system of over-valuation exists throughout the imports, and tends to check the commerce and trade of the country, for it enhances the sale price of the imported article, and frequently puts it beyond the reach of the ordinary buyer's means. This is especially the case with British goods, which are usually higher priced than those from other countries; hence the introduction of cheap wares from Germany. As regards the exports, no such official valuation is made, the market value being taken as a basis for applying the custom dues, which seems a more reasonable system.

Great Britain holds the prominent position she has hitherto held as regards the imports, for out of a total of 5,431,931*l*. she has contributed no less than 1,548,377*l*., or 28 per cent. of the whole.

The most formidable European rival that Great Britain has in the import trade is Germany, which imports 585,168*l*., France comes next with 529,741*l*., then Italy with 485,816*l*. Argentina imported goods to the value of 749,733*l*., but it should be borne in mind that they consisted chiefly of articles in transit for Monte Video.

Great Britain holds a less prominent position in the exports than she does in the imports, for out of a total of 6,468,710*l*. she takes but 421,968*l*., principally in horns, salted hides, wool, extract of beef, and wheat.

As regards imports, Great Britain is first with sugar, closely followed by Germany and France; then come Italy Brazil, Belgium, and Argentina.

Great Britain is foremost in the supply of prepared sheepskins. France sends most prepared calf- and kid-skins, and furs of various sorts.

Germany supplies sulphates and carbonates of potash. Great Britain, sulphate of copper.

Great Britain supplies 90 per cent. of the linseed oil and machine oil. The United States, turpentine.

Italy has the field to herself in sulphur, France in tiles, Great Britain in varnish and earthenware; she is alone in the supply of coal.

Great Britain sends 50 per cent., or more, of the sheep-dips.

Great Britain is second to Germany in the supply of printing paper, followed by Belgium; but she sends 60 per cent. of the paint; Italy and Germany follow.

Spain sends all the salt.

Great Britain supplies all the soda.

Great Britain, Germany, and Belgium an equal amount of Portland cement.

Germany and Belgium have the importation of sheet-glass to themselves. (Here is a field for British trade.)

France is first with drugs, followed by Germany and Great Britain, then the United States and Italy.

Great Britain has to her credit 50 per cent. of all the porcelain and china wares that reach this country; Germany follows her.

France is first in perfumery, Italy in brown paper, Belgium in white paper, and arms, ammunition, and stearine candles.

Germany supplies 50 per cent. of the starch which comes to Uruguay, Belgium 25 per cent., Great Britain but little; but she sends 25 per cent. of the stationery; Belgium an equal amount.

Great Britain is almost alone in window glass and zinc plates. Belgium supplies bottles.

### Camphor in Formosa.

*Japan.—Foreign Office Annual Series*, 1897, 410, *page* 71.

The trade in camphor will probably undergo some modification. Camphor trees are not found in that part of the island occupied by Chinese settlers. They occur only in the country of the aborigines, or upon the immediate border; and up to the present time the destruction of trees has been carried on in the most wasteful manner. The mode of obtaining supplies of camphor was for foreign merchants through Chinese agents to advance money to the savage chiefs for permission to cut down trees. The stills were erected at the expense of the foreigners, who paid a tax of 8 dols. a still to the Chinese authorities, and a local tax of 10 dols. on each picul (133 lb.) of camphor produced. When the island was ceded to the Japanese, the privileges which foreigners had enjoyed under Chinese rule, of having these camphor establishments in the interior, seemed likely to be withdrawn by the Japanese Government. The Chinese treaty, much more than the Japanese, gives freedom of travel and trade to the foreigner; and if the limitations imposed by our treaty with Japan had been strictly enforced in Formosa, foreigners would have had to retire to the treaty ports. They would have been debarred from distilling or purchasing camphor in the interior, and they would have suffered heavy losses in abandoning the capital already sunk there. Considering that the present treaty had only two more years to run, the Japanese Government has consented to let matters remain *in statu quo*; and when, under the new treaty, foreigners obtain the right to settle anywhere in the interior, they will be able to distil as much as they like. But there is also a probability that the preparation of camphor will be made a Government monopoly. With the Formosan supply under its control the Japanese Government could almost secure a monopoly of the camphor trade, for Japan and Formosa are almost the only sources of supply; and advantage may be taken of this to put Formosa's finances on a satisfactory basis. The lands where the camphor trees grow are not privately owned, as is the best portion of Formosa's fertile plains, so the Government could appropriate the camphor-producing districts without interfering with vested interests.

### Dry Distillation of Sawdust in Norway.

*Pharm. J.* 1898, 111.—*British Consul, Christiania.*

The production of acetic acid, wood naphtha (methyl alcohol), and tar from sawdust is one of the latest enterprises in Norway. A factory has been started at Fredrikstad capable of distilling 10,000 tons of sawdust during the year, and also of manufacturing charcoal briquettes, which are exported to the Netherlands. The acids are chiefly placed on the German market, while the tar is mostly consumed at home. The factory is said to be the first of its kind erected in the country.

### Porcelain Industry in Germany.

*U.S. Cons. Reps., Dec.* 1897, 539.

The porcelain industry in Germany has, of recent years, exhibited a marked development.

In addition to the State factories at Berlin and Meissen (Saxony), there are many private establishments which are devoted to the supply of ordinary consumption, and the serious competition that the latter have to contend against has obliged them to turn out articles distinguished by an ever-increasing variety of design and perfection of manufacture.

According to M. Soulange-Bodin, French Chargé d'Affaires at Berlin, the German public could only find, at one time, high-class porcelain among foreign imports, and more particularly imports from France; but at the present time articles of German manufacture are found everywhere, and frequently on the French markets.

In 1880, the exports of German porcelain only amounted to 66,830 quintals (1 quintal = 220 lb.), while 15 years later they exceeded 201,000 quintals. During the last 10 years, exports have more than doubled, and their value now is about 20,000,000 marks (5,760,000 dols.). The principal markets are the United States and England and her colonies. After these countries come Holland, Switzerland, Austria-Hungary, France, Belgium, the Scandinavian countries, Chile, the Argentine Republic, and Brazil.

The porcelain industry is rapidly growing in importance, and the exports for the first three months of the present year amounted to 43,000 quintals, as compared with 34,000 quintals during the corresponding period of 1895.

### Mining in New Caledonia.

*Bd. of Trade J., Jan.* 1898, 87.

A report, dated 20th October last, has been received at the Foreign Office from Capt. Harford, H.M. Consul at Noumea (New Caledonia), forwarding a statement that has just been published in Noumea, showing the amount of mineral exported from New Caledonia since last January, as follows:—

| | Tons. |
|---|---|
| Cobalt | 3,226 |
| Nickel | 11,185 |
| Chrome | 8,050 |
| Silver lead | 2 |

While the recent investigations on behalf of the "International Mining Company" have thoroughly established the fact of the entire island being, for nickel, copper, chrome, and cobalt, probably the most valuable storehouse in the world, the labour question must not be overlooked; and it is probably a fact that, unless convict labour can be guaranteed, mining operations of any description must be uncertain.

### The Trade of Saigon (Cochin China).

*Bd. of Trade J., Jan.* 1898, 86.

In a report, dated 4th November last, H.M. Consul at Saigon states that, generally speaking, the "Tarif" severely handicaps all foreign manufactures; it is the general tariff of France, rather emphasised for the colony.

*Colours, Chemicals, Chinaware.*—Paints, colours, and varnish are manufactured, but they do not seem to prove a financial success; there is a fairly large establishment which exports a considerable quantity of soap to neighbouring colonies.

*Brewing* has been attempted, but turned out a failure financially.

*Tanning.*—Chinese and natives get out a considerable quantity of leather of an inferior quality, but it is good enough for native use, apparently, and even Europeans put up with it on account of its cheapness.

*Dyeing.*—Natives do this very well indeed, and in very beautiful colours.

### Caoutchouc in Madagascar.

*Bd. of Trade J., Jan.* 1898, 41.

The caoutchouc industry is developing on the coast: trials with the *ceara* species have yielded the best results; sowings made in July developed into young trees of 1·50 metres four months later. Madagascar rubber is derived from trees, shoots, and creepers; that of Fort Dauphin has the peculiarity of coagulating in the open air without the aid of any acid. In 1896, 537 tons of rubber were exported

### Paints in Burmah.

*Bd. of Trade J., Jan.* 1898, 47.

Under the head of "false trade descriptions" there was an increase from one detention in 1895–96 to 17 detentions in 1896–97. Of these, 14 cases were consignments of paints, the descriptions of the kegs containing which were held by the Rangoon Custom-house to be false trade descriptions. These paints were branded and declared as "red lead," "white lead," and "white zinc" paints, it being thereby inferred that the contents were pure lead or zinc, but on analysis by the Chemical Examiner they were found to contain very varying and in some instances almost infinitesimal percentages of red lead, white lead, and white zinc respectively, the balance being described as composed, for example, of so much sulphate of barium or (in one case) of an organic colouring matter and brick dust.

### Industrial Establishments in British Columbia.

*Bd. of Trade J., Jan.* 1898, 52.

*Industrial Establishments.*—British Columbia is the largest manufacturer in the Dominion in proportion to population. The following establishments are mostly able to supply present local demands:—Iron foundries, chemical works, paint works, soap factories, breweries, powder works, and potteries.

The manufacture of wood pulp for export has not yet started in the Province, but the natural conditions appear favourable for such an industry.

Metallurgical works have recently been established at Victoria; capacity at present, 20 tons of ore per day. A chlorination plant is in course of erection, and as soon as the works are completed ores will be treated by several different processes. These works are the first of the kind established in British Columbia, and should be of great value to mine owners.

## *GENERAL TRADE NOTES.*

### The Mining of Magnetic Iron Ore.

*Engineering, Nov.* 12, 1897.

In the early days of iron-mining in the United States of America the centres of the industry were the States of New Jersey, New York, and Pennsylvania. More recently the discovery of huge beds of iron ore in the Southern and Western States, existing in "pockets" in mountain sides, so that the ore has only to be quarried, has placed the iron manufactures of Jersey and New York at a serious disadvantage in commercial competitions. It is because of this that attention has now been directed to the veins of magnetic ore known to exist in New Jersey, and that a company has been formed to obtain the ore in a form suitable for smelting.

The company owns about 16,000 acres of land, and in the district there are six large veins extending about 21 miles and about 750 ft. wide. The possible output of the works is 5,000 tons per day, and it is calculated that at this rate the supply will last 100 years.

The most interesting feature of this mining venture is the ingenuity displayed in saving hand-labour. The mining is done by two steam excavators, the larger of which is easily capable of removing four tons of ore per minute—an amount which represents the labour of 250 men. The ore is thrown by the excavators into cars and taken to crushing mills, where it is gradually reduced to cubes of about 1½ in. side. In these mills many new devices have been adopted to obviate damage to the machinery. "Breaking pins," made to bear a certain load, are used for connecting-up the machinery, and when the load is exceeded, as, for instance, by the introduction of some very hard substance with the ore, the "pin" is shattered and the machinery so thrown out of gear, and no harm is done.

The ore is next sent to the "refining" department, where it is reduced to a powder fine enough to pass through a "14-mesh" screen. It is then allowed to fall in a perpendicular stream over three sets of magnets, which cause the particles of magnetic oxide to be deflected from their course, and so fall into a heap by themselves. The powdered ore is in this way separated into three grades; the poorest of these is returned to the crushing mills, and the other two are mixed and made up into "briquettes" by compression with some binding material. The sand produced as a by-product is sold for the manufacture of cement and mortar, and for use in the sand-boxes of locomotives and street cars.

### Georgia Phosphates.

*Eng. and Mining J., Dec.* 25, 1897, 754.

A preliminary report on a part of the phosphates and marls of the State has just been published by State Geologist W. S. Yates. The report has been prepared by the Assistant State Geologist, S. W. McCallie. The conclusions of the document are as follows :—The result of our investigations in the various countries lying along the Georgia-Florida State line demonstrates to a considerable degree of certainty two very important economic facts. First, that there do not exist anywhere along the State line, with the exception, probably, of Thomas County, any deposits of phosphate of sufficient extent and purity to be mined with profit for the manufacture of commercial fertiliser at its present market value. Second, that all these counties contain more or less extensive beds of marl, or low-grade phosphate—a valuable natural fertiliser that might be used to a great extent in replacing the more costly manures. There can be little doubt that the deposits of marl are extensive, and equal in many respects the greensand beds of New Jersey. In only a few instances have they been tried on growing crops, but in all cases they are reported to have produced beneficial results.

### A New Use for Selenium.

*Eng. and Mining J., Dec.* 18, 1897, 731.

Selenium has recently been employed for producing coloured glass. Rose-tinted glass is made by adding selenium directly to the ingredients in the melting-pot, the depth of tint depending entirely on the quality used, and also to some extent upon the character of the glass—whether it be hard or soft. An orange-red colour is produced by mixing cadmium sulphide with the selenium before adding to the contents of the pot. The intensity of the yellow constituent in this case depends directly upon the proportion of cadmium sulphide made use of. A practical advantage claimed for this process is that it is not necessary to reheat the articles after being manufactured and to dip them in a colouring mixture, as in the ordinary process of making red glass.

### Copper Sulphate in the United States.

*Ibid., Jan.* 8, 1898, 44.

The production of copper sulphate in the United States in 1897 was 49,000,000 lb., against 48,732,840 lb. in 1896. The percentage of copper in this material is about 25. The greater part of the production of copper sulphate is made by chemical works in the East, but a good deal is turned out as a by-product by various gold and silver refiners.

### Chrome Ore in the United States.

*Ibid.*, 45.

The chrome ore industry in California, which for many years has been the only domestic source of supply, became practically defunct in 1897. The San Luis Chrome Company has gone out of business, and the Tehama County deposit is worked out. Howe and Jones, of Hazel Creek, Shasta County, turned out a small amount of low-grade ore, but none was shipped, nor were there any shipments from the State to the Atlantic coast. The total production of the State is estimated at 50 tons, and the general opinion is that the industry has no future.

### Corundum in the United States.

*Ibid.*

The production of corundum in the United States in 1897 was 230 short tons, against 250 short tons in 1896. In each year the product was derived entirely from North Carolina, where the Hampden Corundum and Emery Company, which operates the Corundum Hill mine in Macon County, is the chief producer. Besides this concern there were two small producers, namely, the Turkey Knob Corundum Company and the Savannah Mining Company. The conditions in the abrasive trade of the United States at the present time are such as to discourage the production of corundum here. At present prices it is doubtful if the mining of corundum in North Carolina can be carried on at a profit.

### Cobalt Oxide in the United States.

*Ibid.*

The production of cobalt oxide in the United States in 1897 was 19,300 lb., against 12,825 lb. in 1896. The average value of the product in 1897 was 1·70 dol. per lb. It was derived mostly as a by-product from matte from the lead-smelting furnaces at Mine la Motte, Mo. A discovery of cobalt oxide assaying 1·5 per cent. was reported from Lytle Creek Canyon, near Colton, Cal., and it was said that a company was to be orgaised to exploit it, but, so far, we have been unable to learn further details concerning it. Steps were taken to develop more fully the cobalt mines of the Westerwald, a mountainous district on the line between Rheinish Prussia and Westphalia. This ore is said to average 4 per cent. cobalt and 1 per cent. nickel. The mines are operated by the Westerwald Cobalt Works Company.

### Petroleum in Java.

*Ibid.*, 38.

According to a French Consular report, there are in the district of Lidah and Koetei 40 wells, with an average daily production of 19,800 gals. of oil, which could easily be increased to 25,000 gals. Recent reports are to the effect that two new wells have been discovered, producing some 2,400 barrels per day additional. In the district of Panolan there are 15 wells, the largest of which has a daily production of 2,400 barrels. The wells of Tinawen have been but little exploited. A company has obtained a 10-year concession for this district, and the probable yield is estimated at 1,600 cases a day. There are two refineries, and another in process of construction. The paraffin factory at Ploentoeran, which has a capacity of production of 6,000 lb. a day, is only partially in action. A factory at Wonokrano supplies all the oils necessary for the sugar refineries and for the railroads of Java. Statistics for the last eight years show that the production of refined petroleum on this island increased from 8,000 cases in 1889 to 1,250,000 cases in 1896. Java, on account of its geographical position, finds the cost of transportation to the Eastern markets comparatively small.

### Lead Smelting in Spain.

*Ibid.*, 35.

A letter recently received from Cartagena, Spain, notes the arrival there of a cargo of 2,050 tons of washed lead ore from the Broken Hill Mines in Australia, consigned to a firm of Spanish lead smelters. This was the second cargo of the kind received within a few months. The remarkable point is that we find these lead ores from Australia smelted in Spain with coke imported from great Britain, the pig lead made being exported to England.

### The German Chemical Industry.

*Chem. Trade J., Jan.* 8, 1898, 24.

At the annual conference of the German Society for the Protection of the Interests of Chemical Industry, at Baden-Baden, the president, Dr. Holtz, stated that in 1896 the output of chemical products increased by 8·7 per cent., the average number of workmen employed by 5·2 per cent., and the total amount paid in wages from 103,400,000 to 113,700,000 marks, or from 894 to 907 marks per head. The exports increased by 6·5 per cent. in value and by 13 per cent. in quantity. As a consequence of the general fall in prices, there was a reduction in the average net profits (as shown in the reports of the principal chemical companies) from 12·71 to 12·3 per cent.

### THE BERGAMOT CROP.

*Report of Schimmel and Co., Oct. 1897, 25.*

The flowering and fruiting season promised an abundant crop, but the excessive heat during the summer month proved detrimental to the harvest, particularly in the plantations on the lowlands by the coast, while the orchards of the higher regions remained in good condition. It will depend upon the yield of essential oil in the new fruits whether the present crop and yield of oil will equal that of last year.

### BULGARIAN ROSE OIL.

*Report of Schimmel and Co., Oct. 1897, 48.*

The adulteration of otto of rose, in spite of stringent efforts on the part of the authorities, still remains flagrant. Some dealers, and the press, support the chamber of commerce at Sofia in endeavouring to reform the trade in rose soil and abolish adulteration. Several parcels of Indian geranium oil, locally known as "tersche," have been seized and confiscated by the police, but notwithstanding these efforts, until some more reliable methods of detecting sophistication with certainty are available, it is probable that the evil practice will not be definitely eradicated.—J. O. B.

### NITRATE OF SODA.

*Shipments, Consumption, Stocks, and Prices, from 1893 to 1897.*

| — | 1893. | 1894. | 1895. | 1896. | 1897. |
|---|---|---|---|---|---|
| | Tons. | Tons. | Tons. | Tons. | Tons. |
| Shipments from South American ports to all parts for the six months ending 31st December. | 510,000 | 629,000 | 733,000 | 612,000 | 677,000 |
| Do. for the 12 months ending 31st December | 929,000 | 1,079,000 | 1,218,000 | 1,088,000 | 1,037,000 |
| Afloat for Europe on 31st December | 358,000 | 393,000 | 506,000 | 351,000 | 371,000 |
| Stocks in United Kingdom ports:— | 9,100 | 17,300 | 21,500 | 24,000 | 21,000 |
| Stocks in Continental ports on 31st December | 92,900 | 101,700 | 134,500 | 324,000 | 224,000 |
| Consumption in United Kingdom for the six months ending 31st December. | 29,000 | 29,800 | 36,500 | 24,000 | 32,500 |
| Do. in Continent for the six months ending 31st December | 231,000 | 246,500 | 230,500 | 195,000 | 238,500 |
| Do. in United Kingdom for the 12 months ending 31st December. | 100,000 | 114,500 | 117,500 | 105,000 | 108,500 |
| Do. in Continent " " " | 682,000 | 719,500 | 789,500 | 805,000 | 867,500 |
| Do. in United States " " " | 104,000 | 100,000 | 110,000 | 105,000 | 110,000 |
| Do. in other Countries " " " | .. | .. | 7,000 | 8,000 | 14,000 |
| Do. *in the World* " " " | 886,000 | 964,000 | 1,024,000 | 1,023,000 | 1,100,000 |
| Visible supply on 31st December (including the quantity afloat for Europe and Stocks in United Kingdom and Continent). | 460,000 | 512,000 | 662,000 | 690,000 | 616,000 |
| Price on 31st December per Cwt. | 9s. | 8s. 10½d. | 7s. 7½d. | 7s. 10½d. | 7s. 6d. |

| — | 1893. | 1894. | 1895. | 1896. | 1897. |
|---|---|---|---|---|---|
| | Tons. | Tons. | Tons. | Tons. | Tons. |
| Liverpool | 2,000 | 4,250 | 8,000 | 10,500 | 6,000 |
| London | 3,300 | 1,000 | 2,100 | 2,250 | 1,800 |
| Out ports | 3,800 | 12,050 | 11,400 | 11,300 | 13,200 |

—W. MONTGOMERY & CO.'S CIRCULAR.

### NITRATE OF SODA AND SULPHATE OF AMMONIA IN 1897.

*Nitrate of Soda.*—The year opened with a visible supply somewhat larger than that at the beginning of 1896, but it was calculated by the trade that, with an increase in consumption similar to that during the spring of 1896, available supply would be barely sufficient to meet the demand, and that sellers would thus become masters of the situation.

The supply proved to be more than sufficient. There was no great increase in the consumption of nitrogenous material (nitrate of soda included). Consumers largely used cheaper sulphate of ammonia in place of nitrate of soda; and, instead of a large increase in the consumption of the latter article, January-April, as was reckoned upon, there was an actual decrease.

The nitrate of soda interest, being heavily committed to a high range of values, managed to advance spot prices somewhat in January, and to maintain them fairly well until April; but heavy spring demand not having fully set in until late in March, prices gradually drooped up to the end of June, when the value of ordinary was 7s. 4½d. per cwt., and that of fine quality 7s. 6d. per cwt.

Attempts were made in June, and again later, to bring producers into agreement to curtail the quantity arranged to be shipped, April 1897—March 1898, but without success, and in October the combination came to an end. This upshot had for some time been generally anticipated, and discounted, so that prices remained unmoved; but the breakdown of the combination has, with scarcity of tonnage, been the means of decidedly strengthening the statistical position.

Some officinas at once closed down, and others have since ceased to work. As matters now stand, the fittest only among producers can work at a profit. Thus the quantity intended by the combination to be shipped has been curtailed, though the deficiency has been partly removed by new companies and by increased shipments by old officinas still working. There is, however, a shrinkage in visible supply, as the following figures show:—

Visible supply (stock and afloat) at the end of
1895 was 660,000 tons.
" " 1896 " 690,000 "
" " 1897 " about 620,000 tons.

It is anticipated that the shrinkage will further be decreased by larger shipments in January and February 1898 by outside steamers, and by the regular liners; but, making full allowance for these, there can hardly fail to be a decrease in available supply for January-April of some 40,000 or 50,000 tons.

Deliveries, January-April 1896, were 530,000 tons.
" " 1897 " 560,000 "

The deliveries, January-June 1897, were, however, 710,000 tons, against 690,000 tons January-June 1896; but even these figures cannot be considered as indicating a very satisfactory season's business.

The second half of the year's business wears a more healthy look, deliveries July-December having been 40,000 tons in excess of those July-December 1896.

The outlook seems to promise further improvement. Agricultural prospects are brighter than they were a year ago. There is a marked shrinkage in visible supply. Present price is 6d. per cwt. below that at the end of 1896, whilst the price of sulphate of ammonia is 30s. per ton higher than it was then.

The past year has demonstrated that no price at which nitrate of soda can be sold can ever enable it to drive sulphate of ammonia out of the field. In 1897 the driving has been all the other way. The rate of production of sulphate of ammonia is determined by altogether other considerations than its own market value. Whatever the market price may be, it must be produced and sold. It is otherwise in the case of nitrate of soda. The rate of its output must be determined by its selling value, and production cannot go on much beyond the point when it ceases to be remunerative. These being the facts, it would be to the advantage of producers of both articles if nitrate of soda producers would once for all recognise that any attempt to displace sulphate of ammonia must be futile, and even disastrous. The competition is altogether unequal, and whatever calculations nitrate of soda producers may make as to the world's requirements of nitrogenous material, they should always first allow for the estimated world's production of sulphate of ammonia.

*Sulphate of Ammonia.*—The primary determining factor all through the year has been the selling policy of the large London makers, who, to begin with, held the bulk of the stock. Having in October and November 1896 sold heavily, stock and production, for delivery up to April, they proceeded, early in 1897, to offer further ahead—May-August delivery—at a small premium on spot prices. This policy they consistently pursued right up to the end of September, when stocks were reduced to some seven or eight thousand tons.

This measure involved a serious sacrifice in price (on stock and production) on the part of all holders of stock, and a smaller sacrifice (on current production only) on the part of all other producers. Within the nine months—January-September—the stock in the country was reduced by something like 18,000 tons. It was again *primarily* the relative cheapness of sulphate of ammonia, occasioning a further large reversion from the use of nitrate of soda, which enabled the market to absorb in six months—January-June—fully 16,000 tons of stock, in addition to current production, without at any time allowing prices to be depressed more than 2*s.* 6*d.* per ton below the opening quotation in January. In the second half of the year, when the question of *Sulphate of Ammonia* v. *Nitrate of Soda* scarcely arises, a new factor made itself felt. The rapid increase in production in Germany and France in 1894, 1895, and 1896 had much to do with upsetting the balance of supply and demand, which it has taken nearly all 1896 and 1897 to readjust. There has not within the past year been any great development in production in Germany, whilst in France the output has been barely equal to that in 1896. Recourse has consequently had to be had to the English market on a largely increased scale, by both countries, especially since June, in order to fill the gap thus occasioned.

We think we shall serve our friends better if we try to show where the increased demand has come from, and in what direction. As to home consumption, the increase during the past year—practically all in the first six months—is estimated at 2,000 tons. This does not, however, fully represent the increase in actual consumption within the United Kingdom. The quantity taken by home manure manufacturers has always been considered as home consumption, although a very considerable portion of it has subsequently been exported in the shape of compounds, it being impossible to determine approximately the quantity so exported. The development of manure manufacturing and mixing abroad has brought about a serious curtailment of exports of mixed manures in recent years, and, of course, a corresponding reduction in the quantity of sulphate of ammonia considered as home consumption, but actually sent out of the country. The shrinkage in this direction during the past twelve months has been particularly marked. Probably it amounts to several thousand tons, and this would have to be taken into account before the increased quantity actually consumed in the United Kingdom in 1897 could be determined.

It is understood that producers contemplate introducing prizes for the best crops grown with sulphate of ammonia. For many years manure manufacturers have found it to their interest thus to encourage the use of their manures.

The proposed offering of prizes for essays upon the best methods of applying this manure will also, no doubt, stimulate experiment and investigation on the part of experts.

As to exports, the increase to France in 1897 is larger than that to any other country—no less than 10,400 tons, as compared with 1896—5,500 tons January-June, and 4,900 tons July-December. To Germany and Belgium together, the total increase has been 11,800 tons—7,000 tons January-June, and 4,800 tons July-December. France and Germany are beyond all other countries nitrate of soda ground, their consumption being nearly two-thirds of the whole taken by Europe, and it is here where consumption of sulphate of ammonia has made most progress. The reason for the increase January-June—the nitrate of soda season—was the relative cheapness, but for the increase July-December there is another cause. Owing to development of their home production, especially from coke ovens, shipments to France and Germany became a decreasing quantity in 1894 and 1895. In 1896 there was still continued development of production, but increased consumption, owing to relative cheapness, made it necessary to again have larger recourse to the English market for supplies. During 1897 production in France has rather decreased. In Germany there has been increase, but not more than normal. Hence the largely increased shipments to France, Germany, and Belgium in the second half of the year.

The next largest increase in exports was that to Spain—6,200 tons. Here nitate of soda does not come seriously into play, so that there can be no question of displacement. In 1896 there was an actual decrease in shipments, as compared with 1895, of 1,000 tons. The falling off in demand for mixed manures has probably had much to do with the increase. Buyers in Spain, as elsewhere, are now buying raw materials and doing the mixing themselves, and this has been particularly noticeable in 1897.

Demand from the United States has been disappointing, and especially in view of the quite exceptional demand for tanking, dried blood, and kindred nitrogenous material.

American shipments from United Kingdom in 1897 show a decrease of about 1,000 tons as compared with 1896.

There has been some falling off in shipments to sugar-growing colonies, and under present circumstances further decline must be looked for.

Production in the United Kingdom during 1897, from all sources, we estimate at 202,000 tons, *viz.*:—

| | Tons. |
|---|---|
| Gasworks | 136,000 |
| Ironworks | 18,500 |
| Shaleworks | 37,000 |
| Coke and carbonising works | 10,500 |
| Total | 202,000 |

Of this production we estimate that—

| | Tons. |
|---|---|
| England contributed | 130,300 |
| Scotland „ | 69,000 |
| Ireland „ | 2,700 |

The production during the previous five years was:—

| — | 1896. | 1895. | 1894. | 1893. | 1892. |
|---|---|---|---|---|---|
| | Tons. | Tons. | Tons. | Tons. | Tons. |
| Gasworks | 127,500 | 119,600 | 113,500 | 112,000 | 110,750 |
| Ironworks | 16,500 | 14,600 | 10,000 | 8,800 | 11,000 |
| Shaleworks | 38,000 | 38,300 | 33,000 | 28,500 | 23,100 |
| Coke and carbonising works. | 9,000 | 7,000 | 3,500 | 3,200 | 5,000 |
| Total | 191,000 | 179,500 | 160,000 | 152,500 | 149,850 |

Production in 1896 proved to be rather over our estimate, and stock in makers' hands was also larger than we reckoned upon. The continued increase in the consumption of coal-gas is remarkable, having regard to the extended use of the electric light, of water-gas, and other illuminants, and gives evidence of the prosperity of the manufacturing and other industries of the country. It will be observed that there has been a further large increase in the production of sulphate of ammonia from this source. There has also been an increase from blast furnaces and from coke and carbonising works, but from shale there has been a smaller output. Continental production continues to increase, but not at the rapid rate of 1894, 1895, and 1896. In France there has been an actual decrease. Further coke ovens are said to be in course of erection in Germany, from which a considerable quantity will be turned out, but they will not come fully into play for some time.

The year opened with a stock of about 25,000 tons, and there has been an increase in production of 11,000 tons as compared with 1896. Total exports in 1897 have been 153,000 tons, against 127,000 tons in 1896—an increase of 26,000 tons. Home consumption was about 70,000 tons (for agricultural and chemical purposes—including the liquor used in various processes of manufacture)—or 2,000 tons in excess of that in 1896. Stock, at the close of the year, was about 4,000 tons—a bare working stock, and practically an irreducible minimum.

The agricultural outlook is decidedly brighter than it was a year ago. The larger direct use, and wider experience, of sulphate of ammonia will at least predispose farmers to use it again. Current production only will be available, so that if there should be absolutely no increase in consumption over the next six months, there would not be nearly enough sulphate of ammonia to meet the demand, assuming an increased production at the ratio of that in 1896 and 1897.—BRADBURY AND HIRSCH'S REPORT.

## *BOARD OF TRADE RETURNS.*

### SUMMARY OF IMPORTS.

| Articles. | Year ending 31st December | |
|---|---|---|
| | 1896. | 1897. |
| | £ | £ |
| Metals | 20,492,024 | 21,265,363 |
| Chemicals and dyestuffs | 6,776,920 | 6,006,210 |
| Oils | 8,459,394 | 7,641,231 |
| Raw materials for non-textile industries. | 47,241,991 | 52,085,336 |
| Total value of all imports | 141,808,904 | 151,238,683 |

### SUMMARY OF EXPORTS.

| Articles. | Year ending 31st December | |
|---|---|---|
| | 1896. | 1897. |
| | £ | £ |
| Metals (other than machinery) | 33,519,088 | 34,487,868 |
| Chemicals and medicines | 8,242,336 | 8,674,864 |
| Miscellaneous articles | 33,552,986 | 32,039,649 |
| Total value of all exports | 240,145,551 | 234,350,063 |

### IMPORTS OF OILS FOR YEAR ENDING 31ST DECEMBER.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1896. | 1897. | 1896. | 1897. |
| | | | | £ | £ |
| Cocoa-nut | Cwt. | 219,158 | 242,731 | 249,633 | 265,668 |
| Olive | Tons | 18,853 | 15,630 | 612,876 | 515,531 |
| Palm | Cwt. | 1,146,241 | 973,108 | 1,204,679 | 1,001,368 |
| Petroleum | Gall. | 189,953,945 | 186,706,160 | 3,732,056 | 3,351,539 |
| Seed | Tons | 33,610 | 33,174 | 634,995 | 699,594 |
| Train, &c. | Tons | 21,632 | 18,129 | 365,191 | 280,077 |
| Turpentine | Cwt. | 498,791 | 502,790 | 490,381 | 516,887 |
| Other articles | Value £ | .. | .. | 1,169,583 | 1,010,567 |
| Total value of oils | | .. | .. | 8,459,394 | 7,641,231 |

### IMPORTS OF METALS FOR YEAR ENDING 31ST DECEMBER.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1896. | 1897. | 1896. | 1897. |
| Copper:— | | | | £ | £ |
| Ore | Tons | 87,335 | 82,595 | 566,791 | 476,626 |
| Regulus | „ | 90,790 | 88,591 | 2,295,509 | 2,275,780 |
| Unwrought | „ | 61,162 | 61,089 | 2,882,253 | 3,010,758 |
| Iron:— | | | | | |
| Ore | „ | 5,438,307 | 5,968,680 | 3,778,789 | 4,435,934 |
| Bolt, bar, &c. | „ | 71,058 | 68,132 | 570,112 | 539,509 |
| Steel, unwrought | „ | 17,491 | 39,989 | 157,746 | 266,131 |
| Lead, pig and sheet | „ | 167,799 | 167,442 | 1,855,743 | 2,033,300 |
| Pyrites | „ | 589,056 | 623,041 | 999,793 | 1,020,896 |
| Quicksilver | Lb. | 3,536,928 | 4,105,053 | 327,729 | 390,881 |
| Silver ore | Value £ | .. | .. | 1,296,736 | 1,429,842 |
| Tin | Cwt. | 767,495 | 535,716 | 2,289,688 | 1,623,798 |
| Zinc | Tons | 76,635 | 69,812 | 1,245,013 | 1,183,389 |
| Other articles | Value £ | .. | .. | 2,225,822 | 2,542,519 |
| Total value of metals | | .. | .. | 20,492,024 | 21,265,363 |

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR YEAR ENDING 31ST DECEMBER.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1896. | 1897. | 1896. | 1897. |
| | | | | £ | £ |
| Alkali | Cwt. | 176,156 | 227,494 | 96,130 | 109,062 |
| Bark (tanners', &c.) | „ | 309,614 | 313,985 | 113,369 | 108,707 |
| Brimstone | „ | 452,565 | 449,028 | 94,012 | 102,209 |
| Chemicals | Value £ | .. | .. | 1,384,595 | 1,356,380 |
| Cochineal | Cwt. | 5,697 | 4,729 | 36,658 | 29,103 |
| Cutch and gambier | Tons | 26,844 | 25,018 | 548,756 | 418,128 |
| Dyes:— | | | | | |
| Alizarin | Value £ | .. | .. | 256,344 | 223,875 |
| Anilin and other | „ | .. | .. | 485,003 | 471,543 |
| Indigo | Cwt. | 89,911 | 82,526 | 1,533,722 | 1,470,574 |
| Nitrate of potash | „ | 340,750 | 329,614 | 278,484 | 246,699 |
| Valonia | Tons | 32,808 | 29,637 | 333,225 | 300,324 |
| Other articles | Value £ | .. | .. | 1,616,622 | 1,169,666 |
| Total value of chemicals | | .. | .. | 6,776,920 | 6,006,210 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR YEAR ENDING 31ST DECEMBER.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1896. | 1897. | 1896. | 1897. |
| | | | | £ | £ |
| Bark, Peruvian | Cwt. | 35,291 | 21,674 | 61,578 | 52,463 |
| Bristles | Lb. | 4,056,279 | 4,007,892 | 556,068 | 514,123 |
| Caoutchouc | Cwt. | 431,148 | 398,318 | 4,991,122 | 4,546,841 |
| Gum:— | | | | | |
| Arabic | „ | 80,506 | 63,208 | 105,752 | 127,611 |
| Lac, &c. | „ | 157,352 | 177,557 | 776,217 | 751,099 |
| Gutta-percha | „ | 43,805 | 41,442 | 401,190 | 400,875 |
| Hides, raw:— | | | | | |
| Dry | „ | 369,063 | 556,587 | 905,125 | 1,413,166 |
| Wet | „ | 601,734 | 638,658 | 1,319,516 | 1,330,988 |
| Ivory | „ | 10,911 | 10,288 | 459,181 | 422,398 |
| Manure:— | | | | | |
| Guano | Tons | 20,214 | 16,164 | 101,354 | 85,537 |
| Bones | „ | 66,681 | 68,208 | 251,898 | 213,512 |
| Nitrate of soda | „ | 106,445 | 105,832 | 836,552 | 811,865 |
| Phosphate of lime | „ | 291,314 | 325,133 | 465,931 | 493,212 |
| Paraffin | Cwt. | 725,398 | 773,301 | 711,050 | 675,011 |
| Linen rags | Tons | 21,065 | 25,233 | 206,772 | 249,631 |
| Esparto | „ | 187,278 | 204,579 | 796,683 | 825,195 |
| Pulp of wood | „ | 327,080 | 388,204 | 1,681,647 | 1,939,367 |
| Rosin | Cwt. | 1,655,374 | 1,640,745 | 419,563 | 407,771 |
| Tallow and stearin | „ | 2,349,749 | 1,950,976 | 2,178,852 | 1,870,289 |
| Tar | Barrels | 115,939 | 106,355 | 74,080 | 68,362 |
| Wood:— | | | | | |
| Hewn | Loads | 2,432,790 | 2,827,740 | 4,889,374 | 5,781,070 |
| Sawn | „ | 6,031,192 | 7,018,242 | 13,380,580 | 16,628,200 |
| Staves | „ | 138,393 | 126,745 | 656,246 | 569,572 |
| Mahogany | Tons | 53,772 | 73,513 | 477,265 | 658,510 |
| Other articles | Value £ | .. | .. | 10,412,027 | 11,242,659 |
| Total value | | .. | .. | 47,241,991 | 52,085,336 |

Besides the above, drugs to the value of 940,185*l*. were imported, as against 935,978*l*. in 1896.

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR YEAR ENDING 31ST DECEMBER.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1896. | 1897. | 1896. | 1897. |
| | | | £ | £ |
| Brass............ Cwt. | 119,483 | 112,465 | 492,623 | 491,674 |
| Copper:— | | | | |
| Unwrought.... „ | 466.492 | 418,343 | 1,139,511 | 1,076,231 |
| Wrought...... „ | 306,956 | 300,686 | 889,024 | 925,886 |
| Mixed metal... „ | 224,967 | 220,326 | 515,302 | 528,148 |
| Hardware...... Value £ | .. | .. | 2,122,404 | 2,107,264 |
| Implements..... „ | .. | .. | 1,413,669 | 1,364,306 |
| Iron and steel... Tons | 3,550,398 | 3,691,065 | 23,801,700 | 24,639,643 |
| Lead............ „ | 41,214 | 40,503 | 523,367 | 542,088 |
| Plated wares... Value £ | .. | .. | 388,251 | 419,661 |
| Telegraph wires. „ | .. | .. | 857,174 | 1,001,102 |
| Tin............ Cwt. | 123,998 | 99,404 | 397,478 | 323,200 |
| Zinc............ „ | 189,757 | 157,733 | 134,529 | 123,833 |
| Other articles.. Value £ | .. | .. | 874,056 | 944,712 |
| Total value....... | .. | .. | 33,549,088 | 34,487,808 |

### EXPORTS OF DRUGS AND CHEMICALS FOR YEAR ENDING 31ST DECEMBER.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1896. | 1897. | 1896. | 1897. |
| | | | £ | £ |
| Alkali........... Cwt. | 4,841,300 | 4,976,118 | 1,242,173 | 1,278,690 |
| Bleaching materials „ | 1,188,400 | 1,169,351 | 406,486 | 371,480 |
| Chemical manures Tons | 354,357 | 407,982 | 1,807,087 | 1,968,539 |
| Medicines....... Value £ | .. | .. | 1,122,201 | 1,132,636 |
| Other articles... „ | .. | .. | 3,664,989 | 3,923,519 |
| Total value.......... | .. | .. | 8,242,936 | 8,674,864 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR YEAR ENDING 31ST DECEMBER.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1896. | 1897. | 1896. | 1897. |
| | | | £ | £ |
| Gunpowder...... Lb. | 8,089,000 | 8,852,700 | 191,877 | 200,984 |
| Military stores.. Value £ | .. | .. | 1,824,595 | 1,347,462 |
| Candles.......... Lb. | 23,539,800 | 24,461,300 | 357,024 | 331,881 |
| Caoutchouc..... Value £ | .. | .. | 1,261,774 | 1,234,235 |
| Cement.......... Tons | 353,769 | 391,755 | 580,417 | 647,919 |
| Products of coal Value £ | .. | .. | 1,833,726 | 1,668,084 |
| Earthenware... „ | .. | .. | 1,778,735 | 1,725,885 |
| Stoneware...... „ | .. | .. | 188,515 | 172,779 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 1,804,000 | 1,520,561 | 102,538 | 88,469 |
| Flint........... Cwt. | 99,875 | 93,383 | 227,697 | 219,583 |
| Bottles......... „ | 783,660 | 811,285 | 370,749 | 381,463 |
| Other kinds.... „ | 242,887 | 233,679 | 191,505 | 182,392 |
| Leather:— | | | | |
| Unwrought.... „ | 138,071 | 157,392 | 1,280,174 | 1,389,311 |
| Wrought..... Value £ | .. | .. | 394,963 | 409,754 |
| Seed oil.......... Tons | 58,247 | 55,290 | 1,085,551 | 940,345 |
| Floorcloth ..... Sq. Yds. | 23,419,700 | 24,777,700 | 954,621 | 1,041,810 |
| Painters' materials Val. £ | .. | .. | 1,658,180 | 1,598,607 |
| Paper........... Cwt. | 1,060,243 | 1,003,924 | 1,629,971 | 1.523,401 |
| Rags............ Tons | 53,001 | 57,633 | 287,248 | 299,320 |
| Soap............. Cwt. | 719,700 | 738,564 | 745,894 | 762,248 |
| Total value.......... | .. | .. | 33,552,986 | 32,939,649 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

29,500. E. E. Murphy. Improvements in and relating to apparatus for carbonating water and other liquids. Complete Specification. Dec. 13.

29,576. R. H. J. G. Huet. An improved separator for use in connection with granulated substances, liquids, and the like. Complete Specification. Dec. 14.

29,636. F. W. Saatmann. Improvements in drying and absorbing apparatus. Dec. 14.

29,763. H. Kühne. Apparatus for measuring the velocity of air and other gas currents. Dec. 15.

29,786. J. Klein. Improvements in and connected with apparatus for cooling water and other liquids. Complete Specification. Dec. 16.

30,273. G. R. Barclay. Improved pressure filter and percolater. Dec. 22.

30,423. Sir C. S. Forbes, Bart. Improvements in or connected with condensers. Dec. 23.

30,425. Sir C. S. Forbes, Bart. Improvements in or connected with condensers. Dec. 23.

30,595. W. F. Hogg. Improvements in distilling apparatus. Dec. 28.

30,616. J. McNeil and J. Lewkowitsch. Improvements in evaporating or concentrating apparatus. Dec. 28.

30,854. J. Gallegos. Improvements in distilling apparatus. Dec. 31.

1898.

81. J. C. Montgomerie. Improvements in filter-presses and in cloths therefor. Jan. 1.

964. D. Black and J. M. Wright. Improvements in filter-presses and similar filtering apparatus. Jan. 13.

1033. A. J. Boult.—From E. L. Delsol, France. Improvements in or relating to filters. Jan. 13.

1048. P. Y. Cree. An improved composition for preventing corrosion in steam boilers. Jan. 14.

1087. H. Beyer and F. Recke. Economical kiln. Complete Specification. Jan. 14.

### COMPLETE SPECIFICATIONS ACCEPTED.*

1896.

29,576. W. W. Hughes. Electric furnaces. Dec. 29.

1897.

1663. R. Wright. Apparatus for compressing air and other gases. Dec. 22.

3027. Sir C. S. Forbes, Bart. Condensers. Dec. 22.

3482. J. B. Alliott. Evaporating process and apparatus applicable for concentrating, cooling, and other purposes. Dec. 22.

3483. J. B. Alliott. Methods of and apparatus for cooling and evaporating. Dec. 22.

3592. J. E. Campbell. *See* Class VII. Dec. 29.

4095. H. Howell. Valves especially adapted for use in self-discharging saturators used in the manufacture of sulphate of ammonia from gas liquor or ammoniacal liquor. Jan. 12.

4348. C. Bourdon. Improvements in condensers. Jan. 12.

15,529. G. C. Henning. Recording or indicating apparatus for testing materials. Jan. 19.

22,236. J. D. Darling and C. L. Harrison. Porous diaphragms for electrolytic apparatus. Dec. 29.

22,958. L. H. von Gehlen. Air compressors. Dec. 22.

24,041. R. G. Ferguson and A. M. Elkus. Apparatus for mixing liquids with gases. Jan. 19.

26,664. F. W. Lord. Apparatus for cooling and drying air to be used in storage chambers containing perishable produce. Dec. 22.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

29,133. H. Kinsey. Improvements in apparatus for generating and storing acetylene gas. Dec. 13.

29,475. P. Dvorkovitz. Improvements in the manufacture and production of mantles or hoods for what are known as incandescent gas lamps and the like. Dec. 13.

29,571. H. de Thiersant and W. A. Coulson. Acetylene gas producer. Dec. 14.

29,695. W. P. Thompson.—From The Compressed Coal Company, United States. Improvements in fuel. Complete Specification. Dec. 14.

29,617. E. Loup. Improvements in generators for acetylene. Dec. 14.

29,628. H. H. Lake.—From W. A. Shearer, South African Republic. Improved machine for the production of acetylene gas from calcium carbide. Dec. 14.

29,683. J. S. Goodwin. Goodwin's safety acetylene generator. Dec. 15.

29,767. W. B. Smith. Improvements in mantles to be heated by gas. Dec. 16.

29,796. M. Bernstein. A process for manufacturing incandescent bodies. Complete Specification. Dec. 16.

29,797. M. Bernstein. A process for manufacturing incandescent bodies. Complete Specification. Dec. 16.

29,828. E. Jurgens and J. F. C. Jürgens. Improvements in the manufacture of igniting-pills for illuminating-gas. Complete Specification. Dec. 16.

29,830. H. Walker. Improvements in connection with burners for acetylene and other gases. Dec. 16.

29,843. F. H. Smith. Improvements in the method of and apparatus for the production of acetylene. Dec. 16.

29,960. M. Wagner. Improvements in apparatus for the manufacture of acetylene gas. Filed Dec. 17. Date applied for July 13, 1897, being date of application in France. Complete Specification.

29,982. H. Drake. Improvements in or relating to incandescent gas-light burners. Dec. 18.

30,015. H. de Thiersant and W. A. Coulson. "The Sparkler" acetylene gas producer. Dec. 20.

30,080. M. Brand. Improvements in coke ovens and in the accessories appertaining thereto. Dec. 20.

30,112. G. L. Albert, Count de Hamel de Manin. Improvements in or connected with the manufacture of incandescence bodies for illuminating purposes. Dec. 20.

30,145. A. J. Boult.—From The Firm Chemische Fabrik von Max Jasper, Germany. Improved manufacture of incandescent bodies for illuminating purposes. Dec. 20.

30,272. W. H. Baughan. Improvements in apparatus for generating, cooling, and storing acetylene gas. Dec. 22.

30,288. T. Foules and W. Haworth. Improvements in automatic apparatus for the generation and storage of acetylene gas. Dec. 22.

30,321. J. C. H. Kramers and A. E. Kiderlen. Improvements in or relating to apparatus for enriching generator gas. Dec. 22.

30,422. Sir C. S. Forbes, Bart. Improvements in or connected with acetylene lamps. Dec. 23.

30,424. Sir C. S. Forbes, Bart. Improved apparatus for generating acetylene gas. Dec. 23.

30,475. J. St. C. Legge and A. S. Cooper. An improved generator for acetylene gas for use on cycles and other vehicles. Dec. 24.

30,585. J. McConechy. Improvements in apparatus for the production and storage of acetylene gas. Dec. 28.

30,635. W. Ireland. Improvements in acetylene gas generators. Dec. 28.

30,637. J. Mucke and J. Mücke. Improvements in acetylene gas generators. Complete Specification. Dec. 28.

30,673. A. M. Chambers and J. H. Worrall. Improvements in coke ovens. Dec. 28.

30,689. G. W. Lee, F. Elliott, and G. Tatham. Process and apparatus for the manufacture of inflammable gas, and composition to be used therein. Complete Specification. Dec. 28.

30,690. J. St. C. Legge and A. S. Cooper. An improved generator for acetylene gas. Complete Specification. Dec. 28.

30,721. J. H. Noad and F. Hutchins. Improvements in the manufacture of mantles and fringes for incandescent lighting. Dec. 29.

30,833. C. E. Yvonneau. *See* Class VII.

1898.

28. J. McConechy. Improvements in burners applicable to acetylene and rich oil-gas lighting. Jan. 1.

49. B. Puchmüller. An improved fluid, and process of manufacturing same, for impregnating bodies or suitable fabrics, threads, fibres, or the like for incandescent lighting purposes. Jan. 1.

99. J. St. C. Legge and A. S. Cooper. Apparatus for purifying, cooling, drying, and arresting the carbon in acetylene gas. Jan. 3.

157. A. C. Southwell. Improvements in the treatment of gases and vapours of metallic oxides and sulphides, and oxides of sulphur, for the purpose of effecting their deoxidation, and the recovery of useful products, and in apparatus therefor. Jan. 3.

315. A. Z. Germains. Improvements in and connected with means for lighting, heating, and smoke-consuming. Jan. 5.

391. A. S. Bower. Improvements in the apparatus for and in the process of manufacturing water-gas, and in the methods of carburetting or enriching the same for illuminating purposes. Jan. 6.

401. B. W. Levy, of the Firm of D. Cohen and Co., W. Hooker, and R. Morrell. Improvements in the manufacture of incandescing media for lighting purposes. Jan. 6.

457. E. Barnard. An improved apparatus for generating and using acetylene or other gas, either for table or other lamps, or larger installations. Jan. 7.

459. J. F. McCormick. Improved means of purifying and increasing the illuminating power of gas. Jan. 7.

528. A. Williamson. Improvements in or relating to acetylene gas generators for lighting purposes. Jan. 8.

588. B. J. Atterbury and The Alcohol Syndicate, Ltd. An improved production of ethylene gas. Jan. 8.

602. A. M. Clark.—From H. Hiemann and Co., Germany. An improved process for the production of acetylene gas. Jan. 8.

694. C. Jousset. Improvements relating to generators and lamps for the production and use of acetylene gas. Jan. 10.

891. S. T. Richardson and R. Price. Improvements in generators and lamps for acetylene gas. Jan. 12.

934. J. De Brouwer. Improved means and apparatus for charging gas retorts. Jan. 12.

1005. J. O. O'Brien.—From P. P. H. Macé and de Burgue, France. Improvements in apparatus for producing acetylene gas. Jan. 13.

1013. E. Bournonville. Improvements in acetylene gas generators. Complete Specification. Jan. 13.

1154. J. Drummond, jun. Acetylene gas generator and holder. Jan. 15.

1158. R. E. Sprott. Improvements in or relating to the use of calcium carbide in the production of acetylene gas. Jan. 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1896.

26,873. W. B. Hartridge. Fuel blocks applicable as fire-lighters. Dec. 29.

29,736. C. Colas. Means for incandescent lighting. Dec. 22.

29,750. G. Claude and A. Hess. Storing acetylene for lighting and other purposes. Dec. 31.

1897.

1194. H. Simon. By-product coke ovens. Jan. 19.

2071. J. Bowing. Process of and apparatus used for coking. Jan. 19.

2428. H. R. Bean and H. Ringwood. Generator, condenser, and holder for acetylene and other gas. Jan. 12.

3891. J. W. Leadbeater. Admixing and preparing petroleum and other matter in compound form with a view to its final use as a combustible. Dec. 22.

3892. J. W. Leadbeater. Manufacture of briquettes or blocks of fuel from waste products or otherwise enriched by petroleum compounds. Dec. 22.

4583. W. P. Thompson.—From The Continental Gas Gluhlicht Actien Gesellschaft Meteor vormals Kroll, Berger, and Co. Incandescent burners. Dec. 22.

4761. P. A. M. Lacroix. Automatic apparatus for producing acetylene gas. Jan. 12.

6292. A. Stowell. Incandescent lamp. Jan. 12.

9857. E. K. Hution and W. Hutton. Acetylene gas generator for lighting cycle and carriage lamps. Jan. 19.

14,905. H. Sefton Jones. From P. P. H. Macé, France. Production of acetylene gas. Dec. 22.

24,301. J. McConechy. Apparatus for the production of acetylene gas. Jan. 12.

26,435. J. Mücke and J. Mücke. Apparatus for generating acetylene gas. Jan. 19.

26,633. G. W. Chalmers. Incandescent gas burners wherein fragile mantles are used. Dec. 22.

27,767. J. H. Schülke. Acetylene gas generator. Jan. 12.

28,094. W. Tyree. Acetylene gas generator. Jan. 12.

28,102. M. Waguer. Apparatus for generating acetylene gas. Jan. 12.

29,421. D. Kempe. Apparatus for burning vaporised petroleum and the like for heating and illuminating purposes. Jan. 19.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Application.

1898.

108. C. D. Abel.—From E. Theisen, Germany. Method and apparatus for treating the distillation products from carbonaceous materials and the like. Jan. 6.

## IV.—COLOURING MATTERS and DYES.

### Applications.

29,631. J. Imray.—From Société Anonyme de Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of green and blue-green colouring matters derived from triphenylmethane. Dec. 14.

30,118. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of benzaldehyde, *o*-nitrobenzaldehyde, and *p*-nitrobenzaldehyde. Dec. 20.

30,291. I. Levinstein and Levinstein, Ltd. Manufacture or production of colouring matters. Dec. 22.

30,446. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. *See* Class VI.

1898.

75. J. Y. Johnson.—From F. E. Bucher, Jamaica. Improved extract of logwood, and method of obtaining the same. Complete Specification. Jan. 1.

441. S. Pitt.—From L. Cassella and Co., Germany. Production of dinitro and diamido phenylnaphthylaminesulpho acids and phenylamidonaphtholsulpho acids. Jan. 6.

445. S. Pitt.—From L. Cassella and Co., Germany. Production of phloroglucine. Jan. 6.

676. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. The manufacture of new colouring matters. Jan. 10.

### Complete Specifications Accepted.

1897.

4657. J. Turner and Co., Ltd., and P. Schidrowitz and O. Rosenheim. New products from piperidine. Dec. 29.

5119. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Dyeing half-wool with basic azo dyestuffs in an acid bath. Dec. 22.

5259. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Dyeing fast tannin antimony lakes of polyazo dyestuffs and basic safranine azo dyestuffs on the vegetable fibre. Jan. 19.

5351. A. G. Green, A. R. Wahl, and The Clayton Aniline Co., Ltd. Derivatives of diphenylethane and of diphenylethylene. Dec. 29.

5691. H. R. Vidal. Preparation of paradioxythiazine and paramidoxythiazine. Jan. 12.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

29,613. A. Wyser. Apparatus for mercerising yarn. Dec. 14.

29,766. J. V. Esop. Process for rendering fabrics and other materials acid-proof. Complete Specification. Dec. 16.

29,842. T. B. Powell. Improvements in coating textile fabrics and other materials. Dec. 16.

30,023. F. Walton. Improvements in the manufacture of figured mosaic floorcloth, and apparatus for that purpose. Dec. 18.

30,095. E. Stephens. An improved method of and composition for waterproofing textile fabrics and other materials. Dec. 20.

30,142. W. E. Aykroyd, W. H. Aykroyd, and H. E. Aykroyd. Improvements in or relating to the mercerisation of cotton or other fibrous materials or similar substances, and machinery therefor. Dec. 20.

30,916. W. P. Thompson.—From J. Rudolf, Germany. An improved process for rendering textile materials, paper, wood substances, and the like waterproof, and for finishing and protecting them against spotting and moths. Complete Specification. Dec. 31.

1898.

634. F. W. Petrie. Improvements in and relating to machines for drying wool or other fibrous materials. Jan. 10.

703. J. Longmore. Improvements in or relating to the degumming or bleaching of rhea fibre. Jan. 10.

704. J. Longmore. Improvements in or relating to the roving of rhea fibre. Jan. 10.

705. J. Longmore. Improvements in or relating to the spinning or dyeing of yarns. Jan. 10.

706. J. Longmore. Improvements in or relating to the preparation of rhea fibre for spinning. Jan. 10.

707. J. Longmore. Improvements in or relating to the spinning of rhea fibre. Jan. 10.

881. C. Geige. Improved process for the manufacture of chemically prepared peat fibres. Complete Specification. Jan. 12.

925. A. J. Boult.—From A. Monlong and L. Crouzat, France. Improvements in or relating to the rendering of fabrics waterproof. Jan. 12.

### Complete Specifications Accepted.

1897.

2549. S. Shorter and S. Stanbridge. Apparatus for the extraction, cleaning, and preparation of vegetable fibres. Jan. 12.

3218. A. G. Bonbon. Mercerising textile materials. Dec. 22.

6038. J. Morton. Manufacture of figured fabrics. Jan. 12.

7093. C. D. Abel.—From J. Kleinewefers Sohne. Apparatus for mercerising cotton in the form of hanks. Jan. 19.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### Applications.

29,533. W. G. Heys.—From A. Lorthiois, France. Improvements in bleaching camel's hair, goat's hair, and the like. Dec. 14.

30,038. O. P. Amend. Process of mordanting and dyeing animal fibre. Complete Specification. Dec 18.

30,039. O. P. Amend. Process of mordanting animal fibre. Complete Specification. Dec. 18.

30,146. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of fast brown to brown-black tints on the fibre by combining naphthol with the diazo compound of diamidocarbazol. Dec. 23.

30,521. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. An improved process for producing fast shades on cotton. Dec. 24.

1898.

705. J. Longmore. *See* Class V.

### Complete Specifications Accepted.

1897.

5122. S. Pitt. From L. Cassella and Co. Producing fast dyeings upon the fibre by means of direct-dyeing colouring matters. Jan. 12.

5573. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Dyeing cotton. Jan. 12.

6132. W. E. Kay and The Thornliebank Co., Ltd. Fixation of pigments or metallic powders in printing on textile fabrics or similar surfaces. Jan. 12.

9011. L. Koenig, jun. Appliance for dyeing and bleaching textile staples, filaments, or fibres, on tubes and bobbins. Jan. 19.

24,868. J. Stockhausen. *See* Class XII.

27,690. W. Beaumont. Apparatus for dyeing, bleaching, and otherwise treating cops of yarn. Jan. 12.

28,115. C. Spindler and C. Gohring. Treatment of silk for dyeing and bleaching. Jan. 12.

## VII.—ACIDS, ALKALIS, AND SALTS.

### Applications.

29,529. T. Jennings. An improved process for the production of magnesium hydrate. Dec 14.

29,716. P. Jensen.—From L. Rohrmann and H. H. Niedenfuhr, Germany. Process and apparatus for obtaining nitric acid from low graded nitro oxygen compounds. Dec. 15.

30,053. J. Hutton. *See* Class X.

30,319. G. de Bechi. An improved process for the manufacture of nitrate of soda. Dec. 22. Date applied for, June 25, 1897, being date of application in France.

30,344. G. Bischof. Apparatus for recovering acetate liquor from white lead, and washing the white lead. Dec. 23.

30,538. W. G. Sellar. Improvements in the production of cyanides or cyanogen compounds. Dec. 24.

30,574. S. Jaques. Improvements in the recovery of chemicals from spent soda and other liquors. Dec. 28.

30,843. C. E. Yeoman. Improvements relating to the treatment of calcium carbonate. Dec. 30.

1898.

32. J. Leede. Improvements in the art of producing calcium carbide. Complete Specification. Jan. 1.

223. F. G. Scott. Improvements in the manufacture of acetic acid. Jan. 4.

233. Count H. Plater Syberg. Process for the production of acetic acid. Jan. 4.

472. F. H. Haviland and J. Farmer. Improvements in and relating to the manufacture of calcium carbide. Jan. 7.

### Complete Specifications Accepted.

1896.

29,455. H. H. Lake.—From C. Pieper. Apparatus for continuous manufacture of ammonia from nitrogenous materials. Dec. 22.

1897.

422. A. James. Treatment of seaweed and the like for the production of iodine and other useful products. Jan. 12.

3592. J. E. Campbell. Apparatus for the concentration or rectification of sulphuric acid or other acids or liquors. Dec. 22.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### Applications.

29,837. A. Matthews and W. Matthews. Improvements in the manufacture of chemical and other earthenware. Dec. 16.

30,571. W. P. Kelly. An improvement in or in connection with articles of china, Delph, glass, and such like. Dec. 28.

30,598. W. B. Johnson.—From G. W. Blair and H. I. Hays, United States. Improvements in the method of and apparatus for making hollow glass articles. Complete Specification. Dec. 28.

30,835. P. T. Sievert. An improved mode of and apparatus for producing slabs or sheets of glass with inlaid wire. Dec. 30.

1898.

137. F. J. Shippey and D. Forbes. Improvements in or relating to the decoration of ceramic ware. Jan. 3.

682. E. H. Hilton and J. B. Goodman. Improved method of and means for the separation of china-clay from the other constituents of decomposed felspar. Jan. 10.

### Complete Specifications Accepted.

1897.

9351. H. Emery. Saggars for potters' use. Dec. 29.

22,033. T. Krah. Apparatus for pressing and moulding glass and the like. Dec. 29.

23,933. W. Burton and J. Burton. Construction of potters' kilns. Jan. 19.

26,537. A. Macairo and C. Seuvers. Apparatus for printing on brittle surfaces, such as glass, crystal, porcelain, and the like. Dec. 22.

26,901. A. Houghton. Process of and apparatus for drawing glass tubing. Dec. 22.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

29,825. C. A. Day.—From Mayhew Compo-Board Company, United States. Improvements in and relating to the manufacture of compo-board. Complete Specification. Dec. 16.

30,026. O. Imray.—From The Firm of Grünzweig and Hartmann, Germany. Manufacture of a stone or moulded blocks from kieselguhr or infusorial earth. Dec. 18.

30,214. G. Vogerl. Improvements in the manufacture of artificial stone. Complete Specification. Dec. 21.

30,311. F. H. Willis. Improvements in and connected with the construction of fireproof floors. Complete Specification. Dec. 22.

30,361. H. Boyd and T. A. Bayliss. An improved fireproof, damp-proof, and heat non-conducting composition. Dec. 22.

1898.

17. G. T. Smith and T. W. James. Imitation mica, marble, and stone. Jan. 1.

151. J. West and H. J. W. Raphael. Improvements in asphalte block paving. Jan. 3.

238. S. Taylor and J. M. Frith. Improvements in or relating to lime or cement kilns. Jan. 4.

684. W. A. Oakley. An improved artificial stone, and method of manufacturing same. Complete Specification. Jan. 10.

822. H. H. Lake.—From V. Micheli, Italy. An improved process for the manufacture of a substitute for wood or stone. Complete Specification. Jan. 11.

830. H. H. Lake.—From V. Micheli, Italy. An improved process for the manufacture of a compound for use in making pottery, bricks, and the like. Complete Specification. Jan. 11.

1177. L. P. Ford. Improvements in the method of manufacturing artificial stone and concrete. Jan. 15.

1209. H. Rous. An improved fireproof composition suitable for building purposes. Complete Specification. Jan. 15.

### Complete Specifications Accepted.

1897.

6525. G. Christie. Method of and means for making artificial stone. Jan. 19.

25,861. D. H. Ferguson. Material for the construction and decoration of buildings. Dec. 22.

27,104. H. Eichler. Material for flooring. Jan. 12.

## X.—METALLURGY, MINING, Etc.

### Applications.

29,527. E. E. Atkins and W. J. Poate. Hardening and tempering steel. Dec. 14.

29,572. J. C. Richardson. Improvements in or relating to solvents for metals and the treatment of gold and other ores. Dec. 14.

29,574. W. Wood. Improvements in and relating to the production or formation of hollow metal castings. Dec. 14.

29,577. H. W. Neild. Improvements in joining aluminium and its alloys to steel or iron, and the application of the same to the construction of cycle frames. Dec. 14.

29,591. C. P. Shrewsbury and H. R. Gregory. Improvements in or connected with the extraction of gold from quartz or other materials, and in apparatus to be employed in connection therewith. Dec. 14.

29,611. W. F. Lay. Improvements in or relating to the mining of placer deposits, or similar deposits of a sedimentary nature, and extraction of the gold or other valuable material therefrom. Complete Specification. Dec. 14.

29,721. J. C. Montgomerie. Improved means applicable for use in the extraction of metals from metallic solutions. Dec. 15.

29,751. G. F. Redfern.—From P. Argall, United States. Improvements in roasting and drying ores, and apparatus therefor. Dec. 15.

30,053. J. Hutton. The separation of tungstic acid from wolfram and other minerals containing tin and other metals. Dec. 20.

30,100. H. A. Ellis. An improved process of and apparatus for the centrifugal amalgamation and concentration of precious metals. Dec. 20.

30,297. J. L. Stevenson and J. Evans. Cast-steel water-cooled tuyère and tuyère arch for blast furnaces. Complete Specification. Dec. 22.

30,436. C. L. C. Berton. Process of tinning all metals by means of a cold liquid preparation. Dec. 23. Date applied for July 27, being date of application in France.

30,463. J. T. Wainwright. Improvements in art of manufacturing steel armour plate. Complete Specification. Dec. 23.

30,517. J. L. Greenway. A new or improved process in manufacturing sheet-iron and bar-iron. Dec. 24.

30,934. J. T. Wainwright. Improvements in art of manufacturing steel armour plate. Complete Specification. Dec. 31.

1898.

142. A. Pigott. Improvements in the treatment of lead ores. Complete Specification. Jan. 3.

224. A. J. Boult. — From H. L. Hartenstein and G. A. Weber, United States. Improvements in or relating to the utilisation of waste products from metallurgical furnaces and the like. Complete Specification. Jan. 4.

285. G. de Bechi. Improvements in the treatment of ores containing intimately mixed copper, zinc, and lead. Jan. 5. Date applied for July 5, 1897, being date of application in France. Complete Specification.

347. H. Wethered.—From J. A. Gilmour and H. S. Young, Central America. Improvements in the treatment of ores containing precious metal, for the recovery of the precious metal therefrom. Jan. 5.

484. A. Philip. Improvements in the treatment of ores and other substances containing zinc. Jan. 7.

617. S. Cowper-Coles. Improvements in the extraction of iron from its ores. Jan. 10.

625. E. J. Ball. Improvements in the metallurgical treatment of argentiferous and auriferous ores and substances. Jan. 10.

789. W. Thorp. An improvement in the purification of spiegeleisen and similar alloys. Jan. 11.

937. E. Martin. Manufacture of a compound metal of steel and silver. Jan. 12.

938. E. Martin. Manufacture of a compound metal of brass and silver. Jan. 12.

942. E. J. Ball. Improvements in the metallurgical treatment of metallic ores and other metalliferous substances. Jan. 13.

976. C. E. Greene. Improvements in plates for collecting or separating gold. Complete Specification. Jan. 13.

1027. T. Odlum. Improvements in welding compounds. Jan. 13. Date applied for June 23, 1897, being date of application in United States. Complete Specification.

1106. L. Mond. A process for obtaining metallic nickel from nickel carbonyl, and apparatus therefor. Jan. 14

1136. W. S. Simpson. Improved method of and appliances for hardening the surface of steel armour plates. Jan. 14.

### Complete Specifications Accepted.

1896.

28,836. J. W. Spencer. Manufacture of steel. Dec. 22.

30,152. A. M. Clark.—From R. H. T. Biewend and The Actien Gesellschaft für Zinc Industrie vormals W. Grills. Recovering zinc from zinciferous gases in the distillation and smelting of zinc. Dec. 31.

1897.

1034. H. R. Angel. Reduction of zinc blend and other ores, and recovery of the lead with other metals therefrom. Jan. 12.

1508. R. Hodgson. Apparatus for extracting gold from ores. Dec. 22.

1515. C. I. Thorn. Sheet iron and steel castings close annealing heating furnace. Jan. 19.

2226. T. Doherty and P. D. Crerar. Process of melting iron. Jan. 12.

6051. J. E. Preston. Method and apparatus for treating refractory ores containing gold, silver, nickel, &c. Jan. 12.

7218. B. Becker. Extracting precious metals from minerals containing them, and apparatus therefor. Dec. 22.

8577. B. K. Jamison. Armour plates, and apparatus employed therein. Dec. 29.

8578. B. K. Jamison. Treatment of steel or nickel steel, and of articles made from steel, and apparatus employed therein. Dec. 29.

26,046. S. Ganelin. Treating lead sulphide ores. Dec. 29.

27,546. T. Levoz. Construction of converter apparatus for producing steel and wrought iron of superior quality. Jan. 12.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

29,507. M. Engl. Method of and apparatus for producing perforated accumulator-plates. Complete Specification. Dec. 13.

29,512. J. W. Davies and D. Loch. Improved electric storage batteries. Dec. 11.

29,710. D. Browne and P. Kamperdyk. Improvements in accumulators. Complete Specification. Dec. 15.

30,341. W. Antrobus. Improvements in and relating to electric smelting furnace. Dec. 22.

30,441. C. L. R. E. Menges. Improvements in primary and secondary galvanic batteries. Dec. 23.

30,619. J. W. Bulloch. Improvements in primary galvanic batteries or cells. Dec. 28.

30,686. C. J. Hubbell, H. G. Hubbell, W. De W. Boyer, and E. P. Mucklow. Improvements in and relating to primary batteries. Complete Specification. Dec. 28.

30,744. J. Greenwood. Improvements in coating tubes by electro-deposition. Dec. 29.

30,838. C. Marschner. Improvements in apparatus for use in the manufacture of accumulator-plates. Complete Specification. Dec. 30.

30,841. G. C. Marks.—From H. de Solages, France. Improvements in electrolytic apparatus. Dec. 31.

1898.

16. J. W. Towers. Improvements in the manufacture of diaphragms for electrolysis. Jan. 1.

122. D. F. Sinclair. Improvements for separating table or other salts and generating electricity. Jan. 3.

435. S. W. Maquay. Improvements in and connected with primary batteries. Jan. 6.

678. C. A. Jensen.—From A. Sinding-Larsen, Norway. Method of diminishing the affinity of electrolytically produced chlorine. Jan. 10.

848. J. von der Poppenburg. An improved galvanic battery. Jan. 11.

885. G. Langbein. Apparatus for electro-plating articles in bulk and in large quantities. Complete Specification. Jan. 12.

932. C. Pollak. Improvements in the manufacture or production of electrodes for secondary batteries. Jan. 12.

1191. G. Philippart. Improvements in secondary batteries. Complete Specification. Jan. 15.

1213. Sir C. S. Forbes, Bart. Improved apparatus for the electro decomposition of water. Jan. 15.

Complete Specifications Accepted.

1896.

21,019. L. J. H. Rochatte. Electric batteries. Dec. 29.

25,631. F. Parker. Means employed in the electrolytic production of the chlorates of sodium and potassium. Dec. 22.

1897.

1130. E. J. Wade. Electrical storage batteries. Dec. 22.

2291. M. Engl. Insulating frames for electrodes. Jan. 19.

2710. W. E. Heys.—From E. Dumoulin. Method of and apparatus for the electro-deposition of copper and other metals on rotary mandrels. Dec. 22.

2711. W. E. Heys.—From L. Dumoulin. Apparatus employed in the electro-deposition of copper and other metals. Jan. 19.

2712. W. E. Heys.—From L. Dumoulin. Apparatus for the electro deposition of copper and other metals. Jan. 19.

3054. E. S. New. Separator for the plates of secondary batteries. Jan. 12.

3488. E. Schattner. Secondary batteries. Jan. 12.

4916. E. Heyl. Electric accumulators. Dec. 29.

5805. D. G. FitzGerald. Manufacture of elements for secondary batteries. Jan. 19.

15,903. A. D. Seton and J. L. Dobell. Electric batteries. Jan. 12.

17,081. F. J. Patten. Electrical furnaces. Dec. 22.

25,705. W. W. Hanscom and L. A. Hough. Electrical batteries. Jan. 12.

26,656. P. F. Ribbe. Electric accumulator plates. Jan. 19.

## XII.—FATS, OILS, AND SOAP.

Applications.

29,612. J. Hopkinson, T. Cowling, and F. Illingworth. Improvements in the treatment of wool grease and of other like substances, and of derivatives therefrom. Dec. 14.

29,728. S. Banner. Improved compound applicable for making a substitute for linseed oil body for paint, printing ink, floor-cloth making, and the like. Dec. 15.

30,662. W. A. Smith. An improved process of refining oil. Dec. 28.

30,684. C. G. Hepburn. Improvements in apparatus for treating fats and oils. Complete Specification. Dec. 28.

1898.

198. I. Spencer. Improvements in the method of and means for extracting oil from fish livers or the like, melting tallow or other substances, and for purifying oils, greases, and the like; also applicable for drying purposes. Jan. 4.

710. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in the bleaching, purifying, and refining of vegetable fats and waxes, and apparatus therefor. Jan. 10.

711. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in the bleaching, purifying, refining, and hardening of animal fats and waxes, and apparatus therefor. Jan. 10.

Complete Specifications Accepted.

1897.

4830. F. L. Bartelt. Washing and cleansing compound. Dec. 29.

5820. A. F. Dunn and A. Waithman. Antiseptic soap powder. Jan. 12.

5889. E. G. Scott. Concentration or distillation of glycerin. Jan. 12.

14,248. J. Davidson and C. G. Hepburn. Apparatus for refining fats and oils. Jan. 12.

18,550. A. Euston. Apparatus for treating flax and other oleaginous seeds for the extraction of oil therefrom. Jan. 19.

24,779. G. H. Evans. Combined soap. Jan. 12.

24,868. J. Stockhausen. Manufacture of soap for use in dyeing and finishing textile fabrics and materials, and for other purposes. Dec. 29.

26,191. C. Culmann. Process for deodorising oils and fatty substances. Jan. 12.

28,206. A. Euston. Treatment of oleaginous seeds for the extraction of oil therefrom. Jan. 19.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

Applications.

29,635. S. Hansel and E. Hornung. A waterpoof coating and impregnating substance, and a process for the manufacture of the same. Complete Specification. Dec. 14.

29,867. R. A. Young and A. Tracy. An improved method of drying white lead colours, clays, or the like. Dec. 17.

29,868. E. Springborn. An improvement for oil paint and varnish. Dec. 17.

30,242. A. H. Eyles, H. S. Rapelye, and A. Applegate. Improvements in the manufacture of carbonate or sulphate of lead. Complete Specification. Dec. 21.

30,570. A. J. Loftus. Improvements relating to opaline and like substances. Dec. 28.

30,767. T. Storer. New process for the production of pigment of shades from yellow to red from cobalt ores. Dec. 30.

1898.

110. W. Peel. Anti-corrosive composition or paint as a preventative against corrosion on metals. Jan. 3.

119. Blundell, Spence, and Co., Ltd., and S. Hill. An improved varnish especially applicable for giving a frosted appearance to glass, talc, or the like. Jan. 3.

538. F. T. Addyman and J. A. Gardner. A method for bleaching and purifying gutta-percha. Jan. 8.

664. E. Toussaint. An improved waterproof coating for walls and the like. Jan. 10.

1173. E. Garnier. Improvements in or relating to the manufacture of rubber or other similar gums. Jan. 15.

### COMPLETE SPECIFICATION ACCEPTED.

1897.

2667. A. J. Boult.—From F. Fritsche and Co. Preservative paints, pigments, and the like. Jan. 19.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATION.

30,698. J. A. Bragg. An improved process for removing lime from skins and hides preparatory to tanning. Complete Specification. Dec. 29.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

24,083. A. J. Boult. — From G. Brigalant, France. Manufacture of boards, plates, or sheets from leather pulp. Jan. 12.

25,008. H. Streich. Preparation of leather. Dec. 29.

26,760. H. Holmes. Process of depilating hides or skins. Dec. 22.

## XVI.—SUGARS, STARCHES, GUMS, ETC.

### APPLICATIONS.

29,630. E. W. Gerbracht and F. G. Wiechmann. A process for purifying black-strap or refuse syrups. Complete Specification. Dec. 14.

30,448. A. Verley. Improvements in the manufacture of sugar. Dec. 23.

1898.

1131. J. Y. Johnson.—From A. Verley, France. Improvements in the manufacture of sugar. Jan. 14.

### COMPLETE SPECIFICATION ACCEPTED.

1897.

3900. A. Wieckmann. *See* Class XVIII.

## XVII.—BREWING, WINES, SPIRITS, ETC.

### APPLICATIONS.

29,930. M. Preu and G. Himmel. An improved saccharometer or device for testing beer worts. Complete Specification. Dec. 17.

30,208. V. Lapp. A process for the manufacture of non-alcoholic beer. Complete Specification. Dec. 21.

30,252. B. J. Atterbury and The Alcohol Syndicate, Ltd. Improvements in the production of alcohols and analogous products, and in apparatus for use therewith. Dec. 21.

30,658. R. M. de Tangry. An improved process and apparatus for accelerating the ageing of alcoholic liquors. Complete Specification. Dec. 28.

30,681. C. C. Doig. A process and apparatus for evaporating the burnt ale, spent lees, and residual products of distilleries, and for utilising the steam thereby engendered for motive power. Dec. 28.

1898.

162. C. C. Doig. A process and apparatus for evaporating the burnt ale, spent lees, and residual products of distilleries, and for utilising the steam thereby engendered for motive power. Jan. 3.

513. R. C. Scott. Improvements in treating or ageing spirit. Jan. 7.

1208. B. J. Atterbury, T. Macalpine, and The Alcohol Syndicate, Ltd. Improvements in and connected with the production of alcohols and other products used in such production, and in apparatus for use therewith. Jan. 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

3900. A. Wieckmann. Utilising the solid refuse of breweries, distilleries, starch, sugar, poudrette factories, and the like. Jan. 19.

4441. A. J. Boult.—From K. E. N. Fryklind. Treatment of spirituous liquors and the products obtained therefrom. Dec. 29.

15,825. G. Putz. Clarifying or fining worts, and apparatus therefor. Jan. 12.

## XVIII.—FOODS, SANITATION, ETC., AND DISINFECTANTS.

### APPLICATIONS.

*A.—Foods.*

30,081. G. Bamberg. Improvements in the manufacture of tablets of cocoa, tea, and coffee. Dec. 20.

30,500 W. J. McConnell and W. A. B. Mackinlay. Improvements in method of preserving foods. Dec. 24.

30,842. C. Kobbe. A process for the manufacture of alimentary products containing vegetable albumen. Dec. 30.

30,877. A. Backhaus. Improved method for obtaining from milk water-soluble caseïne preparations and compound caseïne albumen preparations. Dec. 31.

30,919. J. L. Grant. A new or improved article of food. Dec. 31.

1898.

62. J. F. Duke. An improved mode of obtaining infusions or extracts of coffee or other substances. Jan. 1.

559. A. D. Watson. Improvements in the manufacture of a nutritious extract of yeast. Jan. 8.

1186. T. S. Mason. Improvements relating to the preservation of sterilised milk. Jan. 15.

*B.—Sanitation.*

30,629. J. Grossmann. Improved method of treating sewage. Dec. 28.

1898.

149. F. Eichen. Improvements in or relating to apparatus for treating and purifying sewage waste and similar waters. Jan. 3.

*C.—Disinfectants.*

30,036. A. Guasco. Improved chemical product for antiseptic purposes, and burner or device for utilising the same. Dec. 18.

### COMPLETE SPECIFICATIONS ACCEPTED.

*A.—Foods.*

1897.

3810. H. Schowell. Preparation of lupines for yeast manufacture, cattle food, and similar purposes. Jan. 19.

26,658. F. H. M. Röver. Preservation of eggs. Dec. 22.

28,359. M. Delavigne, C. Helmerich, and A. Klüglein. Apparatus for the preservation of eggs. Jan. 19.

*B.—Sanitation.*

1896.

29,223. J. S. McDougall. Methods of and apparatus for water purification. Dec. 22.

1897.

654. W. M. Ducat. Method of and apparatus for purifying water, sewage, and other foul liquids. Jan. 19.

1722. P. R. Candy. Apparatus for use in the purification of sewage and polluted waters. Jan. 12.

2228. F. W. Strong. Tanks, and apparatus for use in connection with the same, for the purification and treatment of sewage and impure waters. Jan. 19.

4406. T. Cosham. Sewage precipitating tanks. Dec. 29.

27,004. J. G. Dornig, F. H. Rachlitz, and T. Zeunert. Means for the sanitary disposal of human excrement, and the production therefrom of gas and by-products serving for manure and other purposes. Jan 19.

### *C.—Disinfectants.*

1897.

4115. R. Stewart. Manufacture of detergent fluids. Dec. 22.

5820. A. F. Dunn and A. Waithman. *See* Class XII.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

30,337. T. Coon and Mardon, Son, and Hall, Ltd. Imitation leather, cloth, parchment, and the like. Dec. 22.

30,570. A. J. Loftus. *See* Class XIII.

30,579. S. Jaques. Improvements in the manufacture of paper. Dec. 28.

1898.

4. W. W. Beaumont. Improvements in apparatus for straining paper pulp. Jan. 1.

972. J. Tait. Improvements in bleaching and purifying paper pulp. Jan. 13.

### Complete Specification Accepted.

1896.

29,829. C. D. Abel.—From The Farbwerke Friedrichsfeld P. Remy. Waterproof paper and similar fabrics, and production of compounds therefor. Dec. 29.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

29,451. E. H. Taylor. An improved system, and apparatus in connection therewith, for the emulsification of essential and other oils and water, and for producing extracts or tinctures from other substances with water for use in the manufacture of flavoured mineral waters, and for other purposes. Dec. 13.

1898.

928. H. E. Aspinall. Improvements in the manufacture of artificial camphor and in apparatus therefor. Jan. 12.

### Complete Specifications Accepted.

1897.

6475. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Manufacture of acetylanthranilic acid. Jan. 19.

6686. L. H. C. Darkopf. Manufacture of methylene-ditannic acids from formaldehyde and tannin. Dec. 22.

## XXI.—PHOTOGRAPHY.

### Applications.

29,832. A. Weiss. Method of and apparatus for igniting flash powder in taking photographs by artificial light. Complete Specification. Dec. 16.

30,115. A. L. Feeny.—From F. von Bentivegni, United States. Improved process of applying colours to photographs. Complete Specification. Dec. 20.

30,526. La Société Th. Dupuy et Fils. A novel manufacture of paper for photographic purposes. Dec. 21. Date applied for July 31, 1897, being date of application in France.

1898.

838. R. Haddan.—From L. E. Dugardin, France. Improvements in or relating to the process of photography in colours. Jan. 11.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

30,056. F. Hutchins and L. Greening. Improvements in solvents for pyroxylin. Dec. 20.

30,179. L. Davies. Improvements in explosive cartridges. Dec. 21.

30,253. H. H. Lake.—From G. Cornara, Italy. An improved electro-chemical explosive device. Dec. 21.

30,442. H. Boyd. Improvements in explosives. Dec. 23.

30,516. C. Kastner and A. Swoboda. Improvements relating to the manufacture of matches. Dec. 24.

1898.

127. J. C. Mewburn.—From The Chemische Fabrik von Heyden Gesellschaft mit beschrankter Haftung, Germany. The manufacture of perchlorates and the employment of the same in the manufacture of fireworks. Complete Specification. Jan. 3.

228. W. Salter. Improvements in matches and flaming fusees. Complete Specification. Jan. 4.

447. S. J. Nicholson. Improvements in the manufacture of nitro-cellulose. Jan. 6.

924. R. Sohlman. Improvements in explosive compounds. Jan. 12.

1007. W. E. Gordon. Improvements in lucifer matches. Complete Specification. Jan. 13.

### Complete Specifications Accepted.

1896.

28,220. G. Beneke. Manufacture of explosives. Jan. 19.

1897.

2785. A. J. Fredrikson. Manufacture of matches. Dec. 29.

4062. S. A. Rosenthal.—From J. S. von Romocki. Manufacture of safety explosives. Jan. 19.

24,195. L. Braly. Compositions suitable for the manufacture of matches. Jan. 12.

27,520. J. Craveri. Manufacture of safety matches and rubbing surfaces therefor. Jan. 12.

## PATENTS UNCLASSIFIABLE.

### Applications.

29,804. G. H. Jackson. Improvements in the manufacture of hardened asbestos. Dec. 16.

30,353. C. Maiche. A new oxidising product. Dec. 22.

### Complete Specifications Accepted.

1897.

24,085. G. Barker.—From L. Rissmuller, Germany. Treatment of waste matters. Jan. 12.

27,661. M. Weishaus. Regenerating used whalebone and dressing fin-whalebone without rendering the same brittle. Dec. 29.

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Monday, March 14th, 1898:—R. W. Wallace, Esq., Q.C. "Electrical Industries at the Falls of Foyers."
Monday, April 4th, 1898:—Election of Officers and Five Members of Committee. Nominations to be sent in not later than March 21st.

## Manchester Section.

*Chairman:* George E. Davis.
*Vice-Chairman:* D. Watson.
*Committee:*

B. W. Gerland.
J. Grossmann.
J. M. Irving.
E. Knecht.
M. J. Langdon.
I. Levinstein.
J. Lewkowitsch.
A. Liebmann.
A. Ree.
E. Schunck.
C. Truby.
W. Thomson.

*Hon. Local Secretary:*
J. Carter Bell, The Cliff, Higher Broughton, Manchester.

March 4th, 1898:—
Discussion on Mr. Carter Bell's paper (this Journal, 1898, 11).
Mr. R. J. Flintoff. "Metallic Tannate Lakes of Basic Colouring Matters on Cotton Cloth."
Mr. J. Carter Bell. "Comparative Effect of Lime and other Chemicals on Sewage."
April 8th.—Election of Officers and five Members of Committee. Latest Day for Nominations, March 21st.

## Newcastle Section.

*Chairman:* P. S. Newall.
*Vice-Chairman:* G. T. France.
*Committee:*

A. Allhusen.
P. P. Bedson.
T. W. Hogg.
H. Louis.
T. W. Lovibond.
N. H. Martin.
John Morrison.
John Pattinson.
W. W. Proctor.
W. L. Rennoldson.
G. Sisson.
J. E. Stead.

*Hon. Local Secretary and Treasurer:*
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

March 17th, 1898.—Mr. Jno. Pattinson, F.I.C., and Dr. H. Salvin Pattinson, F.I.C.:—
(1) "On the Separation and Estimation of Arsenic."
(2) "On the Estimation of Tin in Metallic Alloys."

## New York Section.

*Chairman:* Charles F. Chandler.
*Vice-Chairman:* T. J. Parker.
*Committee:*

H. Clementson.
Virgil Coblentz.
H. Endemann.
W. F. Fuerst.
Jas. Hartford.
W. D. Horne.
E. J. Lederle.
G. A. Prochazka.
Clifford Richardson.
Wm. Jay Schieffelin.
R. C. Schüpphaus.
G. W. Thompson.
Max. Toch.

*Hon. Treasurer:* R. C. Woodcock.
*Hon. Local Secretary:*
H. Schweitzer, 77, William Street, New York, U.S.A.

## Nottingham Section.

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*Vice-Chairman:* J. M. C. Paton.
*Committee:*

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S. F. Burford.
F. J. R. Carulla.
R. M. Caven.
F. Clowes.
H. Forth.
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J. F. Kempson.
F. E. Lott.
J. J. Sudborough.
G. J. Ward.

*Treasurer:* S. J. Pentecost.
*Hon. Local Secretary:*
J. T. Wood, 29, Musters Road, West Bridgeford, Nottingham.

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R. Cox.
C. A. Fawsitt.
J. Gray.
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R. A. Inglis.
Robt. Irvine.
A. Macdonald.
R. C. Menzies.
Sir R. Pullar.
J. B. Readman.
H. Rose.
P. Rottenburg.
E. C. C. Stanford.
R. T. Thomson.

*Hon. Secretary and Treasurer:*
Thomas Gray, Technical College, 204, George Street, Glasgow.

Tuesday, March 1st, 1898:—
Dr. Wm. Frew. "The Education and Training of Brewers."
Mr. A. Forbes Watson, B.Sc. "A Process for the Recovery of Beer from 'Bottoms' or 'Grounds.'"

## Yorkshire Section.

*Chairman:* Thos. Fairley.
*Vice-Chairman:* Christopher Rawson.
*Committee:*

H. E. Aykroyd.
F. W. Branson.
J. Cohen.
N. Farrant.
T. Glendinning.
A. Hess.
J. J. Hummel.
W. McD. Mackey.
H. R. Procter.
F. W. Richardson.
A. Smithells.
Geo. Ward.

*Hon. Local Secretary and Treasurer:*
H. R. Procter (*pro tem.*), The Yorkshire College, Leeds

# NOTICES.

## Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List, Each copy 10*s*.
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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,000 per month.

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Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws, the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

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## Telegrams.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

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## LIST OF MEMBERS ELECTED 23rd FEBRUARY 1898.

Allden, Jno., Chemical Laboratory, Pacific Mills, Lawrence, Mass., U.S.A., Chemist.

Barnicott, Jas. A., Messrs. May and Baker, Ltd., Garden Wharf, Battersea, S.W., Chemical Manufacturer.

Baxter, Jno. G., 75, Manchester Road, North Greenwich, E., Chemist.

Clarkson, Thos., Usine du Meglin, Buxière-les-Mines, Allier, France, Chemist.

Crompton, Benj. F., Church Street, Adlington, Lancashire, Printworks Manager.

Curtis, Marvin, 123, California Street, San Francisco, Cal., U.S.A., Chemist.

Davies, Herbert E., The Laboratory, 28, Chapel Street, Liverpool, Analytical Chemist.

Davies, Leyshon, Kames, Kyles of Bute, by Greenock, N.B., Gunpowder Mills Manager.

Deming, Edw. D., 143, Federal Street, Boston, Mass., U.S.A., Editor of "The Leather Manufacturer."

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Golding, Jno., University College, Nottingham, Agricultural Chemist.

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Heasman, Walter, Castle Brewery, Bridgnorth, Salop, Brewer.

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Obach, Dr. Eugene F. A., c/o Siemens Bros. and Co., Woolwich, S.E., Chemist and Electrician.

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Walker, Wm. E., Rose Hill Tannery, Bolton, Tanner.
Wheeler, Edw. J., 79, Chapel Street, Albany, N.Y., U.S.A., Analytical Chemist.
White, Jno., County Offices, St. Mary's Gate, Derby, Public Analyst to County of Derby.

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Lee, T. H., l/o London, (subsn.) Edgecumbe Villa, Clevedon, Somerset; (Journals) St. John Del Rey Mine, Villa Nova de Lima, Brazil.
Lett, Stephen. J., l/o Liverpool; c/o North Charterland Exploration Co., Ltd., Kota Kota, *viâ* Chinde, British Central Africa.
Lymn, Arthur H., l/o Jena; Plattenstrasse 50, Fluntern, Zürich, Switzerland.
McVitie, Robt.; Journals to 12, Greenhill Gardens, Edinburgh.
Mann, E. A.; Journals to Government Chemical Laboratory, Fremantle, West Australia.
Mann, H. H., l/o Hammersmith; Crawley Mill Farm, *viâ* Bletchley, Research Chemist.
Mann, Jno. C., l/o Plaistow; c/o Major and Co., Ltd., Sculcoates, Hull.
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Nelson, H. W.; Journals to 15, Hamilton Chambers, 17, St. John Street, Montreal, Canada.
Picard, Hugh K., l/o Deloro; retain Journals until further notice.
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Wagner, W. G.; Journals (temp.) to Cheriton House, Coldstream Street, Llanelly, South Wales.
Watson, Jas., l/o 48; 3, West Park Terrace, South Shields.
Watson, Jno., l/o Krugersdorp; Jumpers Deep Gold Mining Co., Ltd., per private bag, Cleveland Station, near Johannesburg, S.A.R.

### CHANGE OF ADDRESS REQUIRED.

Larned, J. N., l/o Public Library, Buffalo, N.Y., U.S.A.

## Deaths.

Ashworth, L., The Orchard, Bury, Lancashire.
Tonks, Edmund, Packwood, Knowle, near Birmingham.

## Liverpool Section.

*Meeting held on Wednesday, January 26th, 1898.*

DR. C. A. KOHN IN THE CHAIR.

### SOME FURTHER EXPERIMENTS RELATING TO THE MANUFACTURE OF CYANIDES.

BY JAMES T. CONROY, B.SC., PH.D.

Two years ago it was my privilege to read before the members of this Section an account of some experiments made with the view of comparing the values of the various processes for the production of cyanides and ferrocyanides. I arranged the methods then discussed in two main divisions:—

(*a*.) Methods for the direct production of cyanides, using as the source of nitrogen—

(1) the atmosphere;
(2) ammonia.

(*b*.) Methods in which sulphocyanides are prepared as intermediate products.

This classification I found to agree with that adopted by Caro, an abstract of which appeared in the same issue of the Society's Journal as my paper, and, basing his conclusions on such a division, he had come to regard the sulphocyanides as offering the most likely basis for a successful process.

This is also my view, and to-night I propose to give an account of a new process, starting from the sulphocyanides as raw material.

These sulphocyanides, it will be remembered, result almost quantitatively when ammonia and carbon bisulphide are heated, preferably in the presence of some base such as lime or soda, to 100° in an autoclave.

Their conversion into cyanides is brought about in one of two ways :—

(1.) The sulphur atom may be removed by oxidation, the S being converted to sulphuric acid.

(2.) The sulphur atom may be removed by reduction in the form of a metallic sulphide.

Of the reduction methods, I described two in detail. In the first, the potassium sulphocyanide is heated with zinc (Playfair's method), forming potassium cyanide and zinc sulphide; in the second, iron is the metal employed. In the latter case, on lixiviating the melt, potassium ferrocyanide, of course, results. Both these methods, it will be noticed, involve fusion. Special attention was drawn to this point by Mr. C. A. Fawsitt when Dr. Playfair described his process to the Scottish Section of this Society.

Mr. Fawsitt said (this Journal, 1892, 16): "Instead of converting sulphocyanides into cyanides by fusion, it seemed to him far more rational to try and get an economical method whereby it could be done in solution. It was well known that in acid solutions sulphocyanides could be converted into cyanides by using permanganate of potash . . . but the cost was prohibitive, and the fact of its taking place in an acid solution was inconvenient, as the hydrocyanic acid would require to be condensed, besides the unpleasantness of working an apparatus containing such a poisonous gas."

This paragraph might almost appear to have been the source of inspiration to several recent patentees.

In the first place a very ingenious modification of the oxidation process outlined above has been patented by Raschen (Eng. Pat. 21,678, 1895), in which the oxidising agent employed is nitric acid. This is reduced to nitric oxide (NO), which is evolved with the gaseous hydrocyanic acid. This latter gas is absorbed by alkali to form an alkaline cyanide, and the unabsorbed nitric oxide, after admixture with air, is recovered as nitric acid to enter again into the process.

A second mode of decomposing sulphocyanides in solution is described in the English Patents—

Bower, 361 of 1896: "Improvements in methods of separating the cyanogen compounds from gas liquor or other solutions containing cyanogen compounds."

Conroy and Hurter, 3869 of 1896: "Improvements in the manufacture of ferrocyanides from sulphocyanides."

In both these processes the sulphocyanide is converted into a cyanide of iron, from which, on treatment with alkali, an alkaline ferrocyanide is obtained. The essential part of each is the abstraction of sulphur from sulphocyanide of iron by heating a solution of this under pressure with a metal such as copper or iron.

A study of the history of the Bower process, taken from the patents 8330 and 8381 of 1895, and 361 of 1896, shows that the method originated in an attempt to recover separately the ferrocyanogen and sulphocyanogen compounds present in gas liquor after a certain treatment originally described by Pitt in patent 2918 of 1882.

This treatment consists in adding a sufficient quantity of metallic iron and iron salt to the cold ammoniacal liquor to convert all hydrocyanic acid compounds and all sulphocyanic acid compounds into ferrocyanide and sulphocyanide of iron respectively. Excess of lime is then added and the ammonia recovered by distillation in the usual way. The residual liquor, holding in solution the ferrocyanide and sulphocyanide as calcium salts, is treated with an acidified solution of cuprous chloride, whereby all the cyanogen compounds are precipitated as copper salts.

The precipitate is separated and washed, and treated, while still wet, with finely divided iron to decompose the copper salts into ferrocyanide of iron (which is insoluble), sulphocyanide of iron (which remains in solution), and metallic copper. The filtered ferrocyanide of iron is treated with alkali to give an alkaline ferrocyanide, and the sulphocyanide of iron is obtained in the solid condition by evaporation of its solution.

This solid iron sulphocyanide Bower originally utilised for the manufacture of prussiate, by adding it, in small quantities at a time and only towards the end of the operation, to the melt obtained by fusing alkaline carbonates with iron and animal matter, as in the original prussiate process.

It is only in Bower's last patent that the conversion of the sulphocyanide in solution into ferrocyanide is announced. In this patent he states that if the decomposition of sulphocyanide of copper by metallic iron be carried out at an elevated temperature and under pressure (70 lb. per sq. in.) in a closed vessel, the copper first deposited combines with the sulphur of the ferrous sulphocyanide to produce copper sulphide and ferrocyanide of iron. The importance of this discovery was recognised by Bower, who now made his process applicable to sulpho-cyanides derived from any source. His process, it will be noticed, depends upon the presence of metallic copper which, during the reaction, is converted into sulphide.

Having had occasion to try the above process in the laboratory, and finding it to be particularly good, the idea presented itself that possibly the reaction could be carried on with a cheaper metal than copper, and that by this means a more economical process could be devised. On trial it was found that the copper might advantageously be replaced by iron, which, as will appear, in addition to being cheap, also appears to bring about a more rapid decomposition.

Before describing these, the earliest experiments, it will probably lead to a clearer understanding of what follows if I sketch in outline the course pursued in my work, since the steps taken in this course mark the divisions into which a description of the experiments naturally fall.

The first experiments were made with copper and iron on pure solutions of sulphocyanide of iron. These being satisfactory, the next point was to settle whether or not a pure iron sulphocyanide was necessary; in other words, whether the presence of salts such as potassium or calcium chloride interfered to any marked extent with the reaction. No prejudicial effect being traceable to these salts, experiments were made on a large laboratory scale with what we considered the most suitable material for a manufacturing process. These experiments gave us an opportunity of studying the behaviour of the reaction product when worked up for the production of alkaline ferrocyanides. Here a surprise was in store. This was the fixation of a large quantity of the alkali used for decomposing the cyanide of iron. This loss was so great as to prohibit the use of caustic potash for extraction; the only alkali which directly yielded prussiate in its market form could not be employed. Recourse was therefore had to the cheaper caustic soda. The behaviour of the not very well known product, sodium ferrocyanide, was compared with the statements to be found about it in the various works of reference. These statements were found to be very meagre and somewhat inaccurate. I give some figures which I hope may help to correct one mistake, and other figures which are new, and I hope not without some value. As was natural, attempts were made to convert the sodium into the potassium salt—with what success will be shown later.

The decomposition of iron sulphocyanide is represented by the equation $Fe(CNS)_2 + 2R'' = Fe(CN)_2 + 2R''S$.

I stated in my previous paper (this Journal, 1896–8) that what at first sight would appear to be the equivalent of this reaction, namely, the decomposition of potassium sulphocyanide in hot solution by metallic iron, did not take place. This statement I have since found requires some qualification. Reading in a paper by Sternberg that such a decomposition of potassium sulphocyanide could be brought about, I determined to repeat the experiment, substituting, however, for the iron wire and iron reduced from blue billy ($Fe_2O_3$) by strongly heating with pitch, and which contained a large percentage of carbon, a very finely divided and pure iron (over 99 per cent. Fe) reduced from the oxide by coal gas. This new experiment showed that the reaction certainly took place. The decomposition, however, was slight, amounting to only 5·3 per cent. after seven hours' heating at 140°—160 C. If ferrous sulphocyanide is employed under these conditions, as will be proved later, an almost quantitative decomposition is readily obtained.

This difference of behaviour is in accordance with the generally greater stability of potassium salts as compared with iron salts. Some idea of the difference in the present case may be acquired by looking at the thermo-chemical equations (based on somewhat meagre data) for the two decompositions. These indicate a five times greater heat development for the iron than for the potassium salt.

$$2\mathrm{KCyS} + 2\mathrm{Fe} = 2\mathrm{KCy} + 2\mathrm{FeS}$$
$$2(81{\cdot}7) = 2(64{\cdot}7) + 2(23{\cdot}8) + [13{\cdot}6]$$
$$2\mathrm{KCyS} + 2\mathrm{Fe} + \mathrm{FeCl_2} = 2\mathrm{KCl} + \mathrm{FeCy_2} + 2\mathrm{FeS}$$
$$2(81{\cdot}7) + 100 = 2(100{\cdot}8) + 80 + 2(23{\cdot}8) + [65{\cdot}8]$$

These values are for solutions. With zinc the reaction is:—

$$2\mathrm{KCyS} + 2\mathrm{Zn} = 2\mathrm{KCy} + 2\mathrm{ZnS}$$
$$2(81{\cdot}7) = 2(61{\cdot}7) + 2(43) + [52{\cdot}0]$$

What may further aid the more ready decomposition in the case of the iron compound is that in this compound $\left[\mathrm{Fe}<\begin{matrix}\mathrm{S—CN}\\ \mathrm{S—CN}\end{matrix}\right]$ the two atoms (Fe and S) which go to form one of the reaction products, and that produce a stable and insoluble substance, are already united in the sulphocyanide molecule. The reaction may take place through the elimination of FeS and the formation of cyanogen sulphide, which at the moment of such formation is acted upon by the metallic iron present, to produce iron sulphide and iron cyanide.

A process which probably involves the reaction taking place through the formation and decomposition of iron sulphocyanide is that of Sternberg, patented in Germany in 1882. According to this process the sulphocyanide is decomposed by heating its solution with metallic iron and freshly precipitated ferrous hydrate at 110°–120 C. under pressure. The reaction proceeds more rapidly the greater the concentration; a decomposition of 80 per cent. is said to have been attained in 12 hours.

I now come to describe my own experiments. As already indicated, the first of these were made on such quantities of materials as could be heated in sealed glass tubes of ordinary size. These experiments fall under the two headings:—

I. Experiments made with pure solutions of iron sulphocyanide.

II. Experiments made with a soluble sulphocyanide, other than the iron compound, in the presence of iron salts.

I. The solution of ferrous sulphocyanide employed was prepared by Bowle's method, precipitating cuprous sulphocyanide from a pure solution of the potassium salt, and, after well washing the precipitate, boiling it with water and metallic iron, and filtering from excess of this metal and the precipitated copper. The colourless solution so obtained becomes red, owing to oxidation, when exposed to the air; at the same time ferrous hydrate is deposited. The sulphocyanic acid suffers no decomposition during this charge. The solution appears to be stable at temperatures above 100° C.; a portion heated alone for several hours at 150° C. suffered no decomposition.

On boiling with iron or copper at the ordinary pressure, no trace of ferrocyanide results. So soon, however, as the heating with iron or copper is performed under pressure and at a temperature higher than the ordinary boiling point, decomposition occurs.

The amount of this decomposition is shown in the following table:—

TABLE 1.

| — | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. |
|---|---|---|---|---|---|---|---|---|
| Metal | Fe | Fe | Fe | Fe | Cu | Cu | Cu | Fe & Cu |
| Equivalent metal per equivalent RCNS | 7 | 7 | 7 | 7 | 7 | 6 | 7 | 3 |
| Time of heating, in hours | 3½ | 4 | 2½ | 3 | 3 | 3 | 3 | 2¼ |
| Temperature, ° C. | 115—125 | 150 165 | 190—200 | 130—160 | 130 160 | 140—160 | 140—160 | 170 |
| $\mathrm{Fe(CNS)_2}$ undecomposed | 62·0 | 10·6 | 9·2 | 2·75 | 46·8 | 69·5 | 80·0 | 23·3 |
| „ as ferrocyanide | 36·8 | 88·0 | 89·5 | 90·50 | 35·7 | 30·5 | 20·0 | 68·2 |
| „ lost (by difference) | 1·2 | 1·4 | 1·3 | 6·75 | 17·5 | 0 0 | 0·0 | 8·5 |

*Notes on Table 1.*—The experiments between double lines (4 and 5) (6 and 7) were done concurrently in the one bath.

The iron used was in all cases the finely divided iron of the British Pharmacopœia. It contained only some 60 per cent. real Fe, and the amount of metal used is calculated on this percentage.

(5.) The copper used was precipitated from $\mathrm{CuSO_4}$ by same quantity of iron as used in 1.

(6.) „ „ „ „ by zinc, and freed from this metal by hydrochloric acid and washing.

(7.) „ „ reduced from CuO by coal gas at a red heat.

(8.) „ and iron used was obtained by treating cuprous sulphocyanide with metallic iron (Bower's patent).

25 c.c. of ferrous sulphocyanide solution were taken in each experiment: the quantity of sulphocyanide contained was equivalent to from 0·8 to 1·5 grms. of KCNS.

In most cases a slight pressure, due to evolved gases, was found to exist in the cold tubes. The gases evolved, as found from later experiments on a larger scale, were combustible, and probably consisted of hydrogen and hydrocarbons, since water and $\mathrm{CO_2}$ (previously absent) resulted when the gases were burnt. No sulphurous acid was found in the combustion products.

After heating as described, the contents of each tube were washed on to a filter: the undecomposed sulphocyanide was estimated in the filtrate by titration with silver nitrate solution. The insoluble portion, containing the converted sulphocyanide, was decomposed by warming with caustic soda. The alkaline ferrocyanide in the solution so formed was estimated by precipitating as Prussian blue, and after redissolving the washed precipitate in alkali and acidifying, titrating with potassium permanganate. The precipitation as Prussian blue was necessary to obtain the ferrocyanide free from traces of sulphocyanide, which, when present, causes a much too high result to be obtained. [1 mol. KCNS (97 parts) requires 3O for oxidation; 3O will oxidise 6 mols. $\mathrm{K_4FeCy_6}$ (2,208 parts).]

Table I., given above, shows that in most cases the sulphocyanide decomposed was converted almost quantitatively to ferrocyanide.

Copper, though present in equivalent amount, and the decomposition carried out under similar conditions of temperature as in those experiments in which iron was employed, failed in all cases to act so quickly as this latter metal. A difference also seems to exist between the decomposition occurring with copper prepared in different ways; the more rapid action of the precipitated copper as compared with the reduced metal was probably due to its finer state of division; the copper reduced by coal gas was decidedly gritty.

Some experiments were also made with zinc dust. With this metal considerable decomposition took place at 100° C. Considerable pressure resulted; the gases evolved possessed a very disagreeable odour, probably attributable to the presence of organic amines.

### II.—*Experiments with Alkaline Sulphocyanide in the presence of Iron Salts.*

The decomposition of the sulphocyanide of iron by means of metallic iron, recorded in the last table, being so satisfactory, experiments were next tried to find out how far the decomposition would go when, in place of a pure solution of ferrous or ferric sulphocyanide, such an one as results on mixing, say, sulphocyanide of potassium with a soluble salt of iron was employed.

That in this case equally good results are obtainable is shown in Table II.

Table II.

| — | 1. | 2. | 3. | 4. | 5. |
|---|---|---|---|---|---|
| Equivalent of metal as metal | 2½ | 2¼ | 3 | 4 | .. |
| " " salt | 1·05 | 1·1 | 1 | 1·2 | .. |
| Time of heating, in hours | 2 | 2 | 3½ | 5½ | 16 |
| Temperature, °C. | 160 | 160 | 150—170 | 140—160 | 130—140 |
| RCNS undecomposed | 38·0 | 22·0 | 13·0 | .. | .. |
| " converted to ferrocyanide | 52·6 | 67·5 | 80·5 | 95·0 | 94·5 |
| " lost (by difference) | 9·4 | 10·5 | 6·5 | 5·0 | 5·5 |

(1.) 2·3 grms. of pure KCNS in 25 c.c. of water + 3·5 grms. of $FeSO_4 7H_2O$ + 5·0 grms. of B.P. iron } in glass tubes.
(2.) 2·0 " $Ca(CNS)_2$ 16 " + 4·0 " " " 5·0 " " }
(3.) 9·33 " " 175 " 17 grms. of solid $FeCl_3$ (= 3·3 grms. of Fe) + 16 grms. of B.P. iron } in rotating iron
(4.) 9·33 " " 175 " 20 " " (= 3·9 " ) + 20 " " } cylinder.

The conversion of the sulphocyanide into ferrocyanide was now tried on a somewhat larger scale, and on lines more nearly approaching to manufacturing conditions.

These larger experiments were for the most part carried out jointly by Dr. Hawliczek and myself, and I have also to thank Dr. Clayton for timely and valuable aid in the analyses of some of the products obtained at this stage.

It was decided to use in these experiments the sulphocyanide in the form of its calcium salt, and to add the iron salt, necessary for the conversion of this to ferrous sulphocyanide, in the form of ferrous chloride. Working in this manner, we were able to employ the sulphocyanide solution most commonly, and directly, produced on the manufacturing scale, and also to avoid contaminating the reaction product with any foreign precipitate such as calcium sulphate.

The calcium sulphocyanide employed was prepared in the manner described in my previous paper, and was so far concentrated as to contain about 400 grms. of $Ca(CNS)_2$ per litre.

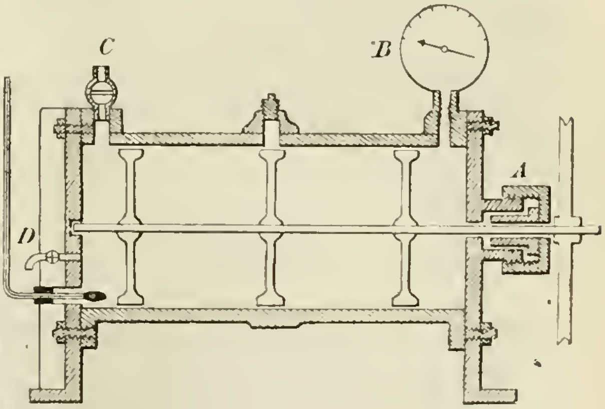
The ferrous chloride solution was made by dissolving iron borings in crude hydrochloric acid. On the large scale the borings would be replaced by the ferrous sulphide formed during the reaction; or spent galvanising liquors, after neutralising the free acid, might be employed. The solution was used nearly saturated, and contained $FeCl_2$ equivalent to about 250 grms. of Fe per litre.

The metallic iron was in the form either of borings (of which, owing to the reaction proceeding only at the surface of the metal, a considerable excess had to be present in each operation), or in the form of a finely divided spongy iron prepared by reducing the iron oxide (blue billy) with coal gas.

The mixed solutions, which occupied about 1 litre, and contained sulphocyanide equivalent to 200 grms. of KCNS, were heated with the metallic iron in an autoclave of special construction.

The autoclave was in the form of a horizontal cast-iron cylinder, in which revolved a shaft carrying a number of paddles as shown. These paddles reached nearly to the circumference of the cylinder, and served to keep the liquid and the iron in constant agitation. The shaft worked through a stuffing box shown at A. The vessel was fitted with a pressure gauge B, a charging hole C, a tap D (above the level of the precipitate) for withdrawing samples during working, and also a discharging hole fixed in the end and at the lowest point of the vessel. A further tap on the top served to release the pressure when this rose too high; it took the place of a safety valve. A thermometer was inserted, as shown, during the operation.

The reaction was in nearly all cases continued until only a small percentage of sulphocyanide remained undecomposed, the paddles meanwhile revolving about 40 revolutions per minute. The temperature was maintained at from 135°—140° C. (15 lb.), and the pressure in the apparatus generally rose to 50—60 lb. On some occasions it showed a tendency to rise above this figure; it was then reduced by opening the valve.



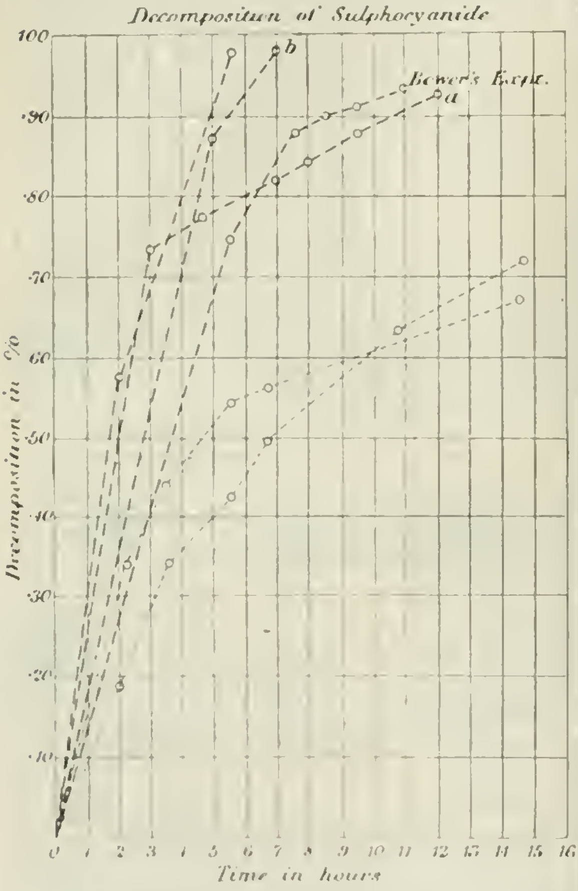
The chief aim of these experiments, as already stated, was to find out, in addition to the yield obtainable, the time necessary for complete conversion of the sulphocyanide to ferrocyanide, together with the rate at which this conversion took place; and further, the behaviour of the decomposition product when worked up for alkaline ferrocyanide.

The time required for decomposition varied considerably according to the quantity and nature of the iron used for reduction. As was to be expected, the more finely divided the iron, the more rapid the reaction. This is shown below:—

| — | Form of Iron. | Times Theoretical Quantity used. | Percentage Sulphocyanide decomposed. | Time required. |
|---|---|---|---|---|
| | | | | Hours. |
| *a* | Borings | 8·00 | 92·5 | 12 |
| *b* | Reduced Iron | 1·55 | 97·5 | 7 |
| *c* | " | 1·55 | 94·0 | 5½ |
| *d* | " | 1·70 | 99·0 | 5½ |
| *e* | " | 1·70 | 85·5 | 5½ |
| *f* | " | 1·70 | 99·5 | 5½ |
| *g* | " | 1·70 | 98·0 | 5½ |

In an experiment with cuprous sulphocyanide and seven times the theoretical amount of iron as borings, a decomposition of 90·6 per cent. was reached in 9½ hours. The reaction sets in fairly rapidly at the start but the rate diminishes very quickly as the amount of sulphocyanide in solution decreases and the iron becomes converted to sulphide.

This is clearly seen in the accompanying curves plotted from values obtained in some of the experiments. The percentage of decomposition reached after any time was deduced by determining the ratio between the chloride and sulphocyanide remaining in solution, and comparing the value so obtained with that existing at the beginning of the experiment. The chlorides present remained soluble throughout, and afforded a standard of comparison absolutely independent of the volume changes occurring in the solution—changes due to removal of the soluble constituents and to loss of steam by leakage. The solution to be analysed contained calcium sulphocyanide and ferrous chloride. The method of analysis adopted was to treat the portion withdrawn (usually 5 c.c.) with caustic soda to precipitate the iron, and, after making the whole up to, say, 250 c.c., titrating aliquot portions with silver nitrate, for chloride *plus* sulphocyanide, and with permanganate solution for the sulphocyanide.



The following yields are typical of those obtained :—

| Experiment. | c. | d. | e. | f. |
|---|---|---|---|---|
| RCNS unchanged ....... | 6·0 | 1·0 | 14·5 | 0·50 |
| ,, converted to ferrocyanide. | 86·0 | 91·3 | 81·5 | 96·75 |
| ,, lost (by diff.) .... | 8·0 | 7·7 | 4·0 | 2·75 |

From these experiments, taken in conjunction with those earlier given, I think it might be safely concluded that sulphocyanide solution can be almost completely decomposed under the conditions described, and that certainly 90 per cent. may be obtained as ferrocyanide. What loss there is, is due to the formation of HCN and $NH_3$, both of which compounds could be detected in the evolved gases.

As before stated, the ferrocyanide produced appears to exist as ferrous ferrocyanide, and, as obtained, it is admixed with sulphide of iron. This reaction product forms a finely divided black precipitate. Its removal from the autoclave was effected by washing out with a good stream of water. The precipitate was then filter-pressed and washed. When iron borings are used, the portion not converted to sulphide may, owing to the greater weight of its particles, be readily separated for re-use from the cyanogen compound during the washing process, and on the large scale this separation would, of course, be made.

The admixed sulphide and cyanide of iron was found to be attacked readily by solutions of the caustic alkalis and earths (KHO, NaHO, $Ca(OH)_2$) with the production of a soluble ferrocyanide and insoluble sulphide of iron. Treatment with hydrochloric acid, on the other hand, dissolved out the ferrous sulphide, and left the cyanide of iron as a pale blue precipitate. This cyanide was insoluble in hot potassium carbonate solution; with this reagent, however, potassium ferrocyanide was produced when air was blown through the solution.

Analysis of the mixed precipitate showed that it practically consisted of the mixture $Fe(CN)_2$, 2FeS with free iron.

In the cyanide of iron left after the treatment of the mixed precipitate with HCl, the ratios $\frac{CN}{Fe} = \frac{2·08}{1}$ and $\frac{2·09}{1}$ were found. The compound has the composition $[Fe(CN)_2]x$; it thus corresponds to ferrous ferrocyanide. This composition is confirmed by its insolubility in alkaline carbonate solutions in which Prussian blue is soluble, and its light colour.

From this precipitate are to be obtained the ferrocyanide in the form of a soluble salt, and the iron salt ($FeCl_2$) to be used in the following operation.

From what I have said above, two modes of procedure are possible. Either the ferrocyanide may be extracted directly by treatment with alkali, or the ferrocyanide may be left insoluble after removal of the ferrous sulphide with acid.

The question arises which is the better method to pursue.

From small-scale experiments it was found to make no difference in the yield of alkaline ferrocyanide whichever method was followed. In our experiments, where the preparation of ferrocyanides was the object in view, simple extraction with caustic alkalis was the first method tried. It was at this stage that a peculiar difficulty presented itself. For complete extraction of the ferrocyanide it was found necessary to use a very large excess of alkali, and this for the reason that part of the alkali was rendered insoluble and therefore was lost in the process. The loss—equally great whether the sulphocyanide decomposed be the copper or the calcium salt—usually amounted to some 12 per cent. It was found to be due to the formation of insoluble compounds from the alkali and iron sulphide.

That loss of alkali occurs when solutions of caustic alkalis and alkaline carbonates are heated with pure sulphide of iron, has been proved by Dr. Zahorski and Mr. Thomasson. The values obtained for caustic soda, caustic potash, and sodium carbonate indicated a fixation of one equivalent of the alkali by each three or four equivalents of the FeS present. The loss observed with these pure materials in the case of the caustic soda is equivalent to a loss of alkali of 22 per cent. in the recovery of the ferrocyanide from the reaction product mentioned above.

Such a loss of course renders extraction with the expensive potash entirely impracticable, and it was on account of this difficulty that the preliminary treatment with acid to remove the ferrous sulphide was tried.

This treatment is effective. Complete removal of the ferrous sulphide in this manner leads, however, to the production of more ferrous chloride than is necessary, or than can be utilised in the process :—

$$2RCNS + FeCl_2 + 2Fe = 2RCl + 2FeCN_2 + 2FeS.$$
$$4HCl + 2FeS = 2H_2S + 2FeCl_2.$$

The sulphur in the sulphocyanide is combined with twice as much iron after decomposition as it was previously.

A certain loss of material thus results whichever method is followed; the loss of acid is much less costly to the process than the loss of potash.

Another projected method of reducing the loss from a monetary point of view was to use a cheaper alkali than potash for the extraction, and to extract direct. The resulting ferrocyanide was then by double decomposition to be converted to the potassium salt, or, if this could not be done, to be used in the form in which it was won.

A certain measure of success was obtained by the use of caustic soda as the extracting agent; and the attempts made to convert this to the potassium salt will be described later.

The solution of alkaline ferrocyanide, whichever way obtained, was filtered clear from insoluble matter, and, after evaporation, allowed to crystallise.

I was particularly struck by the beautiful and ready manner in which the sodium ferrocyanide crystallised out from the alkaline solutions. This was contrary to what I expected. According to Watts' Dictionary, the sodium ferrocyanide crystallises with much greater difficulty than the potassium salt.

I may say at once that the contrary seems to me to be the case, not only in the case of the alkaline extracts obtained as above, but even in solutions of the pure salts. While, however, solutions of the potassium salt appear to be stable after long boiling, the sodium ferrocyanide solution suffers a slight decomposition: ferric hydrate, in small quantity, is deposited, and hydrocyanic acid is given off.

From the alkaline extracts, prismatic crystals of the sodium ferrocyanide, upwards of $1\frac{1}{2}$ inches long and $\frac{1}{4}$ inch broad, were formed after a few hours' standing, and, as a first crop in this form, there were obtained in one experiment 79·5 per cent. of the total ferrocyanide originally present, and from the mother-liquors a further 14·0 per cent.

In addition to the longer prismatic crystals, there were also obtained smaller rhombohedral ones. Crystals of this form seem to result when the crystallisation sets in slowly; a rapid crystallisation gives rise to long prisms and needles. Both forms appear to belong to the same system; they certainly possess the same composition.

This composition is different to that given in Watts' Dictionary, and I propose to make it and the solubility of sodium ferrocyanide the subject of a communication immediately following this.

To sum up the matter so far described, sulphocyanide of iron solutions may be completely decomposed by heating under pressure at a temperature of 140°—150° C. with a metal such as iron or copper. As products there are obtained the sulphide of the metal employed for the decomposition, and an insoluble cyanide of iron containing practically the whole of the cyanogen originally present in the iron sulphocyanide. This cyanide of iron is split up by alkali to form its equivalent of alkaline ferrocyanide.

Further, it is not necessary to employ pure sulphocyanide of iron solutions; a solution of calcium sulphocyanide in the presence of an excess of soluble iron salt is sufficient. This affords a ready means of converting commercial sulphocyanide of calcium liquors to alkaline ferrocyanide without handling of solids, and consequently without application to the methods of fusion which I have previously shown to be subject to many difficulties and disadvantages.

Before concluding this portion of my work, it may be of interest to state that an attempt at the direct production of cyanide of iron from $CS_2$ and $NH_3$—by heating under pressure at 140°—160° C. with metallic iron and either ferric hydrate or ferrous sulphate and caustic soda—led in three experiments to yields of 25, 26, and 28 per cent. respectively calculated on the $CS_2$ employed. The reaction resulted in a very dirty and mixed product, which was not fully investigated. It is possible that it contained, in addition to sulphocyanide, formates and trithiomethylene (trithioformaldehyde), since Loew has shown that these substances result when $CS_2$, water, and iron filings are heated together (Ber. **13**, 324).

## THE COMPOSITION AND SOLUBILITY OF SODIUM FERROCYANIDE.

BY JAMES T. CONROY, D.SC., PH.D.

As mentioned in the previous paper, the composition of the sodium ferrocyanide crystals obtained in my experiments differed from that given for this salt in Watts' dictionary.

Here the salt is stated as crystallising with 12 molecules of water of crystallisation. My preparation appeared to contain only 10 molecules of water.

A search through the better-known works of reference made it clear that the formula $Na_4FeCy_6$, $12H_2O$ (due to Berzelius) was the one most generally regarded as correct. It is the one accepted by Morley and Muir in their re-issue of Watts' Dictionary, but in addition a formula $Na_4FeCy_6$, $9H_2O$ (Weith) is also quoted.

Both these formulæ are mentioned in Comey's Dictionary of Solubilities, and, in addition to them, also one of Pebal's: $Na_4FeCy_6$, $10H_2O$.

Considering such conflict of opinion, I thought it would be as well to determine the water of crystallisation in sodium ferrocyanide obtained from more than one source, and below are the results obtained:—

*Water of Crystallisation of Sodium Ferrocyanide.*

| Source of Crystal. | Weight taken. | Temp. of drying. | Loss, $H_2O$. |
|---|---|---|---|
| | Grms. | | Per Cent. |
| Own preparation, Lot 1 ..... | 20 | 110° C. | 38·00 |
| " " " 2 ..... | 2 | " | 37·30 |
| Prism-shaped crystals, Lot 3 | 2 | " | 37·28 |
| Rhomb " " " 4 | 2 | " | 35·97 |
| Pure salt, bought ........... | 250 | " | 37·20 |
| " " ........... | 2 | " | 36·23 |
| | | 210° C. | 36·68 |
| " (recrystallised) .. | 2 | 110° C. | 36·45 |
| | | 210° C. | 37·32 |
| | | Mean........... | 36·94 |
| Calculated for $Na_4FeCy_6$, $10H_2O$ ............ | | | 37·20 |
| " $Na_4FeCy_6$, $9H_2O$ ............ | | | 34·75 |
| " $Na_4FeCy_6$, $12H_2O$ ............ | | | 41·50 |

An analysis of the residue left after drying at 110° to 120° C. by titration with $KMnO_4$ semi-normal solution showed it to consist of 99·5 per cent. anhydrous sodium ferrocyanide.

This result and the above figures make it very evident that the formula $Na_4FeCy_6$, $10H_2O$, given by Pebal, is to be regarded as the correct one.

### *Solubility of Sodium Ferrocyanide.*

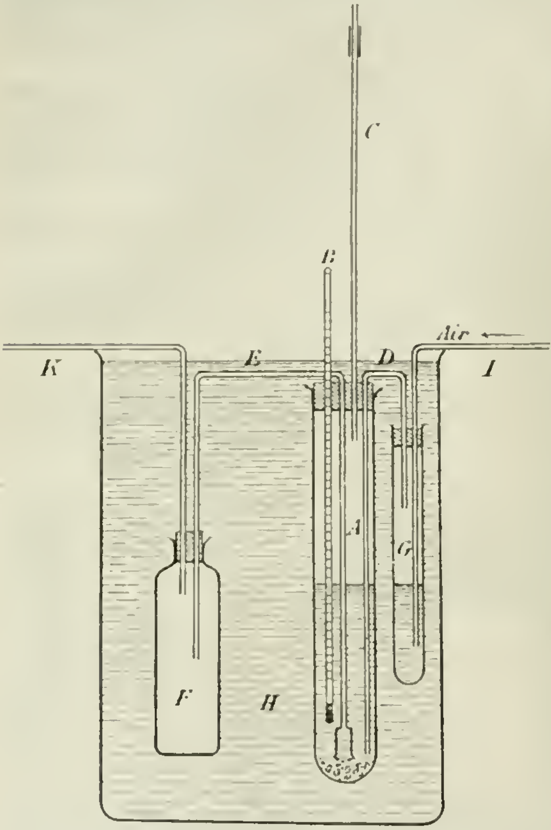
The solubilities, so far as known, are given in Comey's Dictionary. They are:—

1 part $Na_4FeCy_6$, $12H_2O$ in 4·5 parts water at 12° C., and
22 parts " " 100 " " 15°·5 C.,
the two values being practically identical.

The data being so meagre, some experiments were made with the view of finding the solubility curve for this salt between ordinary temperature and 100° C.

The apparatus employed was so constructed that the salt could be dissolved at any desired temperature, and the portion of solution required for analysis be withdrawn without any change of temperature taking place during such withdrawal. The arrangement employed is shown in the accompanying sketch.

The wide test tube A containing the salt to be dissolved is fitted with a rubber stopper bored with four holes. Through these enter a thermometer B, a glass tube C to serve as an escape for the air used to agitate the solution and solvent, and also to clamp the apparatus, a glass tube D for the passage of the agitating air into the apparatus, and a tube E fitted at the one end with a filter and serving to conduct the saturated solution into the collecting vessel F. The tube E slides within a wider glass tube set in the rubber stopper of A, and it is only during the withdrawal of a sample that the funnel is immersed in the solution in A. By means of rubber tubing the sliding tube E is connected in a water-tight manner with the tube through which it slides. The whole apparatus is encased in a water-bath H. In working, air passes in at I, and, after passing through the water in G (found necessary to allow for the evaporation in A which occurs at boiling point), bubbles through the liquid and salt in A, finally escaping through C.



When the solution in A had been agitated for some time and at a constant temperature, the filter tube E was lowered into the solution and the air supply stopped. By sucking and blowing at K, the filter was washed out several times with the saturated solution, and finally, if the temperature was still constant, a sample of the solution was drawn over into F.

The solution so obtained was weighed and then evaporated to dryness, and dried at 110 to constant weight in a platinum dish, and the weight of the dried salt taken. The result was also confirmed in some cases by titration. The difference in the two weights gave the quantity of water requisite for the solution of the anhydrous salt.

In nearly all determinations the agitation was continued for some time after the withdrawal of the sample and a second portion of the solution collected. This acted as a check on the first determination.

Two series of readings were made:—

I. Temperature taken from water-bath only.

II. Temperature taken from thermometer in the ferrocyanide solution.

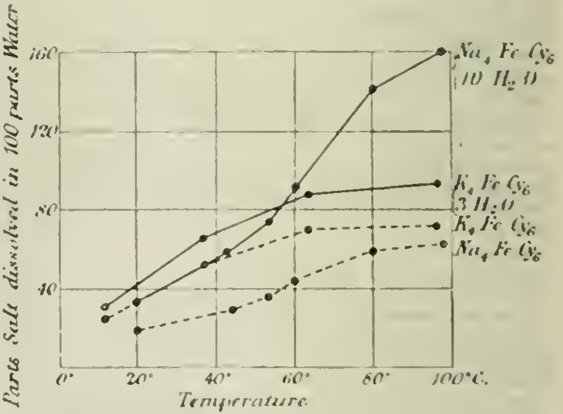
A comparison of the solubilities of the sodium and potassium ferrocyanides is shown in the accompanying curves, which are given for both anhydrous and crystallised salts.

It will be observed that at temperatures below 54 C. the sodium salt is less soluble than the potassium salt. Above this temperature the solubility of the sodium salt ($Na_4FeCy_6$, $10H_2O$) becomes the greater.

Wyrouboff, in a paper dealing with the ferrocyanides (see Watts' Dictionary, 7, 112), states that the sodium salt, "which is less soluble than the potassium salt, may be prepared by double decomposition of the latter with *any* salt of sodium."

| Series. | Temperature, °C. | 100 parts Water dissolve | |
|---|---|---|---|
| | | $Na_4FeCy_6$ | $Na_4FeCy_6$ $10H_2O$. |
| I. | 18 | 16·5 | 28·9 |
| | | 16·9 | 30·0 |
| II. | 20 | 17·85 | 31·8 |
| | | 17·90 | 31·9 |
| I. | 42 | 30·2 | 58·5 |
| II. | 53 | 36·8 | 75·0 |
| | | 37·4 | 76·8 |
| I. | 58 | 41·7 | 83·4 |
| | 60 | 42·5 | 90·2 |
| II. | 77 | 54·8 | 129·5 |
| | 80 | 59·2 | 146·0 |
| | 96 | 62·1 | 157·0 |
| I. | 98 | 61·8 | 155·0 |
| | | 61·4 | 158·0 |
| II. | 98·5 | 63·0 | 161·0 |

That part of his statement regarding the solubility is, we have seen, only partially true; the same may be said also of that portion relating to the preparation of the sodium salt.



The solubility curve just given tells us at what concentration sodium ferrocyanide may be expected to crystallise from its solution *in water*; it does not follow that crystallisation will set in at the same concentration when the solvent is not pure water, but is a solution of some salt other than sodium ferrocyanide. The solubility of sodium ferrocyanide, not in water, but in such mixed solutions, is, under fixed conditions of temperature, the key as to whether this ferrocyanide will crystallise out. Indeed, the sodium ferrocyanide may not separate at all if potash salts are present in solution, but only potassium ferrocyanide crystallise out.

This the following experiments, tried after making the solubility determinations to be given later, will prove.

### *Crystallisation of Alkaline Ferrocyanides in presence of Alkaline Carbonates.*

I. 60 grms. (0·87 equiv.) $K_2CO_3$ were dissolved in 60 c.c. hot water and added to 50 grms. (0·41 equiv.) $Na_4FeCy_6$, $10H_2O$ dissolved in 50 c.c. hot water.

Immediately on cooling, crystals of potassium ferrocyanide began to separate out, and in a few hours 86 per cent. of the total ferrocyanide present had crystallised as the potash salt. This value rose on longer standing to 93 per cent.

II. 50 grms. (0·91 equiv.) $Na_2CO_3$ were dissolved in 150 c.c. hot water, and added to 50 grms. (0·47 equiv.) $K_4FeCy_6$, $3H_2O$ dissolved in 100 c.c. hot water.

No crystal formation occurred until the bulk of the liquid had been reduced by one half. Well-formed crystals of the potassium ferrocyanide admixed with more massive crystals of the sodium ferrocyanide were then obtained in not very large amount. The presence of each ferrocyanide was confirmed by the analysis of the two lots of separated crystals.

From such admixtures, then, the sodium ferrocyanide may be obtained; but the tendency is to form the potassium ferrocyanide, and this almost quantitatively if potassium carbonate is present in large excess.

### *Crystallisation of Alkaline Ferrocyanides in presence of Alkaline Chlorides.*

I. Experiments were carried out in a similar manner to those performed with the carbonates.

With 60 grms. NaCl (1·02 equiv.) and 50 grms. $K_4FeCy_6$, $3H_2O$ (0·47 equiv.), it was found possible to obtain 91 per cent. of the ferrocyanide in the form of the crystalline compound $Na_4FeCy_6$, $10H_2O$.

II. On the other hand, with a mixed solution containing 60 grms. KCl (0·8 equiv.) and 50 grms. $Na_4FeCy_6$, $10H_2O$ (0·41 equiv.), it was found possible to obtain 60 per cent. of the ferrocyanide as the potash salt. Further crystallisation led to an admixture of sodium and potassium ferrocyanides with chlorides.

Here again we see that by altering the conditions it is possible by means of alkaline chlorides to convert sodium ferrocyanide into potassium ferrocyanide, and *vice versâ*.

The tendency is, however, towards the formation of the sodium salt.

The explanation of these phenomena is given by the following table:—

| Number of Determinations. | Salts present. | Temperature. | Grms. of each Salt per 100 grms. Water (in Mixture). | | Separate Solubilities of the Salts in Pure Solution (Grms. Salt per 100 $H_2O$). | |
|---|---|---|---|---|---|---|
| | | ° C. | $Na_2CO_3$ | $K_4FeCy_6$ | $Na_2CO_3$ | $K_4FeCy_6$ |
| 8 | Potassium ferrocyanide, sodium carbonate | 22 | 29·9 | 26·7 | 23·8 | 21·85 |
| 4 | | 97 | 42·0 | 27·5 | 45·5 | 71·00 |
| | | | $Na_2CO_3$ | $Na_4FeCy_6$ | $NaCO_3$ | $Na_4FeCy_6$ |
| 1 | Sodium ferrocyanide, sodium carbonate.... | 22 | 22·6 | 6·5 | 23·8 | 19 |
| 3 | | 95 | 29·8 | 36·8 | 45·6 | 62 |
| | | | | $K_4FeCy_6$ | | |
| .. | Potassium ferrocyanide, potassium carbonate | 20 | .. | 0·0 | .. | .. |
| | | | NaCl | $K_4FeCy_6$ | NaCl | $K_4FeCy_6$ |
| 3 | Potassium ferrocyanide, sodium chloride ... | 20 | 26·6 | 17·8 | 36·0 | 21·0 |
| 2 | | 93 | 27·4 | 35·9 | 39·3 | 62·0 |
| | | | KCl | $K_4FeCy_6$ | KCl | $K_4FeCy_6$ |
| 2 | Potassium ferrocyanide, potassium chloride | 21 | 27·2 | 4·2 | 35·0 | 21·0 |
| 1 | | 99 | 39·6 | 17·0 | 56·0 | .. |
| | | | NaCl | $Na_4FeCy_6$ | NaCl | $Na_4FeCy_6$ |
| 2 | Sodium ferrocyanide, sodium chloride...... | 21 | 29·0 | 5·8 | 36·0 | 18 |
| 1 | | 90 | 24·7 | 21·3 | 39·0 | 61 |

The values given for the solubilities are the mean values calculated from the number of concordant experiments given in col. 1.

In the admixed potassium ferrocyanide and sodium carbonate the same results were obtained at the lower temperature (22° C.)—

(*a*) By agitating $K_4FeCy_6$ andydrous + $Na_2CO_3$ in $Na_2CO_3$ solution.

(*b*) By agitating $K_4FeCy_6$ anhydrous + $Na_2CO_3$ in $K_4FeCy_6$ solution.

(*c*) By agitating 1 equiv. $Na_4FeCy_6$ with 1 equiv. $K_2CO_3$ in water.

(*d*) By agitating after addition of $Na_2CO_3$ 1 equiv. to *c*.

The values given for 97° were obtained by dissolving $K_4FeCy_6$ in $Na_2CO_3$ solution.

In the admixed chlorides and ferrocyanides of the same metal, the values given for the ferrocyanide solubilities at 20° are probably too high. This is due to the method of analysis, the ferrocyanide being estimated by titration with semi-normal potassium permanganate solution. As in some of these cases the permanganate required amounted to only 0·2 c.c., any error in the titration would make a big difference in the relative amount of ferrocyanide found.

At ordinary temperature we might say that ferrocyanide of potassium is very slightly soluble in saturated KCl solution, and ferrocyanide of sodium is very slightly soluble in saturated NaCl solution.

Similarly we find that in admixtures of ferrocyanides and carbonates, each ferrocyanide is very little soluble at ordinary temperature in the solution of the carbonate of its own metal.

The table shows this clearly tor sodium ferrocyanide in sodium carbonate solution.

With regard to a mixture of potassium carbonate and potassium ferrocyanide, Dr. Hetherington has shown that the latter salt is practically insoluble in presence of the former. Serving as a confirmation to this is the fact that addition of excess of potassium carbonate to the solution of admixed sodium and potassium ferrocyanides and carbonates (equal 1 equiv. $K_2CO_3$ to solution *c* given above), throws all but traces of the ferrocyanide out of solution.

Returning now to the experiments on the mutual convertibility of sodium and potassium ferrocyanides, we find that what practically decides the result is the solubility of the desired ferrocyanide in the presence of the salt from which it derives its metal—sodium or potassium, as the case may be.

Thus, considering the conversion of potassium ferrocyanide into the sodium salt by means of sodium chloride, the first addition of a small quantity of the sodium chloride gives a solution in which we may consider four salts to exist side by side. These salts are the chlorides and ferrocyanides of potassium and sodium. The addition of more sodium chloride tends to increase the quantity of sodium ferrocyanide existing in the solution. It also reduces the solubility of this salt, and at a certain concentration, begins to throw it out of solution. This allows the conversion of more potassium ferrocyanide to sodium ferrocyanide to take place, and as this fresh portion formed is removed, the reaction proceeds further, until, as I have shown, a conversion of over 90 per cent. may be effected.

A similar explanation applies to the other conversions.

These explanations may possibly be considered long and unnecessary. I give them, however, because they were forcibly brought to my mind by a study of the solubility factors of the mixed solutions. It is a fact which can only be made clear by experiment, that the convertibility of one salt into a second, where both salts are soluble, cannot be correctly argued from the solubilities of the individual salts in water, as is frequently done. For safe guidance in a technical process, the solubility of the products of each possible combination in any particular system must be known.

In conclusion, I would like to state that I am permitted to read these papers through the kindness of the Directors of the United Alkali Company, Limited.

The experiments were all carried out in the Company's Central Laboratory, Widnes, and under the direction of Dr. Hurter. To him are my thanks especially due for his many valuable suggestions, and for the interest taken in this work during its progress from stage to stage.

### Discussion.

The Chairman remarked that the preparation of such substances as ferrous and cuprous sulphocyanide on a large scale was extremely interesting, owing to the many reactive properties these substances possessed. He asked whether Dr. Conroy had tried them as absorbents in gas analysis, as, owing to the ease with which they were oxidised, they might be of value for the estimation of oxygen. In regard to the formation of "cyanide of iron" from cyanogen sulphide as an intermediate product, as suggested, he thought it more probable that a complex cyanogen compound was the origin of the "cyanide of iron"; and would like to know whether Dr. Conroy had found any evidence of a compound containing proportionately more iron than ferrous sulphocyanide in his experiments. The loss referred to in the table given seemed considerable. Had Dr. Conroy been able to ascertain what products were formed to account for this loss, or did the ammonia and other gases that were evolved in the decomposition really represent it? The Chairman also mentioned the value of Victor Meyer's air bath for drying substances at a constant temperature, and described the apparatus. In respect to the neat apparatus Dr. Conroy used for his solubility determinations, how long did he find it necessary to pass the current of air in order to obtain a saturated solution?

Dr. Conroy, in reply to the Chairman, said, with regard to the loss shown during decomposition of the sulphocyanides, the amount of gas given off did not, apparently, correspond with the loss of even $2\frac{1}{2}$ per cent. The quantity of gas was small, amounting to four litres or so. Most of the loss he thought was due to errors in manipulation. Such loss as was due to formation of ammonia was not separately estimated; the loss was really very slight. With regard to the intermediate formation of cyanogen sulphide, he only threw out this as a suggestion. He had tried some experiments in which cyanogen sulphide was heated with metallic iron, and certainly got Prussian blue formed, but this was admixed with much sulphocyanide. These experiments were not carried out under pressure, but were merely qualitative and performed in test tubes. Cyanogen sulphide seemed to have been accurately investigated, and it was known to yield sulphocyanide and cyanide when treated with reducing agents. He had performed no experiments which would show whether the compound "cyanide of iron" would contain more iron than that shown in the formula $[\mathrm{Fe(CN)_2}]x$, and it was quite possible that the compound was not originally existent in the reaction product; it had been formed on dissolving in hydrochloric acid the ferrous sulphide present in this product.

As to any probable use of ferrous sulphocyanide as an absorbent in gas analysis, he had not tried any experiments, but he thought it might prove a useful absorbent for small quantities of oxygen.

In reply to the Chairman's questions in connection with his second paper, Dr. Conroy said that solution was complete after a quarter of an hour's agitation; a sample was then taken and the agitation continued another 10 minutes or so, when a second sample was withdrawn. The two values for the solubility given at any particular temperature were obtained from two portions of solution so collected. His experiments were done in an ordinary bath, but the Victor Meyer constant-temperature bath would have been much more convenient.

# London Section.

*Meeting held on Monday, January 3rd, 1898.*

DR. MESSEL IN THE CHAIR.

## THE EFFECT OF DIFFERENT TEMPERATURES IN THE EXTRACTION OF TANNING MATERIALS.

BY J. GORDON PARKER, PH.D.

*Contribution from the London Leather Industries' Research Laboratory, Herold's Institute, Bermondsey.*

(Abstract.)

In a paper that appeared in this Journal, 1895, 124, of the joint authorship of Prof. H. R. Procter and myself, a new method was described of measuring the colour contained in a solution of tanning material of a known strength. A second paper of theirs, on the effect of different temperatures in the extraction of tanning materials, appeared in the same volume of the Journal, p. 635. The latter paper dealt with eight of the most common tanning materials. As the information given in these papers has been found to be of considerable use in the tanning trade, I have carried the research further, and examined all the other tanning materials that are used to any extent in this country. The accuracy of the results formulated in the two published papers has in most cases been borne out; and on comparing the figures now to be presented to you with those of the papers referred to, it will be clearly seen that the greater part of the tanning materials give off their strength most readily at a temperature of about 60 degrees. No hard-and-fast rule can be laid down, however, as each material appears to possess some peculiarity of its own.

The method of extraction and of colour measurement of the tanning materials now examined has been the same as that before adopted. This course was followed so that the results might be comparable of the research as a whole.

A suitable quantity of the tanning material was ground to a moderately fine powder in one of Burroughs and Wellcome's drug mills; a portion of the powder was weighed out and placed in a beaker, at the bottom of which was some pure silver sand; and in this a siphon filter was inserted, as described in a published paper by Prof. Procter. In employing this method, I have tried to approximate as much as possible in principle to the process of the tanyard.

The beaker was now put into a water-bath, and the material was covered with distilled water, and allowed to macerate over night (*circa* 16 hours); after which the bath was heated so that a thermometer inside the beaker reached the required temperature, and the extraction commenced; the liquor, as it siphoned slowly over, was replaced by water of the same temperature as that required for extraction, and percolated through the material; a litre of liquor was thus extracted in from $2\frac{1}{2}$—3 hours.

This supply of water was found to be practically sufficient to extract all the available tannin and colour; very small additional quantities of these being obtained, when, for experiment, a second litre was percolated at the same temperature.

The extract-solution was now allowed to cool, filtered, analysed, and measured in a $\frac{1}{2}$-inch cell by Lovibond's tintometer, and the results calculated for a $\frac{1}{2}$ per cent. solution of tanning matter; this was shown to be permissible in the former of the papers above referred to.

*Scotch Larch Bark.*—The first material investigated was a sample of Scotch larch bark; 30 grms. of the ground material being extracted, as described above, at each of ten temperatures. I have tabulated the results in percentages of the highest amount of tanning and colouring matter obtained from the sample. For the sake of simplicity, the total colour is calculated in this way, but as the tint continually varies, and red and yellow are not of equal practical importance, the actual measurements at each temperature are also given, reckoned for a ½-inch layer of a solution containing exactly ½ per cent. of tanning matter. The sample gave by analysis the numbers under A. in Analytical Table below.

*Analytical Table of Maxima of Tanning and Colouring Matters Extracted.*

| — | A. | B. | C. | D. | E. | F. | G. | H. | I. | J. | K. | L. | M. | N. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Per Cent. | | | | | | | | | | | | | |
| Tanning matters | 17·8 | 46·1 | 45·7 | 36·7 | 24·6 | 30·6 | 60·0 | 50·0 | 16·5 | 70·0 | 4·3 | 27·6 | 29·6 | 26·6 |
| Soluble non-tannins | 9·6 | 25·0 | 23·6 | 15·1 | 13·7 | 14·8 | 23·0 | 27·8 | 13·0 | 4·6 | 2·6 | 15·5 | 9·0 | 13·0 |
| Insoluble fibre, &c. | 60·4 | 18·3 | 25·1 | 36·1 | 50·2 | 42·5 | 7·4 | 4·1 | 59·1 | 9·3 | 80·9 | 0·7 | .. | 2·0 |
| Moisture | 12·2 | 10·6 | 9·6 | 12·1 | 11·5 | 12·1 | 9·6 | 18·1 | 11·4 | 16·1 | 12·2 | 56·2 | 61·4 | 58·4 |
| Total | 100·0 | 100·0 | 104·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 | 100·0 |
| Maximum of colouring matter ... Red | .. | 2·6 | 1·8 | 3·6 | 6·8 | 5·6 | 3·2 | 5·0 | 4·3 | 10·4 | 15·5 | 6·0 | 3·4 | 18·6 |
| Yellow | .. | 12·4 | 14·6 | 8·4 | 9·2 | 8·6 | 14·8 | 15·0 | 8·7 | 22·0 | 33·5 | 11·0 | 7·6 | 31·0 |
| Black | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | 1·0 | .. | .. | 0·4 |
| Total | .. | 15·0 | 16·4 | 12·0 | 16·0 | 14·2 | 18·0 | 20·0 | 13·0 | 32·4 | 50·0 | 17·0 | 11·0 | 50·0 |

Taking the 17·8 total amount of tannin ("tannin" is used throughout this paper in the sense of "tanning matters absorbed by hide-powder") as 100 per cent., and the 4·6° red and 16·8° yellow, or 21·4 total, degrees of colour in a ½-inch layer of the solution containing ½ per cent. of tanning matter also as 100 per cent., and the results were calculated in percentage of these.

### *Larch Bark.*

By cold-water extraction, 53·9 per cent. of the available tannin was extracted, and 46 per cent. of the colouring matter. Both the tannin and colouring matter gradually increased, until between 60° and 70° the total available tannin was easily extracted, and at the same temperature only 61·2 per cent. of the colouring matter. By increasing the temperature of extraction, the yield of tannin again decreases, but the colouring matter rises. It would therefore be advisable in practice, when using this material for the tannage of sheep skins, to never exceed 70° in the extraction.

*Divi-Divi* (Cæsalpina coriaria).—Investigation was made of a sample of this material, 12 grms. of it, ground, being taken in each experiment. As divi-divi is stronger in tannin than larch bark, a larger quantity would have given too strong liquors for conveniently detannising by the hide-powder method. Each 12 grms. was allowed to soak for 16 hours, as with the larch bark, before the extraction was commenced. The analysis of the sample gave as maxima the numbers under B. in above Table.

### *Divi-Divi.*

By cold extraction, this material parts with 85 per cent. of its available tannin, and gives off its maximum yield between 50° and 60°. The colouring matter does not materially increase until the temperature is raised to boiling.

By its use, a light-coloured leather is produced. The best results to leather tanned with this material will probably be secured if it is extracted at this temperature. It may here be noticed that as the temperature of extraction is increased the tannins decrease, with a corresponding increase of the non-tannins. This would point to the tannins decomposing and being changed into non-tannins, or probably anhydrides of different tannic acids which have no affinity for hide substance. The colour does not seem to be materially affected until the temperature is raised to 100°. By boiling, over 32 per cent. more of colouring matter is given off. This very likely is due to the tannins being changed into soluble reds.

*Algarobilla.*—Twelve grammes of this material, which is a pod of the "Cæsalpina brevifolia," and somewhat closely resembles divi-divi, were extracted. The maximum analysis of the sample gave figures under C. in above Table.

Algarobilla, whilst giving off less than 40 per cent. of its colouring matter at a temperature below 30°, gives off all its tannin at that temperature. Looked at from the practical standpoint, this is a fortunate circumstance. Here again we notice a decrease of tannin as the temperature rises, with a corresponding increase of the non-tannins. And the same explanation as with divi-divi may be given, *viz.*, that the tannins are decomposed into non-tannins.

*Smyrna Valonia.*—This material was dealt with in the published papers referred to, but a very choice sample has since come to hand. Examination was made of this, as it was of interest to ascertain whether a specially fine sample would behave in the same way as the ordinary commercial article. Of 12 grms. taken, the maximum analysis gave figures under D. in above Table.

### *Smyrna Valonia.*

This sample will give off 90·5 per cent. of the available tannin and 31·6 per cent. of the total colouring matter by cold extraction; required to be raised to a temperature exceeding 90° before it could be fully extracted, and at that temperature only 68·3 per cent. of the colouring matter dissolved.

Comparing these figures and the figures already published, it will be found that the sample now examined did not part with its maximum amount of tannin at so low a temperature. The earlier results show the maximum yield of tannin at 60° in one case and 70° in another; but here the temperature was about 90° before the maximum was reached. This may have been due to the sample being of a different variety, and coming from another district. In other respects the samples behaved pretty much alike. It would seem to be best never to exceed 80° in the extraction of this material.

*Canaigre Root.*—This is a comparatively new tanning material in this country. It is a root about 4 ins. long by 2—3 ins. in diameter, very much resembling the parsnip, and is now being extensively cultivated in Mexico and California. It may be used when two years old, but, at three years old it contains a larger amount of tanning matter. The analysis of two-year-old roots, 12 grms. being treated, gave as maxima numbers under E. in above Table.

*Canaigre Root*, two years old parts with 65·4 per cent. of the available tannin, and 46·2 per cent. of the available colouring matter by cold extraction, the maximum yield of tannin being given between 40° and 50°.

Maximum analysis of three-year-old roots, 12 grms. being treated, gave numbers under F. in above Table.

In a three-year-old canaigre, 78 per cent. of tannin is given off by cold extraction, the maximum yield between 50° and 60°. The colour measurements show the older roots to contain less colouring matter in proportion to the tannin than in the younger roots.

It will be noticed that the maximum of tannin was given off in both samples below 60° C., which is to be thus accounted for. Canaigre contains upwards of 20 per cent. of starch; as the temperature rises this coagulates around the tannin cells and prevents the extraction of the tannin. Great difficulty is experienced in the extraction of this material for analytical purposes, seeing that as soon as the temperature exceeds 60°, coagulation of the starch commences and blocks up the extraction apparatus. Extraction can be effected only with great care, and by patient stirring. In the first sample, as the temperature rose from 60° both tannin and non-tannin matters decreased, whereas in the three-year growth only the tannins decreased, the non-tannins increasing slightly. In the canaigre of three years' growth, the tannin matter increased in a higher ratio than the colouring matter; in fact, the colouring matter of the second sample is lower in proportion than in the first. In the tanyard this material must always be extracted under 55°, otherwise the starch by coagulation would completely block the extraction pits.

*Gambier.*—This material is of great importance to the tanner. It is used very extensively both in the manufacture of light dressing leathers and of heavy sole leathers. Different methods of extraction are used in practice, some tanners extracting cold, others at a temperature of 40°, some bringing the material almost to a boil, and some extracting by boiling under pressure even. Gambier is an extract of the "Uncaria Gambier" plant, which grows in the islands of the Malay Archipelago; the manufacture is almost entirely in the hands of the natives. It comes to our market in small (inch) cubes, in which form it is purest; and in blocks or bales weighing sometimes 2 cwt. The latter are generally very impure; for the natives seem to better understand the adulteration of the material than the manufacture of a good article. A sample of each form, cube and block, was investigated. The sample of cube gambier gave by analysis numbers under G. in Table on page 107.

The block gambier on analysis gave the numbers under H. in the Table (*ibid.*).

The tannin of cube gambier dissolves to the extent of 78 per cent. in cold water; block gambier parts with only 50 per cent. at the same temperature. By increasing the temperature of the water used in the extraction of both materials, the tannin gradually increases until the water is used in the boiling state. On being boiled for half an hour, the yield in both cases is again increased, probably due to some difficultly soluble anhydrides being transformed into their corresponding tannic acid.

It will be noticed that boiling gives a very marked increase in the tannin yield, especially in the cube gambier; while the yield of colouring matter increases even more rapidly still.

The figures prove that in the preparation of this material for practical use it should be heated to at least 98°; at this temperature over 90 per cent. of the tannin is extracted and only 80 per cent. of the colour. Those tanners who follow the custom of extracting the gambier with cold water, waste about 25 per cent. of the available tanning matter. They gain in the colours imparted to the goods, but this gain in colour cannot possibly compensate for the loss in tannin.

*Pine Bark.*—This material is not much used in England; but is extensively used in Germany and some parts of Austria. A sample received from Saxony analysed as under I. in Analytical Table.

This material requires a high temperature in order to extract all the tannin from it, between 80° and 90° being the best for practical purposes; 99·9 per cent. being then given off with only half the available colour.

*Quebracho Extract.*—This was one of the materials treated, although it is only a prepared extract made from the quebracho wood. Analysis of the wood itself is given in one of the 1895 papers. This extract, which is sold in solid form, is being more and more used in the tanning trade. The analysis of a sample of solid, quebracho extract gave the figures under J. in Table.

QUEBRACHO EXTRACT.

*Effect of Different Temperatures.*

| Temperature of Extraction. | Tanning Matters absorbed by Hide. | Soluble Non-tanning Matters. | Per Cent. of Tannin on Maximum Yield. | Colour of ½ per cent. Solution in ½-in. Cell. Red. | Yellow. | Total. | Per Cent. of colour on Maximum Yield. |
|---|---|---|---|---|---|---|---|
| ° C. | Per Cent. | Per Cent. | | Deg. | Deg. | | |
| 0–15 | 41·2 | 2·6 | 58·8 | 2·6 | 12·0 | 14·6 | 45·0 |
| 15–30 | 42·8 | 2·6 | 61·1 | 2·9 | 13·0 | 15·9 | 48·8 |
| 30–40 | 50·2 | 2·8 | 71·7 | 3·6 | 13·8 | 17·4 | 53·7 |
| 40–50 | 56·4 | 3·1 | 80·5 | 3·9 | 14·2 | 18·1 | 58·9 |
| 50–60 | 68·2 | 3·6 | 97·7 | 4·5 | 16·4 | 20·9 | 64·5 |
| 60–70 | 68·6 | 3·7 | 98·0 | 5·1 | 18·2 | 22·3 | 68·8 |
| 70–80 | 69·7 | 3·7 | 99·7 | 6·1 | 19·6 | 25·7 | 79·3 |
| 80–90 | 70·3 | 3·8 | 100·0 | 6·9 | 20·2 | 27·1 | 83·6 |
| 90–100 | 68·2 | 4·6 | 97·4 | 7·1 | 21·3 | 28·4 | 87·6 |
| Boiled ½ hr. | 60·6 | 5·9 | 85·7 | 10·4 | 22·0 | 32·4 | 100·0 |

The remarks as to the practice of tanners with gambier apply also to quebracho extract, varying temperatures being employed in dissolving it. The figures obtained show that the temperature for dissolving should not exceed 90°. Quebracho extract seems to behave exactly as does the quebracho wood; and it is probable that on raising the temperature higher than 90°, some of the tannins are decomposed into red anhydrides, as would be expected in tannins of the catechol series.

*Oakwood.*—This material is never used in this country as a tanning material, but in Slavonia, Belgium, and elsewhere, large works have been erected to manufacture an extract made from it, and the bulk of this extract finds its way to the English market. In the extract factories the wood is boiled; the liquid being afterwards treated with blood in order to remove the colouring matter. The extract is then concentrated to a density of about 1·3 specific gravity. As it was desirable to ascertain whether the best results were obtainable by boiling the wood or by treatment of it at a lower temperature, an analysis of a sample of best Slavonian oakwood was made, the bark having been removed. The results are as under K. in Table.

With oakwood it is evident that a high temperature must be used in its extraction for the manufacture of extract. By cold extraction nearly 12 per cent. of the tannin is extracted; at 90° 97·6 per cent.; but the total yield is not given off until boiling point is reached. It would probably be better, however, to extract the wood just below boiling, in order to avoid the high percentage of colouring matter.

*Oakwood Extract, Chestnut Extract, and Hemlock Extract.*—It is customary in using these extracts to mix them direct with cold water or cold tanning liquors. Some experiments made have demonstrated, however, that it is better to warm the water or liquor with which the strong extract is mixed, as this prevents the precipitation of any of the tannins as insoluble or difficultly soluble anhydrides. The following figures show the advisability of doing this. The sample of oakwood extract analysed gave the results shown under L. in Table.

*Effect of Different Temperatures.*

| | Temperature of Extraction. | Tanning Matters absorbed by Hide. | Soluble Non-tanning Matters. | Per Cent. Insoluble. | Per Cent. of Tannin on Maximum Yield. | Colour of ½ per cent. Solution in ½-in. Cell. Red. | Yellow. | Total. | Per Cent. of Colour on Maximum Yield. |
|---|---|---|---|---|---|---|---|---|---|
| Oakwood Extract. | ° C. | Per Cent. | Per Cent. | | | Deg. | Deg. | | |
| | 0—15 | 26·4 | 16·1 | 3·1 | 95·6 | 5·1 | 9·2 | 14·3 | 84·1 |
| | 30—40 | 27·2 | 15·6 | 1·4 | 98·9 | 5·2 | 9·4 | 14·6 | 85·9 |
| | 60—80 | 27·6 | 15·4 | 0·8 | 100·0 | 5·3 | 9·6 | 14·9 | 87·7 |
| | 90—100 | 27·6 | 15·5 | 0·7 | 100·0 | 5·5 | 10·2 | 15·7 | 92·3 |
| | Boiled ½ hr. | 27·4 | 15·4 | 1·0 | 99·1 | 6·0 | 11·0 | 17·0 | 100·0 |
| Chestnut Extract. | ° C. | Per Cent. | Per Cent. | | | Deg. | Deg. | | |
| | 0—15 | 28·0 | 9·0 | 1·6 | 94·9 | 1·8 | 6·1 | 7·9 | 71·8 |
| | 30—40 | 29·0 | 9·0 | 0·6 | 98·0 | 1·9 | 6·3 | 8·2 | 74·7 |
| | 60—80 | 29·6 | 9·0 | 0·2 | 100·0 | 2·0 | 6·6 | 8·6 | 78·7 |
| | 80—100 | 29·6 | 9·0 | 0·0 | 100·0 | 2·1 | 6·7 | 8·9 | 81·8 |
| | Boiled ½ hr. | 29·3 | 9·1 | 0·3 | 98·9 | 3·4 | 7·6 | 11·0 | 100·0 |

The outcome of analysis of a sample of chestnut extract was as under M. in Table (page 107).

The sample of hemlock extract gave the figures under N. in the Table (page 107).

HEMLOCK EXTRACT.

*Effect of Different Temperatures.*

| Temperature of Extraction. | Tanning Matters absorbed by Hide. | Soluble Non-tanning Matters. | Per Cent. Insoluble. | Per Cent. of Tannin on Maximum Yield. | Colour of ½ per cent. Solution in ½-in. Cell. Red. | Yellow. | Black. | Total. | Per Cent. of Colour on Maximum Yield. |
|---|---|---|---|---|---|---|---|---|---|
| ° C. | Per Cent. | Per Cent. | | | Deg. | Deg. | | | |
| 0—15 | 22·6 | 14·6 | 4·8 | 84·9 | 12·8 | 27·2 | 0·0 | 40·0 | 80·0 |
| 30—40 | 25·8 | 13·1 | 3·1 | 96·6 | 15·3 | 28·0 | 0·1 | 43·7 | 87·4 |
| 60—80 | 26·3 | 13·0 | 2·6 | 98·5 | 16·1 | 29·0 | 0·2 | 45·3 | 90·6 |
| 80—100 | 26·6 | 13·0 | 2·0 | 100·0 | 16·8 | 29·6 | 0·2 | 46·6 | 93·2 |
| Boiled ½ hr. | 26·0 | 13·2 | 2·4 | 92·3 ? | 18·6 | 31·0 | 0·4 | 50·0 | 100·0 |

The results above tabulated were obtained by rather a different procedure than that adopted with the solid tanning materials. The extraction bath was not employed, but 12 grms. of the extract were weighed out and mixed with water at the temperature given, the solution being then made up to a litre. When the mixing was with cold water, the insoluble matter in the case of oakwood extract amounted to 3·1; whereas when the extract was mixed with water just below boiling, the insoluble matter was reduced to 0·7. In the case of chestnut extract, the insoluble matter was completely eliminated, and in hemlock very much reduced; and in all cases of mixing with hot water, a corresponding increase of tannin manifested itself. These warm solutions did not deposit sediment on cooling. There is indicated by this that the sudden mixing of a concentrated tanning extract with cold water tends to change some of the tannins into insoluble anhydrides. In order to ascertain whether the same results would come about if the extract were first mixed with cold water and the solution were afterwards warmed, samples of oakwood, chestnut, and hemlock extracts were so mixed, and the solutions were then warmed to 98°. As the sediment which was deposited in the cold-water mixing was reduced to 0·7 by the warming, the inadvisability of cold-water dilution was fully established.

Summarising the results now submitted to you and those of the published papers, some interesting figures present themselves—figures which will perhaps be of assistance in the future from the scientific point of view (they will undoubtedly be of use in practice) as throwing some light upon the nature of the different tannic acids contained in different tanning materials. All the results obtained point in one direction, *viz.*, to an indication of the existence of more than one tannic acid in each tanning material; and it may be that, by extracting a material for a long time at a low temperature, and afterwards the same material at a much higher temperature, it may be possible to isolate the different acids. It will also be noticed that boiling the material in every case not only destroyed some of the tannins, but also very much darkened the colour.

All the above research was conducted in duplicate; three samples of each material being mixed together, and two analyses made at each of the temperatures stated. The results at various temperatures have also been checked independently; and the author desires to tender his sincerest thanks to Prof. Procter and Dr. Hellon, who confirmed results in algarobilla and canaigre, and to Messrs. R. Paget and W. S. Hammersley for divi-divi and valonia and larch bark.

## DISCUSSION.

MR. J. STEWART wished to ask, in connection with this paper also, what was the nature of the precipitate formed. He had his own ideas on the subject, and hoped that Dr. Parker would have answered it more fully in connection with the last paper. It seemed to him that such precipitates must contain a large proportion of gummy matters. He had tested some of the precipitates from bark tanning, and they appeared to contain both resins and gummy bodies. He would like to know whether Dr. Parker had gone beyond this, and could state what the actual constituents were.

MR. A. SEYMOUR JONES said that some eight years ago, when on a visit to Russia, he had been much struck by the brewing of tea there, and it had suggested to his mind that nearly all vegetable substances had a definite leaching point. On returning home he mentioned the matter to his firm's chemist, who, in consequence, made some experiments on sumac and oak bark. The result of that work was that in the case of sumac they had reduced their consumption over 50 per cent. in the first year, and in the case of bark they had reduced it by more than one-third. With a large consumption such as theirs, that represented a very material saving. He thought that was a great point to the credit of the leather-trades chemist, and was a sufficient answer to the feeling, which existed very generally, that a chemical laboratory was a needless expense. In the case of his own firm, the saving effected covered more than twice the cost of the laboratory.

Dr. Parker, replying to Mr. Stewart, said that he was not in a position to add to the information he had already given as to the nature of the precipitates. He did not profess to have any special knowledge on this point. It was, he thought, generally known that many of the tannins were of a gummy or resinous nature, and the sediment formed in casks of extract generally consisted of the less easily soluble tannins or hydrides of the tannic acids. The tannins contained in quebracho extracts were undoubtedly very resinous, and sometimes formed compounds on the leather itself which it was impossible to remove. This fact had been brought out in recent cases of litigation. The testimony which Mr. Seymour Jones had just borne to the value of the chemist in the tanning industry was most important, since his firm were the largest light-leather manufacturers in Great Britain, if not in the world; and he hoped that the general tanner would give his serious attention to it.

*Meeting held on Monday, February 7th,* 1898.

DR. R. MESSEL IN THE CHAIR.

## CLERGET'S METHOD OF ESTIMATING SACCHAROSE (CANE SUGAR).

BY ARTHUR R. LING.

Clerget's method of estimating saccharose, or cane sugar, is widely used for the evaluation of many industrial products. The sugar refiner, the distiller, and perhaps also the brewer, are accustomed to gauge the value of certain saccharine materials which they employ on the basis of the "Clerget Value."

The term "Clerget Value" is, in fact, used as an abstract expression, and the actual information which the method was originally intended to convey—namely, the percentage of saccharose—by the chemist who formulated it, is often lost sight of. Seeing also that numerous modifications of the original plan of carrying out the process have been described, in which the authors omit to mention that the essential object is the estimation of saccharose, there is a danger that Clerget's method may, in the course of time, be degraded to a purely empirical one. I venture to think, therefore, that a brief survey of some points in the history of the subject may not be without value, if only for the sake of averting such a catastrophe as that just mentioned.

This paper will accordingly be devoted, in the first place, to reviewing the literature of the subject, and in the second place, to placing on record some values for the rotatory power of invert sugar confirmatory of those obtained by Herzfeld. I wish, however, to disclaim any idea of dealing exhaustively with all that has been written on the subject.

In the early days of quantitative sugar analysis, when the polarimeter was first employed for that purpose, it soon became evident that a single polarimetric observation was inadequate for the deduction of the percentage of saccharose present in many samples of raw sugar. For, neglecting certain optically active non-saccharine substances that occur in raw sugars, many samples—and especially those derived from the sugar cane—are known to contain notable amounts of cupric reducing sugars, consisting in all probability mainly of invert sugar. It is obvious that in any mixture of saccharose and one or more optically active substance, the true percentage of saccharose may be calculated from the values obtained for the optical rotatory power before and after hydrolysis or inversion, provided the agent employed to effect the hydrolysis is without action on the rotatory power of the other constituent or constituents contained in the mixture.

The first to suggest the idea of estimating saccharose in presence of invert sugar from the optical rotatory power before and after hydrolysis with acid, appears to have been Biot (1842). It was, however, Clerget who subsequently (1849) elaborated this process from the results of his exact experiments on the rotatory powers of inverted saccharose solutions at known temperatures.

The polarimeter employed by Clerget was that of Soleil; an aqueous solution of 16·471 grms. of saccharose per 100 c.c. gave a dextro-rotation of 100 scale divisions on this instrument when the solution was observed in a 200-mm. tube. Clerget found that when to a solution of 8·235 grms. (half the normal weight) of saccharose in 50 c.c., 5 c.c. of concentrated fuming hydrochloric acid are added, and the liquid heated on the water-bath in such a manner that its temperature is raised to 68° C. in 10 minutes, a constant polarimetric reading is obtained on observing the cooled solution at a definite temperature.

In order to correct for the increased volume due to the addition of acid, the solution was polarised in a 220-mm. tube. The temperature of the solution must be kept constant by providing the tube with a water jacket. The results gave the rotation of a solution of 16·471 grms. (the normal weight) of saccharose after inversion per 100 c.c. in a 200-mm. tube. This, according to Clerget, is − 34 sugar divisions at 20° C. From Clerget's results, which have been confirmed by Cassamajor, Ross, Creydt, Bornträger, and others, the following formula is to be deduced:—

$$S = \frac{100\ K}{144 - 0{\cdot}5\ t}$$

in which K = the difference between the polarimetric readings of the solution before and after inversion.

As before stated, Clerget's results have been confirmed by several other observers, and numerous modifications of the method, as originally devised, have been proposed. For a discussion of these I would refer to an excellent paper by A. Bornträger (Zeit. Ver. Rübenzuck.-Ind. 1890, 876).

In this country the polarimeter most generally used for saccharimetric purposes is the German instrument, the Soleil-Ventzke-Scheibler, or the improved form of it, the half-shadow polarimeter with quartz compensation, manufactured by Schmidt and Haensch, of Berlin. One of the distinguishing characteristics of these German instruments is that the scale first introduced by Ventzke is adopted, 100 divisions of which correspond to the rotation produced by a solution of 26·048 grms. of saccharose per 100 c.c., observed in a 200-mm. tube. If determinations are carried out according to Clerget's instructions with these instruments, employing, as they do, a higher weight than the French instrument, the inversion constant is somewhat higher.

In the year 1888 a modification of Clerget's method was devised and elaborated under Herzfeld's directions. It is carried out in the following manner:—

The half-normal weight (13·024 grms.) of sugar is dissolved in 75 c.c. of water in a graduated 100-c.c. flask, 38 per cent. hydrochloric acid (5 c.c.) is then added, and the flask, in which a thermometer is inserted, is placed in a water-bath at 70° C. The flask is shaken continuously, and in this way the temperature of the solution is raised to 67°—70° C. in about 2½ minutes, after attaining which it is kept constant for an additional five minutes, when it is rapidly cooled and polarised in a water-jacketed tube, 200 mm. in length, at a known temperature. It was found that when the polarimetric observation is made at 20° C., and the reading is multiplied by 2 to raise it to that of a normal solution, the value obtained is − 32·66. sugar divisions.

The formula, including the correction for temperature when the observation is made at a temperature other than 20° C., is—

$$S = \frac{100\ K}{142{\cdot}66 - 0{\cdot}5\ t.}$$

The temperature correction may also be made by Hammerschmidt's formula—

$$l_{20} = l\,t + 0{\cdot}0038\ K\ (20 - t),$$

in which $l_{20}$ is the reading of the inverted solution in a 200-mm. tube at 20° C., multiplied by 2, $l\,t$ the same at the temperature of observation, and K the difference between the polarisations of the direct and inverted solutions.

If, instead of the half-normal weight in 100 c.c., a solution of a different concentration is employed, the inversion constant varies. The following table gives the inversion constants for solutions containing 1—20 grms. of sugar in 100 c.c. The values under I′ are calculated by the formula—

$$I' = -\left(31\cdot84 + \frac{i}{20}\right),$$

in which *i* is the polarimetric reading of the inverted solution in a 200 mm. tube, I being the same multiplied by two.

| Sugar, Grms. in 100 c.c. | I. | I′. | Sugar, Grms. in 100 c.c. | I. | I′. |
|---|---|---|---|---|---|
| 1 | 31·85 | 31·90 | 11 | 32·52 | 32·53 |
| 2 | 31·91 | 31·96 | 12 | 32·59 | 32·60 |
| 3 | 31·98 | 32·03 | 13 | 32·66 | 32·66 |
| 4 | 32·05 | 32·09 | 14 | 32·73 | 32·73 |
| 5 | 32·12 | 32·15 | 15 | 32·79 | 32·79 |
| 6 | 32·18 | 32·21 | 16 | 32·86 | 32·85 |
| 7 | 32·25 | 32·28 | 17 | 32·93 | 32·92 |
| 8 | 32·32 | 32·34 | 18 | 33·00 | 32·98 |
| 9 | 32·39 | 32·40 | 19 | 33·06 | 33·04 |
| 10 | 32·46 | 32·46 | 20 | 33·13 | 33·11 |

The corrections for both temperature and concentration are included in the formula—

$$S = \frac{P - I}{141\cdot84 + \frac{i}{20} - \frac{t}{2}}$$

P = the direct polarisation.

I = the observed polarisation of the inverted solution corrected proportionately to a normal solution.

P − I = K = the observed Clerget constant at $t^\circ$; *i* = the observed polarisation of the inverted solution in a 200-mm. tube without any correction.

Although the accuracy of Herzfeld's constants has been established by Strohmer, Heyer, and others, it appeared interesting to record the values obtained by me several years ago; these are also completely in accord with those of Herzfeld.

The sugar with which these experiments were made was prepared by shaking a concentrated syrup (obtained by dissolving the purest refined sugar in water, and concentrating the solution *in vacuo*) with absolute alcohol, collecting the precipitated crystals, washing them with alcohol, again dissolving them in water, and repeating the process. After being well washed with absolute alcohol, the sugar was spread out on a porous tile, and finally dried at a temperature between 60° and 70° C. Normal solutions (26·048 grms. in 100 c.c.) gave a mean reading in the polarimeter of exactly 100 divisions.

Instead of inverting 100 c.c. of a solution, as Herzfeld recommends, it has always been my custom to work with half that volume; and, as I shall show, the results are unaffected by this difference. The sugar (one-fourth the normal weight) is dissolved in 37·5 c.c. of water, and 2·5 c.c. of 38 per cent. hydrochloric acid added. The heating is conducted in the manner prescribed by Herzfeld.

| No. of Experiment. | C = Grms. per 100 c.c. | I *t*. | $I_{20^\circ}$ A. R. L. | $I_{20^\circ}$ (from the above table). | Difference between Columns 4 and 5. |
|---|---|---|---|---|---|
| 1 | 2 | $I_{16} = -33\cdot92$ | − 31·92 | − 31·91 | + 0·01 |
| 2 | 3 | $I_{17} = -33\cdot05$ | − 31·55 | − 31·98 | − 0·43 |
| 3 | 5 | $I_{16\cdot5} = -33\cdot92$ | − 32·17 | − 32·12 | + 0·05 |
| 4 | 7 | $I_{17} = -33\cdot90$ | − 32·40 | − 32·25 | + 0·15 |
| 5 | 10 | $I_{17} = -33\cdot90$ | − 32·40 | − 32·46 | − 0·06 |
| 6 | 13·024 | $I_{17} = -34\cdot10$ | − 32·54 | − 32·66 | − 0·12 |
| 7 | 13·024 | $I_{17\cdot5} = -33\cdot80$ | − 32·53 | − 32·66 | − 0·13 |
| 8 | 13·024 | $I_{16\cdot6} = -34\cdot20$ | − 32·47 | − 32·66 | − 0·19 |
| 9 | 13·024 | $I_{16\cdot5} = -34\cdot50$ | − 32·72 | − 32·66 | + 0·06 |
| 10 | 13·024 | $I_{16\cdot8} = -34\cdot28$ | − 32·65 | − 32·66 | − 0·01 |
| 11 | 13·024 | $I_{19\cdot6} = -32\cdot60$ | − 32·40 | − 32·66 | − 0·26 |
| 12 | 13·024 | $I_{19\cdot6} = -32\cdot88$ | − 32·68 | − 32·66 | + 0·02 |
| 13 | 13·024 | $I_{20} = -32\cdot44$ | − 32·44 | − 32·66 | − 0·22 |
| 14 | 13·024 | $I_{19\cdot4} = -32\cdot92$ | − 32·61 | − 32·66 | − 0·05 |
| 15 | 13·024 | $I_{19\cdot8} = -32\cdot72$ | − 32·62 | − 32·66 | − 0·04 |
| 16 | 13·024 | $I_{19\cdot6} = -32\cdot92$ | − 32·72 | − 32·66 | + 0·06 |

Taking the 11 experiments where the concentrations of the solutions were 13·024 grms. of sugar per 100 c.c., the mean experimental value for $I_{20}$ is − 32·61, instead of − 32·66, the value obtained by Herzfeld.

## A NEW MODIFICATION OF CLERGET'S METHOD, SPECIALLY APPLICABLE TO AFTER-PRODUCTS AND MOLASSES.

BY ARTHUR R. LING AND JULIAN L. BAKER.

Wilhelmy and, later, Ostwald have shown that the hydrolysis of saccharose in aqueous solution proceeds according to the well-known law of mass action of Guldberg and Waage, so that, for example with acids, the amount of hydrolysis in a given time is proportional to the product of the reacting masses.

The speed of the inversion of saccharose by acids, other conditions being constant, varies with the nature of the acid employed. Acids such as hydrochloric, sulphuric, &c., which according to theory exist in aqueous solution for the most part dissociated into their ions, hydrolyse much more saccharose in unit time than do weak organic acids; and, as a matter of fact, this same reaction has been employed by Ostwald as a means of determining the strength (affinity) of acids.

Now it is well known that the dark after-products with which are generally dealt with in the sugar industry, contain, besides sugar, other organic matters consisting to some extent—in all probability for the most part—of the salts of organic acids. At all events, a sufficiency of these salts is present to react with the hydrochloric acid added in carrying out Clerget's process, and to partially replace it by much weaker organic acids. The matter is not improved when the solution under investigation is previously clarified, as it usually is, with basic lead acetate, for here the hydrochloric acid is to some extent neutralised by the alkalinity of the basic lead acetate, besides which it is partially replaced by the comparatively weak acetic acid, and there is some danger that the inversion of the saccharose may not have reached completion in the prescribed time. To obviate the presence of an organic acid, it has been proposed by Herles (Zeit. Zuckerind. Böhmen, 1889, 557) to use a solution of lead nitrate in conjunction with one of sodium hydroxide as clarifying agent. The experiments of Herzfeld and others have shown, however, that the rotatory power of saccharose is diminished by the employment of this clarifying agent.

There is, however, another objection to the use of Clerget's method, as it is at present carried out, for the estimation of saccharose in products containing much "other organic matter." For, although the presence of a great many definite substances has been demonstrated in the juice of both sugar cane and beetroot (see this Journal, 1893, 533), the nature of this organic matter in a given sample of sugar or molasses cannot be determined. It is, however, well known that these substances are to some extent optically active, and experience shows that even after clarification with basic lead acetate some optically active substances besides sugar remain in solution.

As one of us has stated in the preceding paper, Clerget's method presupposes that no other constituents of the sample than the saccharose suffer any change in optical activity by the treatment adopted. But since the agent employed—acid—is a general hydrolyst, we can be by no means certain that this is the case. This objection will not count, of course, with those who are accustomed to speak of the "Clerget value," and who regard the method merely as an empirical one, capable of yielding good comparative results for purposes of buying and selling. But the sugar refiner, the distiller, and other technologists require something more than comparative accuracy if they are to use the results of chemical analysis to control their manufacturing operations. To them the method can have no value except as one which furnishes the percentage of saccharose in a given sample.

Now it is obvious that if, instead of employing a general hydrolyst such as an acid, we made use of one which would act selectively on saccharose only, the absolute accuracy of the method would be considerably enhanced, as would also its value, *pari passu* to the chemical manufacturer. Such a hydrolyst is the invertase of yeast, which hydrolyses, or to use the more familiar term, inverts saccharose and other substances having a similar configuration.

As you all know, saccharose may be regarded as an ether composed of residues of dextrose (*d*-glucose) and levulose (*d*-fructose), these residues being arranged in space in certain relative positions with respect to each other. The researches of Emil Fischer have shown that enzymes such as invertase probably act on substances of similar configuration, that is to say, substances in which the spacial arrangements of the various atomic groups within the molecule is the same. Fischer has produced by synthesis two methyl derivatives of glucose, one of which—the α-compound—contains a methoxyl group, arranged probably in the same relative position to the glucose residue as the levulose residue is arranged to the glucose residue in the molecule of saccharose. Both saccharose and α-methyl glucose are hydrolysed by invertase.

Now the only substance, the configuration of which is in any way similar to that of saccharose, which is known to occur in commercial sugar products is raffinose, a sugar which may be regarded as an ether of glucose, levulose, and galactose. In this substance the levulose residue is apparently combined with the remainder of the molecule in the same manner as it is in saccharose, since ordinary invertase hydrolyses it. Raffinose, as far as we know at present, only occurs in those products obtained from beetroot, and if it be true that it is hydrolysed by invertase, this in no way invalidates the method we are about to propose, since, when its presence is demonstrated, it will only be necessary to employ another formula, and the percentage of saccharose and raffinose may be calculated from the optical rotatory powers obtained before and after inversion, just as is done at present with the acid process. We make these remarks, however, with reservation, for Loiseau, and more recently Bau, have shown that the enzymes of high and low yeasts exhibit a different behaviour towards raffinose. It is, however, important to point out that both these chemists agree that the invertase of high yeasts hydrolyses raffinose to levulose and melibiose just as does the acid employed in Clerget's process; this requires the fixation of one molecule of water. The enzymes of low yeasts, on the other hand, are said by the same authorities to hydrolyse raffinose to galactose, glucose, and levulose (see this Journal, 1895, 54 and 80) which requires the fixation of two molecules of water. We will say no more on this point at present, as we intend to repeat the experiment of Loiseau, Bau, and others, in order to ascertain whether the method of inversion with high yeast can be made use of for the simultaneous estimation of saccharose and raffinose.

Our proposal is therefore to use invertase, or rather English high-fermentation yeast, as hydrolysing agent in carrying out Clerget's method. We are aware that our idea has been to some extent anticipated by other workers. Thus Kjeldahl (Res. du Comptes Rend. des trav. du Lab. Carlsberg, 1881, **1**, 192) showed that the saccharose in malt may be estimated by acting on the cold-water extract with yeast in presence of thymol, which latter arrests alcoholic fermentation; whilst some years afterwards C. O'Sullivan (J. Chem. Soc. Trans. 1886, 58) described a method for the same purpose, using invertase. In the year 1891, it was shown by O'Sullivan and Tompson (J. Chem. Soc. Trans. 1891, 46) that the addition of thymol was unnecessary in acting on sugar with yeast, as no alcoholic fermentation occurs at a temperature of 55° C. Both Kjeldahl and O'Sullivan used this method for determining small amounts of saccharose, the highest percentage of that sugar which the process was used to estimate being about 10 per cent. in a sample "treacle" (O'Sullivan and Tompson, *loc. cit.*). Such commercial products as beetroot molasses, however, frequently contain as much as 50 per cent. of saccharose together with a large amount of other organic matter, and we were anxious to ascertain if the method of inversion with yeast could be used to estimate the saccharose in these.

It was obviously necessary to ascertain if the value obtained for the rotatory power of invert sugar produced by the Herzfeld-Clerget's method was true for invert sugar obtained by the action of yeast. We were fortunate in securing from a sugar refinery three samples of refined sugar, which for convenience we may denote as A, B, and C. These were analysed by the usual method, with the following results:—

| —— | | | Specific Rotatory Power in Laurent Polarimeter C. = 15. |
|---|---|---|---|
| | | Per Cent. | |
| Sample A ..... | Saccharose........ | 99·910 | $[\alpha]_D = 66{\cdot}43$ |
| | Ash........ | 0·033 | |
| | Water ............ | 0·050 | |
| | | 99·993 | |
| Sample B ..... | Saccharose........ | 100·000 | $[\alpha]_D = 66{\cdot}53$ |
| | Ash................ | 0·013 | |
| | Water ............ | 0·027 | |
| | | 100·040 | |
| Sample C ..... | Saccharose........ | 99·920 | $[\alpha]_D = 66{\cdot}50$ |
| | Ash................ | 0·003 | |
| | Water ............ | 0·017 | |
| | | 99·940 | |

Now it was certainly to be expected that Herzfeld's value for the rotatory power of invert sugar would also be obtained when the inversion was carried out with yeast. Having regard to the purity of the three samples above mentioned, which are, however, merely typical refined sugars, we decided not to attempt any purification, but merely to dry off a portion used for experiment at about 50° C.

The method adopted is as follows:—The quarter-normal weight (6·512 grms.) of sugar is rinsed into a 50-c.c. measuring flask with about 20 c.c. of water. The sugar is dissolved, and the temperature of the solution raised to 55° C., after which about 0·5 grm. of fresh pressed and washed brewery yeast is added. This is best done, when several experiments have to be carried out, by suspending a known weight of the yeast in a definite volume of water, and addding a certain amount of this suspension to the sugar solution by means of a pipette. We have satisfied ourselves, by conducting blank experiments, that no optically active substances pass into solution during the digestion of the sugar solution with the yeast. The inverted solutions were polarised in a water-jacketed 200 mm. tube at a definite temperature, which was determined by means of a thermometer reading to 0·1° C. When the readings were made at any other temperature than 20° C., they were duly corrected to this (see preceding paper).

| Experiment. | Sample used. | Inversion Constant $I_{20^\circ}$ | Remarks. |
|---|---|---|---|
| 1 | A | $-32{\cdot}67$ | Fresh yeast. |
| 2 | A | $-32{\cdot}52$ | " " |
| 3 | B | $-32{\cdot}57$ | " " |
| 4 | B | $-32{\cdot}50$ | " " |
| 5 | C | $-32{\cdot}67$ | " " |
| 6 | C | $-32{\cdot}67$ | " " |
| 7 | A | $-32{\cdot}47$ | " " |
| 8 | A | $-32{\cdot}47$ | " " |
| 9 | A | $-32{\cdot}46$ | Yeast 2 days old. |
| 10 | B | $-32{\cdot}64$ | " " " |
| 11 | B | $-32{\cdot}48$ | Yeast 6 days old (liquefied). |

The mean of these 11 experiments gives an inversion constant of $I_{20} = -32{\cdot}56$, instead of $-32{\cdot}66$, as observed by Herzfeld. Bearing in mind the fact that the experiments were made with refined sugar without any subsequent purification, we consider we are justified in adopting Herzfeld's formula for calculating the results, the more so as one of us, using sugar purified by treatment with alcohol and inverting with acid, has confirmed Herzfeld's value (see preceding paper).

We will now record same values obtained with commercial samples, and we may state that of these we have selected only those referring to dark products, in which it is impossible to obtain concordant values by the ordinary process with acid. We at first attempted to act on solutions which had been previously clarified with basic lead acetate, filtered, and rendered slightly acid with acetic acid, but we found that the presence of lead entirely arrests the invertive action of the yeast. All our experiments were made therefore on unclarified solutions.

The ¼ normal weight is dissolved in about 20 c.c. of water, the solution rendered slightly acid with acetic* acid if necessary, heated to 55° C., and 0·5 grm. of yeast added. At the end of 5 hours, basic lead acetate and a little alumina are added, the solution diluted to 50 c.c., and polarised in a 200 mm. tube at a known temperature. We are aware of the objections against clarifying a solution of invert sugar with basic lead acetate, and a better plan would probably be that recommended by Herzfeld (Zeit. Ver. Rübenzuck. Ind., 1890, 208), namely, to clarify the solution by the addition of 0·38 grm. of extracted charcoal after making it up to the mark, a correction being applied for the absorptive power of the charcoal in the time it remains in contact with the solution, by means of a factor ascertained by a separate experiment with a solution of pure invert sugar.

Having regard to the nature of these products, all of which, with the exception of the last sample of "cane molasses," were *very* low grade samples, we think we are justified in concluding that the agreement between the duplicate determinations is satisfactory. The fact that the values obtained with acid do not agree with those obtained with yeast, may, we contend, be taken *a priori* to be due to the defects already pointed out in connection with the former process.

| Description of Sample. | Direct Polarisation, P. | Clerget (Acid). | | Clerget (Yeast). | |
|---|---|---|---|---|---|
| | | Inversion Value $I_{20}°$ | Saccharose. | Inversion Value $I_{20}°$ | Saccharose. |
| Beetroot molasses ............ | 51·5 | −14·00 | 49·0 | −14·83 | 50·10 |
| Same sample ............ | 51·8 | −14·29 | 49·7 | −15·30 | 50·60 |
| Jaggery ............ | 72·9 | .. | .. | −18·12 | 68·60 |
| Same sample ............ | .. | .. | .. | −17·38 | 68·10 |
| Egyptian molasses............ | 28·3 | .. | .. | −16·20 | 33·54 |
| Same sample ............ | .. | .. | .. | −16·00 | 33·39 |
| Egyptian molasses............ | 28·3 | .. | .. | −16·90 | 34·07 |
| Same sample ............ | .. | .. | .. | −16·50 | 33·80 |
| Egyptian molasses............ | 28·4 | .. | .. | −15·94 | 33·40 |
| Same sample ............ | .. | .. | .. | −15·42 | 33·10 |
| Cane molasses†............ | 42·4 | −15·76 | 43·8 | −14·92 | 43·20 |
| Same sample ............ | .. | .. | .. | −14·90 | 43·19 |

† Mr. A. C. Halse, to whom we are indebted for this sample of molasses, finds with acid, saccharose = 43·80 per cent., thus corroborating our value.

In conclusion, we may add that although the yeast inversion process extends over more time than the ordinary Clerget method with acid, the former needs less attention; and where a great many determinations have to be done, less of the operator's time is taken up.

### Discussion.

Dr. G. H. **Morris** had listened with great interest to the papers, and regretted that the authors appeared to have confined their researches to the action of invertase on sugar, and had not mentioned the action on it of the salts of the organic acids and other matters which were always contained in molasses. It was known that enzymes were easily influenced by foreign substances, and it was quite conceivable that the substances sometimes present in molasses might have some influence upon the invertase of the yeast, which influence would affect its action, and lead to an incorrect result.

Mr. B. E. R. **Newlands** suggested that Mr. Ling should put before the meeting the results, if any, obtained from black molasses. There was but little difficulty, when working with light-coloured solutions, in getting results which agreed with Clerget's value; the trouble was to get them to agree when one had dark materials, such as Egyptian molasses, to deal with, entailing the use of defecating agents.

Mr. John **Heron**, while recognising the interest of the paper before the meeting, felt bound to differ from the opinions and conclusions expressed by the authors. In the first place he disagreed with the title. Clerget's process consisted in what was known as double polarisation of sugar, *viz.*, polarisation before and after inversion. To him it appeared to be quite immaterial how, or by what means, inversion was produced. He therefore failed to see how the authors' process could be called a modification of Clerget's. Next, the yeast process was very objectionable in some cases and very faulty in others. Those accustomed to deal with raw sugars knew that many of them were alkaline in character, and in such a case inversion by yeast was uncertain. Then again, the process was a very slow one, taking at least five hours; and, as bearing on this point, it was to be remembered that according to the views of O'Sullivan, Tompson, and others, it was desirable to allow samples to lie until the following morning and then polarise again to be quite sure of the results. Now, in dealing with a large number of samples, it would be almost impossible to carry out this slow yeast process. On the other hand, one could invert with acid in a very short time, and, by paying careful attention to the instructions laid down by Herzfeld (every word of which he could fully confirm), it was quite possible to get as accurate a determination of cane sugar as by inversion with yeast. The only remaining question was that of molasses; and with regard to that, he would like to know if the authors had actually separated out any of those dark bodies, and if they knew whether acid, in the proportions in which it was used in this process, as improved by Herzfeld, had any action on those bodies, and, if so, what was the nature of that action. In an article which he had himself contributed to Thorpe's Dictionary of Applied Chemistry, Vol. III., p. 656, he had there described the process of Clerget and Herzfeld's improvement of it, and in connection therewith went on to say, "instead of carrying out the inversion of cane sugar by means of acid, O'Sullivan and Tompson's invertase process (Journal Chemical Society, Vol. 59, p. 46) may be employed," &c.

The authors admitted that their method had been anticipated by others, and if Mr. Ling would look up the bibliography of the subject, he would find that the anticipitation had gone further than he seemed to be aware of. Nevertheless the paper was interesting, inasmuch as it at least constituted a very careful *résumé* of the whole subject, from which point of view it would be very useful.

Mr. J. L. **Baker** remarked that since the writing of the paper, they had carried out an inversion of pure raffinose with yeast. The number obtained was in accordance with the well-known raffinose formula of Herzfeld, that is to say, the inversion apparently stopped at the first stage in the hydrolysis of raffinose, namely, that in which melibiose and levulose were produced.

Mr. **Ling** (who had given but a brief account of the papers) said, in reply to Dr. Morris, that he and his colleague had made no direct experiments to ascertain whether the organic substances contained in the after-products had any action in arresting or slackening the invertive power of invertase or yeast. They had found, however, that when these organic substances were precipitated, to the greater part, with basic lead acetate, the rotatory powers of the acidified filtrates were entirely unaffected on treatment with yeast in the manner described. The presence of lead salts, in fact, entirely arrested the invertive action of the yeast, but when these salts were removed by precipitation, inversion by yeast proceeded as usual. He believed that the salts of some of the heavy metals behaved similarly to those of lead, and this was doubtless due to the precipitation of certain proteïd substances which formed the vehicle of the active enzyme.

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* We would insist on the necessity of acidifying the solution, when necessary, with an acid which is known to exert no retarding influence on the hydrolytic function of invertase; such an acid is acetic acid. In the absence of data as to their action, we have avoided the use of stronger mineral acids.

In answer to a request by Mr. Newlands, Mr. Ling wrote on the black-board the Clerget values, obtained with dark molasses, given in the paper.

Mr. Heron had said that he did not regard the process they had described as a modification of Clerget's method at all, the principle of which was double polarisation before and after inversion, and nothing else. In reply to this, he would repeat what had been stated in the paper, namely, that although formulæ had been constructed for the estimation of small quantities of saccharose by inverting with yeast, no one had ever applied the yeast inversion method for the estimation of comparatively large quantities of saccharose, such as Clerget's method was originally devised for. The point which they claimed to have established was that the formula elaborated by Herzfeld for the estimation of saccharose, by a modification of Clerget's acid process, also held good when yeast was used as the inverting agent. They therefore failed to see how their process could be regarded as anything else than a modification of Clerget's. In fact, working with a Schmidt and Haensch half-shadow polarimeter, they had found that when half-normal solutions of pure saccharose were inverted by yeast in the manner they had described, and subsequently polarised in a 200-mm. tube at a constant temperature, the results, multiplied by two and corrected, if necessary, to the standard temperature, agreed very closely with Herzfeld's value, $I_{20} = -32 \cdot 66$. Then Mr. Heron said *he* had suggested inversion by yeast in carrying out Clerget's process. He (Mr. Ling) believed that a mere statement to that effect was to be found in the article quoted, but no attempt had been made to justify it by experimental evidence. The yeast process was specially adapted for cases in which a large number of determinations had to be carried out, for, as they had stated in their paper, less of the operator's time was demanded than with the acid method.

With regard to the disparity in the duplicate results obtained with dark products when the inversion was carried out by acid, that was doubtless due to the action of the acid on the dark bodies present, some of which were optically active. All this was discussed in the paper. Their point was, that in yeast they had a selective hydrolyst, which acted only on the saccharose (and also raffinose when present), and therefore complied with Clerget's presupposition that the rotatory power of no other constituent should be affected.

## NOTE ON THE ESTIMATION OF MOISTURE IN INVERT SUGAR.

BY L. T. THORNE, PH.D., F.I.C., AND E. H. JEFFERS.

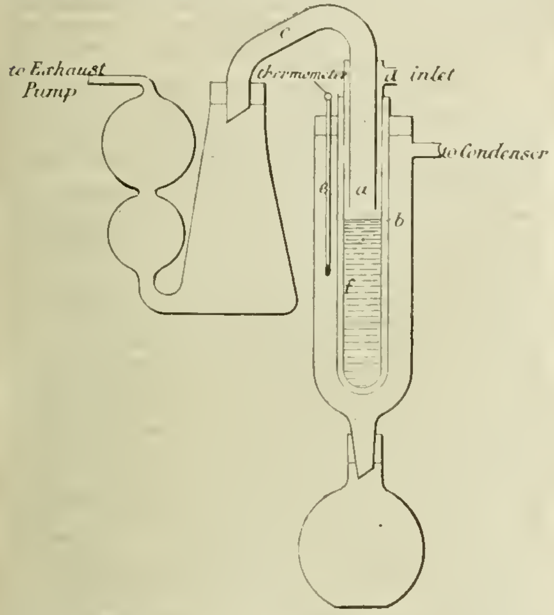
THE direct estimation of the water in sugars is both tedious and, owing to the unstable and easily oxidisable character of many of the sugars, very uncertain. The estimation is therefore generally deduced from the gravity of a 10 or 20 per cent. solution of the sugar. In the ordinary text-books the method given is to determine accurately the gravity of a 10 per cent. solution at 60° F., and to divide the excess gravity over that of water (taken at 1000°) by 0·386, this latter being the average increase of gravity due in a 10 per cent. solution to every 1 per cent. of pure sugar in the sugar taken. In the case of highly refined cane sugars, where the mineral matter is almost negligible, this method gives approximately the correct percentage of total solids, the difference between that number and 100 giving of course the percentage of water present. But Heron has pointed out (Journal of Federated Institutes of Brewing [1896], II., 450) that whilst the factor 0·386 approximately represents the excess gravity due to the sugar, that due to mineral salts is much higher, and that consequently a correction is necessary with sugars containing more than a few tenths of a per cent. of mineral matter. He dissolved the ash obtained from various sugars and found the excess gravity for each one gramme dissolved in 100 c.c. of water to be 8· as against the 3·86 due to sugar. He therefore proposed to deduct from the original excess gravity 0·8 time the ash, and divide the remainder by 0·386 to obtain the percentage of total organic matter. Adding to this the percentage of ash, the difference from 100 then gives the percentage of water.

Heron's correction has been adopted by some analysts, but not by others, who contend that his figures deduced from the ash do not necessarily represent the values of the mineral matter as it exists in the sugars. The result is that great discrepancies in total solids and water occur in analyses of sugars by different analysts. This is especially the case with invert sugars, where, on the one hand, there is always an appreciable amount of mineral matter, and on the other, checking by direct determination is especially difficult, owing to the exceptional instability of the levulose therein. These discrepancies amount to slightly more than the percentage of ash present. Thus, in an invert sugar containing 3 per cent. of ash, the total solids found by an analyst not using Heron's correction, would be slightly more than 3 per cent. higher than that found by one adopting the correction, and of course the water would be correspondingly lower. Of the total solids, the invert and cane sugars, ash, and albuminoids are more or less directly determined, and the residual amount to make up the total solids is classed as "other carbohydrates," "inert matters," or some similar term, and consequently the whole of the error in the determination of total solids or water falls on this class. Thus in the example given, on identical analytical data the "inert matter" might be given as 0·1 or 3·1 according to whether the calculation of solid matters were made with or without the correction for ash—a result which is highly unsatisfactory.

We therefore thought it desirable to endeavour to obtain accurate and reliable direct determinations of the water in various invert sugars in order to ascertain definitely what correction was required in deducing the total solids and moisture from the gravity of a 10 per cent. solution.

Many methods were tried before accurate and constant results could be obtained, but finally constant, concordant, and we believe very closely correct results were obtained by the following modification of the methods hitherto used.

A thin tube (*a*) about 15 mm. in diameter and 85 mm. long, and closed at one end (a part of a test tube answers well), is taken, and into this is fitted a strip of filter paper (*f*) about 60 mm. wide, coiled round a small glass rod. The coil should be of such a size as to fit freely, but not loosely, into the tube. This tube then slips easily into a test tube (*b*), about 20 mm. wide and 150 long, a small wire carrier being so arranged that the inner tube does not quite touch the bottom of the outer one. This larger tube forms the heated chamber. An inlet (*d*) and outlet tube (*e*), the latter as wide as possible, are fitted to the tube by an air-tight joint, so as to allow a current of any gas to be passed through it. This larger tube (*b*) fits to about five-sixths of its length into a cylinder (*c*) serving as a vapour jacket, and this is connected at its lower end to a boiling flask and at its upper end to an inverted Liebig condenser. This vapour jacket might, of course, if preferred, be replaced by a water jacket kept at a regulated temperature. The liquid first used in the flask was water, but the temperature thus produced was found to be too high, and methylated spirit was substituted. With the latter substance the temperature of the inner tube could easily be maintained constant at between 65° and 70° C. It was still found that neither in air nor in an ordinary vacuum (containing, of course, a little residual air) could constant results be obtained. By washing out with an inert gas and then evacuating, better results were obtained, but the method was very slow and still not quite satisfactory. It was then decided to pass through the inner vessel a current of inert gas—various gases were tried and carbonic abydride eventually adopted as most convenient. The cocks are so arranged that only a *very* small quantity of gas can pass into the chamber, whilst the outlet is freely open to a powerful and constantly working exhaust pump. The carbonic anhydride is, of course, carefully dried through sulphuric acid before entering the tube, and passed out through a sulphuric acid absorbing flask (*g*) offering a large surface of acid. In this way the inner vessel (*b*) contains a highly rarefied atmosphere of carbonic anhydride, of which the pressure does not exceed 50—80 mm. of mercury, whilst a current of dry carbonic anhydride still passes continuously through it.



The tube and paper are kept in this apparatus till the weight becomes constant, which occurs in about four to five hours. About 1 to 1·5 grms. of the sugar to be analysed are then quickly weighed into the tube on to the end of the coil of paper, care being taken, by keeping the tube tilted, that the sugar only covers about half the end of the coil. About 1 c.c. of boiling water is then dropped on to the *uncovered* part of the roll and 2 or 3 c.c. on to the sugar. This is sufficient to liquefy the sugar and distribute it evenly over the greater part of the paper coil without allowing any of it to reach the bottom of the roll or accumulate on the bottom or sides of the tube. If the whole of the end of the coil of paper is covered with the sugar, it is found less easy to efficiently dissolve and distribute it, and the addition of the 1 c.c. of water to the paper before solution of the sugar not only much facilitates the even distribution over the paper, but also obviates the fear of enclosed bubbles of air, which are liable to cause spurting when the tube is put into the heated chamber and the pressure removed. It is necessary to add a minute trace of ammonia to neutralise any acid present, as the smallest trace of free acid prevents the obtaining of constant results by causing some decomposition. The tube is then re-introduced into the apparatus and weighed at intervals until constant. About 6—10 hours is sufficient, and further heating causes absolutely no change in weight. If, however, the least air is admitted, further reduction in weight occurs, decomposition commences, and constancy cannot again be obtained. Decomposition similarly sets in if the temperature is much raised (even below 100 C.), and to obtain correct results *both* an atmosphere of inert gas and a a temperature not exceeding 70° C. are essential. Many determinations of various invert sugars were made, and in most cases two or three separate determinations of the same sugar. These latter were very concordant, not varying from one another more than 0·2 per cent.

In the appended table the results of a few typical examples are given, and are compared with determinations deduced from the gravity of a 10 per cent. solution, both with and without correction. In applying Heron's correction, the question of ash is, of course, an important one; and here, again, discrepancy of custom occurs, for whilst almost everyone uses the sulphate process for determining ash, some take the weight of the sulphated ash found as representing the true mineral salts present in the sugar, whilst others deduct one-tenth from the weight of the sulphated ash. From the results of our experiments just described, we believe that a more correct plan would be to adopt a middle course, and deduct *one-twentieth* from the sulphated ash. We have adopted this in our calculations, with Heron's factor of 0·8, and with this combination certainly find the results more concordant than with those obtained by any other combination of factors, though we have tried taking sulphated ash and $\frac{9}{10}$ sulphated ash with factors from 0·72 to 0·8. In the table, *a*, *b*, *c*, and *d*, are ordinary commercial invert sugars, *e* is an invert specially prepared with very high ash, *f* and *g* are cane sugars simply taken for comparison; *f* was a syrup made from the purest crystallised sugar, and was very difficult to handle, owing to its very great tendency to crystallise. This probably accounts for the somewhat wide divergence between the determinations; *g* was a sample of molasses specially prepared from raw Java sugar so as to contain a high percentage of ash.

In calculating the inverts, the corrected density factor (3·888 for this density) lately given by Brown, Morris, and Millar (Chem. Soc. Journ. 1897, 1., 80) was employed; for the cane sugars the ordinary factor 3·86 (which also agrees with these found by Brown, Morris, and Millar for cane sugar).

| | Sulphated Ash. | Corrected Ash. | Density of 10 per Cent. Solution. | Water deduced from Density of 10 per Cent. Solution. | | Water by Direct Determination. | Difference of Direct from Corrected Indirect Determination. | Difference of Direct from Uncorrected Indirect Determination. |
|---|---|---|---|---|---|---|---|---|
| | | | | Uncorrected. | Corrected. | | | |
| *Invert Sugars.* | Per Cent. | Per Cent. | | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| *a* .................... | 0·48 | 0·46 | 1032·88 | 15·43 | 15·93 | 15·97 | + 0·04 | + 0·54 |
| *b* .................... | 2·04 | 1·94 | 1033·12 | 14·81 | 16·86 | 17·11 | + 0·25 | + 2·30 |
| *c* .................... | 3·02 | 2·87 | 1033·14 | 14·77 | 17·81 | 17·69 | − 0·12 | + 2·92 |
| *d* .................... | 3·67 | 3·49 | 1033·13 | 14·79 | 18·48 | 18·28 | − 0·20 | + 3·49 |
| *e*.................... | 6·03 | 5·73 | 1030·62 | 21·24 | 27·29 | 26·50 | − 0·79 | + 5·26 |
| *Cane Sugars.* | | | | | | | | |
| Pure cane sugar....... | 0·07 | 0·07 | 1026·64 | 31·05 | 31·13 | 30·57 | − 0·56 | − 0·48 |
| Java molasses ........ | 6·00 | 5·70 | 1034·23 | 11·44 | 17·54 | 16·67 | − 0·87 | + 5·23 |

From these results it is clear that when the determination of water and total solids in a sugar is to be made by deduction from the gravity of a solution of known strength, a correction for ash must be made if the result is to be considered in any way reliable; but that, with a proper correction, this method may safely be relied on to give results very approximate to the truth. We have not been able to find a correction which shall give exact correspondence with all the direct determination results, but the divergence is only at all appreciable when the ash is very high. And it is in these sugars with abnormally high ash that the process of direct determination, above described, may become faulty. For in these sugars, it is quite probable that hygroscopic salts (such as sodium sulphate, alkaline carbonates, and perhaps calcium chloride, &c.) present in more or less quantity, prevent the removal of the last traces of moisture under the conditions and at the temperature employed. It is thus possible that in these cases the direct determination of water comes out rather too low, and that the calculated number is actually nearer the truth than the direct determination.

In conclusion, we would suggest for ordinary commercial analyses the adoption of $\frac{19}{20}$ of the sulphated ash as the true mineral matter, and 0·8 as the correction for ash in the density determinations. And we believe that the process described will prove an easy and useful means of checking the calculated results by direct determination where this is thought desirable.

### Discussion.

Mr. John Heron said that he was pleased that the authors had investigated this matter, and that to a large extent the result had been a confirmation of the figures adopted by himself. The reason that he had adopted 8 as the factor for ash was that, after having saved the ash of invert sugars of several years' working in the factory, he dissolved that ash in water as well as in pure cane-sugar solutions, and the result of a large number of experiments carried out in this way proved to him that 8 was the correct factor, and this conclusion was supported by the gravity tables given in various works for sulphate of potash, sulphate of lime, and sulphate of soda. It was obvious, he thought, that in the case of invert sugar, where sulphuric acid was used for the purpose of inversion, that when the inversion was completed, most, if not all, of the mineral salts occurring in the sugar would exist as sulphates. If that were so, then in determining the ash of a sample of invert sugar (the salts being considered as sulphates), he did not see why any correction should be made on the ash. In working out that idea he had come to the conclusion that it would be nearer the mark to take the sulphated ash as the true ash occurring in the sugar than to apply any correction. That idea was supported by the fact that if one determined the different organic salts in raw sugars and the proportions in which they occur, one would find that their molecular weight would correspond more closely with the molecular weight of sulphate of lime than with that of carbonate of lime.

Mr. A. R. Ling wished to congratulate the authors on the very ingenious apparatus they had devised. Messrs. Brown, Morris, and Millar had employed a modification of Lobry de Bruyn's apparatus in their recent well known researches to dry various sugars. Dr. Frew had suggested a method of determining the solid residue in beer by absorbing it by means of bibulous paper, and subsequently drying in a current of air under diminished pressure at 110—120 C. (see this Journal, 1897, 313). With regard to the deduction of one-tenth from the sulphated ash, that was generally done in the case of beetroot sugars, in which it had been found to give results approximating the truth. When other products were dealt with, however, it was probable that the correction would require modification.

Dr. G. H. Morris desired to congratulate the authors on solving a problem which had troubled many analysts who had tried, but hitherto without success, to determine satisfactorily the amount of moisture in commercial invert sugars. He had recently attempted to do the same thing with the apparatus to which Mr. Ling had referred, but, in spite of the most careful working, had found it impossible to dry commercial sugars without such decomposition occurring as rendered the results obtained absolutely worthless. He considered the modification described by the authors a very valuable and interesting one.

Mr. J. A. R. Newlands said that with regard to the determination of moisture in sugars, it had formerly been usual to adopt the plan of mixing with sand and alcohol. That method, however, failed entirely when applied to bodies containing levulose, on account of its unstable nature. Some months ago, after careful examination, he had concluded that the plan proposed by Mr. Heron was worthy of adoption, and had carried it out in his own practice, though with some difficulty, because he was often confronted with the results of other chemists showing a difference of even 2 or 3 per cent. It was, however, satisfactory to find that the method was now established on a firm footing, and he congratulated Dr. Thorne on the able and unassuming manner in which he had brought forward his results, and also Mr. Heron on the fact that his system of working had been substantially corroborated.

Mr. Alfred C. Chapman observed that in dealing with raw cane sugars, unless one knew the specific gravity of the organic salts actually existing in this sample, one could not apply the specific gravity ash correction with certainty in the determination of moisture in such sugars.

Dr. L. T. Thorne, in reply, said that the examples of cane sugar referred to in the paper were only given as showing that the results in that case were approximately on the same lines as in the case of invert sugar. They did not claim the application of the factors given except to invert sugars. With regard to Mr. Heron's point about sulphated ash, he quite admitted the justice of his argument up to a certain point, but was inclined to doubt whether going over the char does not modify the sulphates present in the sugar. The liquor, after leaving the char, certainly sometimes contains other salts than sulphates. They had compared the results obtained by this process with the results calculated from the solution densities when correcting on the basis of the sulphated ash and all factors up to 0·85, and could obtain no results so concordant as when they adopted the ash standard as $\frac{19}{20}$ of sulphated ash and the 0·8 factor. They did not claim novelty in the apparatus, but merely the slight modifications they had made, and which they considered improvements. They did emphasize, however, the need for the combination of a very low temperature with as nearly a vacuum as could be got with a current of inert gas passing through.

## AN IMPROVED DRIP-PROOF BUNSEN.

BY THOS. FLETCHER.

(*See also this Journal*, 1897, 395 *and* 863.)

The forms which have been recently introduced are more or less a return to forms which have been tried and discarded many years ago. When a tube bunsen is used for dirty work, it will be found that the dirt is as much in evidence on the inside of the tube as anywhere else; and, consequently, if the gas comes in at a side slit there is liability to choke, and also unusual difficulty in cleaning and readjusting. The makers of laboratory bunsens have also learnt by experience that a fine slit for the gas inlet is inadmissible in practice, as it becomes choked by the dust in the air, and it must be remembered that any departure from one straight line in the tube, or direction of the gas jet, means a lower temperature of flame. Where a bunsen is used for very dirty work, or where there is a liability of breakage of the vessel, the burner can be perfectly protected by simply tilting it over sideways towards the side opening. Anything which falls runs down the side of the tube and escapes by the side opening without coming near the jet, and the temperature of the flame is not interfered with by bends or departures from the one straight line. It is an easy matter to mount the tube with a small swivel between it and the stand, or any other makeshift, and any maker can easily supply these at a small extra cost. The worst possible work for an open bunsen is the melting of wax for sealing bottles, &c., and experience has proved for this work that a horizontal tube is the best, and never causes any trouble, even in the most careless hands. The fixed vertical burner has become universal, not from its advantages, but because it is the cheapest possible form which can be made. I have myself never used any unless mounted on a swivel, to admit of the angle being varied to any required extent.

# Newcastle Section.

*Meeting held on Thursday, January 27th, 1898.*

MR. JOHN PATTINSON IN THE CHAIR.

## FLOCCULATION AS AN AID IN LABORATORY WORK.—PART II.

BY R. GREIG SMITH, M.SC.

(*See also this Journal*, 1897, 871.)

In the short discussion that followed the reading of my paper, the remarks tended towards the reason of flocculation, that is, to the mechanism of the process. No doubt this is connected with the use of flocculating agents; and although I did not intend to speak of this side of the question, yet a short *résumé* of some previous papers on the subject may not be without interest.

The first to write about the matter appears to have been W. Skey, who, in an article in the *Chemical News* (XVII., 160, 1868) upon "The Coagulation and Precipitation of Clay by Neutral Salts generally," calls attention to the fact that the salts used for the clarification of sewage and purification of water depended upon the formation of a bulky precipitate, which enclosed suspended particles and carried them down with it in the settling tanks. There were other salts, however, which, although not forming gelatinous precipitates, had the power of coagulating suspended particles. The chlorides of the alkalis and of the alkaline earths, sulphate of soda, and sulphuric acid acted in this way. The coagulating powers of these substances were different, and when the salt was removed by washing, the sediment remained suspended on being again shaken up with water, The volume of water relative to the amount of salt employed was of primary importance, while the amount of clay suspended was only secondary. From several considerations he concluded that these salts acted solely on account of their affinity (stronger than that of clay) for water. In this respect they were like certain other substances which, although soluble in water, were precipitated by others possessed of a superior affinity for the solvent; for example, Prussian blue is precipitated by salts generally, silica dissolved in ammonia by ammonium chloride, and barium nitrate by nitric acid.

Six years afterwards, in 1874, Durham read a paper before the Royal Soc., Edin. (extracted in the *Chemical News*, August 1874), in which he instanced cases of sedimentation by certain salts, acids, and alkalis, and further showed that when using comparatively strong solutions of reagent, sedimentation varied inversely with the percentage of acid and directly with the percentage of alkali. Simultaneously, Dr. Storey Hunt read a paper before the Nat. Hist. Soc. of Boston, U.S.A. (see letter by "T. R. O.," *Chemical News*, XXX., 97, 1874), upon the sedimentation of Mississippi mud by salts and acids. He considered that the mechanism was explained by Guthrie's researches (Proc. Roy. Soc. XIV., 1864) upon drops of water. Falling from a ball of ivory, drops of a 0·8 per cent. solution of calcium chloride, for example, were one-ninth smaller than drops of pure water, and from this the conclusion was arrived at that salts reduce the cohesion, that is the viscosity, of water. This is undoubtedly the case with some flocculating agents (as ammonium chloride), but not with others (as zinc sulphate); and, furthermore, the microscope shows that the particles before flocculation are separate, but after flocculation they are aggregated into groups.

Ramsay showed (Proc. Roy. Geol. Soc. 1876, 129) that those salts which absorb most heat in going into solution, cause suspended mud to flocculate most rapidly. He considered that, owing to the absorption of heat, the liquid particles vibrate faster, and the particles of solid have in consequence a better chance of gravitating without meeting the resistance of impact. Sedimentation was also found to vary according to the specific gravity of the salt solution and to the temperature.

The most noteworthy contribution, however, is by Durham, who, two years afterwards (*Chemical News*, XXXVII., 47, 1878), described a number of experiments with solutions of salts, acids, and clay particles, and from their behaviour arrived at the following conclusions. "1st. There seems to be a regular gradation of chemical attraction from that exhibited in the suspension of clay in water up to that exhibited in the attraction of sulphuric acid for water, which we call chemical affinity. 2nd. That chemical combinations, solutions, and suspension differ only in degree, and are manifestations of the same force; and definite chemical compounds are just the points where the affinity exerts force enough to hold an exact number of atoms with a certain strength. 3rd. The attraction of chemical affinity is not in every case, at least, exhausted when a definite chemical compound is formed, but has sufficient power left to form solution or suspension compounds."

This hypothesis contends that not only have we chemical affinity such as that exhibited by an anhydrous base for an anhydrous acid, but we also have "residual affinity," which may be exerted by substances upon water to form solution compounds—as the crystallo-hydrates colloidal solution, and suspension.

The formation of aggregates of solid particles can be explained, as Whitney has done (Wiley's *Agricultural Analysis*, I., 172), by surface pressure. The molecules at the surface of a solid particle suspended in water have, within their sphere of influence, a greater attraction for molecules of water than these have exerted upon them by other water molecules; consequently, more molecules of water gather at the surface of the solid particle than at any other place in the solution. There is in consequence a surface pressure, and the compressed layer of molecules of water prevents any two particles of solid approaching or adhering. Should, however, a flocculating agent be dissolved, it will, by its superior attraction for the molecules of water, withdraw them from the surface of the solid, thus lessening the surface pressure; and it may be lessened so much that, from being a pressure, it becomes a tension which, being exerted on all the surfaces, causes the particles to aggregate into groups.

Should the aggregation of solid particles be sufficiently explained by surface pressure, then could we not also explain precipitation by the surface pressure and tension upon a molecule or molecular system of any substance in solution? Surface pressure or surface tension is another expression for molecular attraction or chemical affinity of a weak type, and appears to be identical with residual affinity. When the surface pressure is relieved, or the molecular influence overpowered—that is to say, when the molecules of water are withdrawn from the influence of the residual affinity—the molecules of the substance about to precipitate, aggregate, and when the molecular aggregates become sufficiently large they settle out as a precipitate. Heat appears to annul the solution or residual affinity just as it lessens surface tension and pressure. Ramsay has shown that sedimentation varies with the temperature, and, besides, we know that gelatinous precipitates become less gelatinous on heating. This contraction has been shown to be due to dehydration. According to Wiley, ferric hydrate, which, when precipitated cold and air-dried for 18 days, contains 38 per cent. of water, is converted by boiling for some hours from a bulky red-brown precipitate into a bright red substance containing only 2 per cent. The partial dehydration of aluminium hydrate from $Al_2O_3, 5H_2O$ to $Al_2O_3, 2H_2O$ by prolonged boiling has also been observed. Furthermore, when clay is boiled for some days, the individual particles become aggregated into small, rounded, opaque, brown granules, which, when pressed, are seen under the microscope to break up into a multitude of very fine particles (Wiley, *loc. cit.* pp. 244–5).

While, with precipitation and coagulation generally the separating out of the molecular aggregates may be said to be caused by the superior residual affinity of the coagulating substance, yet it is a case of give and take; the precipitate exchanges the water molecules over which it had influence for a portion of the base of the flocculating agent. That the residual affinity is small compared with the affinity of a metal for an acid radicle, goes without saying; still it is quite sensible. Durham assumed it might be sufficient to raise the atomic weight of calcium from 40 to 40·4, and Messrs. Picton and Linder found that about a gramme of arsenious sulphide retained firmly 2·4 mgrms. of barium. The latter case was one of colloidal solution, where the substance was just on the point of precipitating. By the molecular pressure hypothesis, the residual affinity may, in great part, be exerted among the molecules of substance themselves instead of upon the molecules of water, but eventually in the colloidal solution we have molecular aggregates holding tenaciously the last molecules of water. Part of this water is attracted by the flocculating agent and part is replaced by the flocculating base. This gradation of attraction for water is general with chemical substances. It is exhibited in anhydrous acids, as sulphuric and phosphoric which form numerous hydrates with water, the last few molecules of which are attracted with strong affinity, while to eliminate the last molecule of water a base is necessary. The same differences are to be seen in the crystallo-hydrates, where generally the last molecules of water are very firmly held. Again there is the affinity of neutral substances like alcohol for water.

These ideas regarding the cause of flocculation are associated with the hydrate theory of solution, among the supporters of which are Pickering, Mendeléef, Fitzgerald, and Armstrong. The theory is that when a soluble substance is placed in water, the molecules of water are attracted by and surround first the molecular aggregates and finally the individual molecules in gradually increasing zones until each molecule is the centre of a water system somewhat like a solar system. Diffusion becomes, not a distinct phenomenon, but a phase of solution; the molecules of a salt or saline body spread or diffuse on account of new molecules of water inserting themselves between and joining themselves on as parts of the systems. It is evident, then, that coagulation is the reverse of solution, and is similar to crystallisation, since both result from the withdrawal of water from the systems. The water systems are supposed to be exceedingly unstable; the molecules of water of one system are continually changing with the molecules of water of other systems. As Mendeléef expresses it, "Solutions may be regarded as fluid, unstable, definite chemical compounds in a state of dissociation."

In explaining flocculation by the hydrate theory of solution, we ought not to lose sight of the association hypothesis, which has Armstrong as an active supporter. This hypothesis supposes that, previous to chemical change of any kind occurring in solution, a complex association of elements or groups of elements is formed; the various elements are brought into close contact, and should they react with one another to form new combinations, the association becomes unstable and breaks down. This idea of association is undoubtedly a step in helping us to understand the function of catalytic and of hydrolytic agents. In flocculation or coagulation we have the same idea, but in connection with particles or aggregates of molecules instead of elements or small elementary groups, the base of the flocculant or coagulant in the final stage replaces the molecules of water, and forms a stable association of particles or molecular aggregates which settle out from suspension or solution.

# New York Section.

## ON THE NATURE AND ORIGIN OF ASPHALT.

BY CLIFFORD RICHARDSON.

(*This Journal*, 1898, 13—32.)

ERRATA.

| Page. | Col. | Par. | Line. | — |
|---|---|---|---|---|
| 14 | 2 | 5 | 11 | Delete ";" between "1,100 ft." and "from the centre." |
| 15 | 2 | 5 | 2 | For "1893" read "1894." |
| 17 | 1 | 3 | 2 | The percentage of bitumen soluble in $CS_2$ is 52·31, not 53·31. |
| 19 | 1 | 5 | 3 | For "patch" read "pitch." |
| 20 | 2 | 5 | 2 | For "lileration" read "liberation." |
| 21 | 1 | 2 | 9 | Under H. in table, the carbon should be 83·71. |
| 22 | 1 | 7 | 8 | For "votalitisation" read "volatilisation." |
| 22 | 1 | 7 | 9 | For "decomposition" read "deposit." |
| 22 | 1 | 7 | 13 | For "true" read "soft." |
| 23 | 2 | 1 | 23 | For "Konoalo" read "Konowaloff." |
| 23 | 2 | 1 | 25 | For "sulphuric" read "sulphonic." |
| 23 | 2 | 1 | 31 | For "Markoinko" read "Markownikoff." |
| 24 | 1 | 5 | 6 | After "15 per cent." read "of its volume." |
| 24 | 1 | 5 | 10 | For "value" read "vacuo." |
| 24 | 2 | 1 | 2 | For "forms" read "strong." |
| 26 | 1 | 3 | 2 | For "paraffine of paraffine hydrocarbon" read "paraffin or paraffin hydrocarbons." |
| 26 | 1 | 5 | 10 | For "Guanace" read "Guanaco." |
| 28 | 2 | 8 | 1 | For "Company" read "Co., Texas." |
| 29 | 1 | 1 | 4 | For "unsaturated" read "saturated." |
| 29 | 1 | 1 | 8 | Before "quite" read "is." |
| 29 | 1 | 2 | 7 | For "Bittsburg" read "Pittsburg." |
| 30 | 1 | 2 | 1 | For "those which contain" read "the form of." |
| 30 | 2 | 4 | 9 | For "and 100° C." read "at 100° C." |
| 30 | - | - | - | In both tables, for "consolidated" read "Waldorf." |
| 31 | 1 | 5 | 6 | Before "derivatives" read "sulphur." |
| 31 | 1 | 5 | 8 | After "bitumen" read "containing less sulphur." |
| 31 | 1 | 5 | 9 | For "asphalt" read "hard pitch." |
| 31 | 2 | 4 | 17 | For "is" read "consists." |
| 32 | 1 | 1 | 3 | For "originate under such circumstances as to be" read "are." |
| 32 | 1 | 1 | 4 | After "polymerisation" read "especially where sulphur is an element in the reaction." |
| 32 | 1 | 2 | 5 | For "derivatives" read "derivation." |
| 32 | 2 | 4 | 3rd from last | Insert "that" before "there was." |

*Meeting held at the College of Pharmacy on Friday, January 21st, 1898.*

PROF. CHAS. F. CHANDLER IN THE CHAIR.

## DIASTATIC SUBSTANCES FROM FUNGUS GROWTHS.

BY JOKICHI TAKAMINE.

MOST alcoholic beverages are made from cereals. The starch of the cereals is converted into sugars by diastase, and the resultant sugars are changed into alcohol by yeast. The production of diastase constitutes the most expensive part of this big industry. In America and Europe the germination of cereals is used as the only source of diastase. In Japan, however, a totally different process is in use for its production. A certain kind of microscopic plant, which has the property of generating diastase during its growth, has been utilised for this purpose. This diastatic substance (corresponding to the malt of this country) is known as koji, and the material bearing the matured spores of this plant is called tane-koji, or moyashi. The natures of these products have been studied by several investigators, such as Prof. Atkinson, Korscheldt, and others, and methods of manufacture have been described in detail in various modern literatures.

The process of making koji is as follows:—

The whole, unbroken grains of rice, which have been first cleaned of the outer bran, are steeped in water for from 10 to 20 hours, in order to thoroughly soak them. This soaked rice is then drained, and steamed until the starch cells of the grains of rice are opened. The mass is then cooled down to a temperature of 30° C., and about one thousandth part, by weight, of the rice employed, of tane-koji is added and thoroughly mixed with the steamed rice. The mass is then bedded up and allowed to remain for about six hours, at the expiration of which time the same amount of tane-koji is added as before. At the expiration of about 20 hours the mass is divided into small portions and placed in trays, which are then left in the growing-room under proper manipulation, temperature, and humidity for about 50 hours, or until the fungus growth has sufficiently matured. It is then taken out of the growing-room and is ready for immediate use. Koji thus prepared has an opaque, white, velvety appearance, and is employed in the process of making "sake," as a converter of starch into sugar. The diastatic power of koji thus produced is only $\frac{1}{2}$ or $\frac{1}{4}$ that of malt. I have observed that while the rice koji converts a considerable amount of starch into sugar, yet it is incapable of converting thoroughly its own starch. This is proved from the fact that the main part of the starch contained in "sake" residue is derived from rice koji. There are various other drawbacks in the method of manufacture; and closer study of this subject gave me sufficient encouragement to devote my attention to it with a view to its improvement. In the course of experiments I have found out that various kinds of fungi have the property of generating diastase; and thus *Eurotium Oryzæ* is not the only one. It became therefore necessary to select the best kind, and keep it free from other weaker and non-diastatic growths. The nutriment in the soil on which it is grown was found to have great influence on the diastase-producing property of the plant. This fact naturally led me to use an artificial fertiliser to give the plant a healthy and abundant growth.

Phosphate, potash, and nitrogen are the principal ingredients, and particularly is the former essential for producing healthy seeds of the matured fungus. In preparing the matured spores, a carefully-selected species of the fungus, which was cultivated on a culture medium with usual precautions, is now sown in a suitable material which has been thoroughly sterilised, treated, and artificially fertilised. The mass is then put into the growing-room, allowed to grow for five to seven days, and then thoroughly dried and preserved. The product thus obtained contains matured spores of the fungus, and is called taka-moyashi, and is used for producing the diastatic substance called taka-koji.

Careful observations have shown that the amount of diastase generated is directly proportional to the density of the growth on a given area, provided there is supplied a sufficient amount of nutriment in the soil; that is, the closer the fungus is planted, the greater the quantity of diastase produced on a given area. It was also found out that the thickness, or depth, of the soil required for the growth is extremely thin. Therefore, in the case of this fungus growth the possibility of increasing the diastatic power on a given weight or area of substance is very great, while in the case of malt the maximum possibility is limited, and we are producing nearly its maximum now. By keeping the above points in view, I have succeeded, at times, in producing a mass whose diastatic power is twice that of the best malt. The product obtained by this improved process is called taka-koji. (The prefix "taka" means "strong" in Greek, "high" in Japanese.)

In making taka-koji, various articles can be used with success, but for ordinary purposes I use wheat bran as a basis for growing the fungus. The bran, being of a loose and coarse nature, affords a large surface for the growth of the fungus, and a ready access of air—one of the necessary conditions to its growth. Being rich in albuminoids and phosphates, it supplies the most necessary ingredients for the production of the enzyme. It contains a large percentage of woody fibre, which renders its use of special advantage in the process of extraction, as described below. It is cheap and abundant, and in constant supply at all seasons of the year. The wheat bran is moistened with water so as to give the mass 30 to 40 per cent. of moisture, and then live steam is passed through it, in order to sterilise the mass as well as to gelatinise the starch cells. After the mass is cooled below 40° C., a small quantity of the taka-moyashi (varying in quantity from 1 in 100 to 1 in 1,000) is added and thoroughly mixed.

The mass is spread out in thin layers of 1 to $1\frac{1}{2}$ ins. on a cemented floor, or on shelves in a growing-room, where the temperature of the air is kept about 25° C., and humidity near saturation. It will be observed that the temperature of the mass will gradually rise as the growth of the fungus advances. This should not be allowed to rise above 40° or 41° C. This is regulated by adjusting the thickness of the layer and the temperature of the room. After about 24 hours the growth becomes visible to the naked eye, and the mass will begin to acquire the diastatic power. Both the growth and diastatic power increase rapidly after 36 hours, and the latter reaches its maximum point at about 48 hours, when the mass is taken out of the growing-room and cooled to the atmospheric temperature.

Taka-koji, thus obtained, has a light yellowish, velvety appearance, and can be used in the green state as a diastatic agent, or, when it is thoroughly dried, can be kept indefinitely, as is malt or other grain. It is highly diastatic. Some was found to possess twice the diastatic power of the best malt. Taka-koji can be used as it is in solid form, or, more frequently, the diastase may be dissolved out with water by percolation, and the extract thus obtained used as the diastatic agent. About 20 per cent. of the dried taka-koji is soluble in water, and the extract contains all the diastase generated by the growth. In the case of alcohol distilleries, the extract containing 4 to 5 per cent. of the soluble matter of taka-koji is employed.

When it is to be transported, or to be preserved, the extract containing 25 per cent. of solid can be obtained by percolation, and then evaporated in a vacuum pan to a thick syrup, or even to a solid. The concentrated extract thus obtained is of a dark brown colour, similar to that of malt extract, and has strong diastatic power, a half-gallon being about equal in diastatic power to one bushel of best malt. The extract of taka-koji, either fresh or in concentrated form, is used as a converter of starch into sugars in various industries, such as alcohol distilling, beer brewing, the manufacture of grain-vinegar, &c. In fact, wherever malt is used, this product can be used with advantage.

The residue of taka-koji, after it is extracted with water, when pressed and dried, can be used over and over again for making taka-koji, or it can be used as cattle feed. About 40 per cent. of the original weight of the bran is lost during the growth of the fungus and extraction. This diminution of the weight is chiefly due to the elimination of the carbohydrates of the bran. Consequently, this treatment increases the percentage of protein in the residual bran. On one occasion, at one of the Western distilleries, a 50-ton lot of bran had been used four times in making taka-koji, and the residue, amounting to about three carloads, was assayed at one of the Western agricultural colleges for protein, and found to contain a little over 24 per cent. of protein, while the original fresh bran contained only 12 per cent. This cattle feed was shipped to Europe at the time, owing to the fact that it contained a high percentage of protein, and therefore it could stand the expense of longer transportation than bran.

While the above-mentioned taka koji extract is stronger in its diastatic power, many times, than anything that has been manufactured on a commercial scale, yet it still contains a good deal of foreign matter; some small quantity of sugars and mineral matters give it a hygroscopic property, and make it difficult to keep in solid form.

Various methods have been tried, such as dialysis, precipitation with various agents, &c., with a view to isolating the diastase from the extract. The simplest and most practical one is the precipitation by alcohol. An aqueous extract containing 18 to 20 per cent. of the extractive matter of taka-koji is made, and four to five times its own volume of strong alcohol is added slowly with vigorous agitation; a white, flocculent precipitate is formed. After the supernatant alcohol is decanted, the milky liquid is put into a centrifugal machine (cream separator) and further separated from the mother-liquid. This thick paste is then washed with strong alcohol to dehydrate it, and then the mass is pressed under a hydraulic press to squeeze out as much alcohol as possible. The precipitate is now broken, air-dried, and, when powdered, forms the taka-diastase of commerce.

Taka-diastase thus obtained is a yellowish-white amorphous powder, perfectly odourless, possessing a pleasant, nutty taste. It is readily soluble in water, with little or no sediment; it is non-hygroscopic. It possesses a remarkably strong diastatic power, or power of converting gelatinised starch into soluble starch, dextrins, and sugars. Its diastatic power, unlike other diastatic substances, is perfectly stable for all practical purposes. This property is particularly valuable for use as a standard for comparison of the diastatic power in the assays of the other diastatic substances. If the diastatic power of the given taka-diastase be once determined by any of the known methods, it can be depended upon as such for future comparison. The taka-diastase now on the market is capable of converting at least 100 times its own weight of dry starch into sugars in 10 minutes, as determined by modified Junk's test. Taka-diastase may be purified further by reprecipitation or other methods. By such means diastase of wonderful strength has been obtained.

The diastase obtained from this fungus growth differs from malt diastase in the fact that it does not give Lintner's characteristic reaction of malt diastase, that is, giving an intense blue colour when treated with the tincture of gum guaiacum mixed with hydrogen peroxide, but, instead, it gives a milky coloration when so treated. I am inclined to believe that the Lintner's guaiacum hydrogen peroxide test above referred to is not the test for diastase, but it is the test for a certain albuminoid substance which coexists with diastase in malt. I found that an aqueous extract of any of the cereals which have not been germinated, and therefore, in the ordinary sense of the term, are non-diastatic, gives this blue coloration. At all events, the above test is not necessarily indicative of the diastase formed by the germination of the cereals.

Another characteristic difference between this taka-diastase and malt diastase is the fact that for a given amount of sugar-forming power in both kinds of refined and isolated diastase, the taka-diastase has at least three or four times the starch-cell-breaking, or starch-liquefying, or dissolving power, compared with the diastase of malt. This fact is better understood by the following examples:—Suppose we take a refined and purified malt diastase, and its diastatic power is tested by Lintner's method, *i.e.*, its starch-saccharifying power is determined. The same is done with the taka-diastase. And suppose that both (weight for weight) were found to have the same diastatic power, or same saccharifying power. Now take the same weight of each diastase, and let each act upon a great excess of known quantity of starch paste for a given time. On testing the quantity of the sugar formed, both will naturally be the same. But a remarkable difference will be observed in the consistency of the starch. While the contents of the vessel containing malt diastase may still be thick, the contents acted upon by taka diastase will be very thin. This, along with other facts hereafter referred to, indicates that there seem to exist at least two kinds of diastases, *viz.*, one a starch-liquefying diastase, the other a starch-saccharifying diastase, and the speculation may be made that intermediate kinds are starch-dextrifying diastases.

In the practical application of the diastase—whether it is in the manufacturing industries, such as distilling, brewing, &c., or in its physiological uses, *viz.*, the digestion of starch foods, &c.—this starch-liquefying property of diastase is just as important as its starch-saccharifying property. In my opinion, in the future, the artificial production of starch-liquefying property will become of more technical importance than that of the saccharifying power. I have found that all cereals, and in fact all starch-containing crops, in their ordinary, natural condition (without undergoing germination), contain a large quantity of starch-saccharifying diastase which could be utilised in the arts; that is to say, all starch-containing crops naturally and without germination contain almost enough starch-saccharifying diastase to convert their own starch into sugars, provided the starch be made soluble by some other liquefying diastase.

In the case of malt diastase, these two kinds of action seem to be in such proportion that neither of them is in excess, and consequently the quantitative measure of one is naturally the measure of the other; *i.e.*, the diastatic power of malt is measured by its saccharifying power, which is determined by the sugar formed.

This kind of test answered the practical purpose as far as malt is concerned. But now, as another kind of diastase comes into the field, the determination of the liquefying power of diastase became of practical importance, or, at least, the commercial value of the diastase must not be dependent upon its saccharifying power alone; that is to say, the Lintner's method of determining diastatic power, which is now very extensively adopted, and other similar methods, where the soluble starch prepared by acid is acted upon by the diastase to be tested, are not applicable for the valuation of the diastase like taka-diastase, which has extra virtue over that of malt diastase.

## DIASTATIC BODIES IN CEREALS, AND THEIR UTILISATION.

BY JOKICHI TAKAMINE.

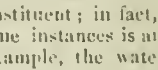
Exhaustive chemical investigations have been made on the nature and constituents of various cereals and other starch-containing crops. However, there seems to be one point that has been overlooked, *viz.*, the diastatic power of the natural ungerminated grains. This is not surprising, as in the ordinary sense of the term there is no diatatic power in the ungerminated cereals; that is to say, the cereals have no power to convert gelatinised starch into sugar. But closer study on the subject brought to light one remarkable and very important property of the cereals and its practical application.

During the course of numerous experiments with diastatic substances generated by fungus growth, I discovered, in the early part of 1892, that a certain constituent of cereals, which in itself has no diastatic power, possesses the property of acquiring a remarkably augmented and increased diastatic power when used in conjunction with diastase of taka-koji. I have found that the said peculiar constituent is soluble in water, and can be dissolved out from the grain. For instance, make an aqueous extract of comminuted wheat or corn. This extract will be found to possess no diastatic power. Now mix it with a known quantity of taka-koji diastase of known determined diastatic strength. Then the diastatic power of the mixture will be found very much more than the sum of the diastatic strength of each constituent; in fact, the diastatic power of the mixture in some instances is augmented more than three times. For example, the water extract from one part of wheat flour, which has practically no diastatic power, is mixed with the aqueous extract from one part of taka-koji, the diastatic power of which is taken as a unit; then the diastatic power of this mixture will be found to be more than three, instead of only one.

My explanation of this remarkable phenomenon is as follows:—The natural ungerminated cereals contain in soluble form a large quantity of starch-saccharifying diastase. This saccharifying diastase, however, has no action on raw or gelatinised starch, but it has strong power of converting soluble starches and dextrins into sugars. Now the starch that has been made soluble by the starch-dissolving power of taka-diastase is acted upon by the saccharifying diastase existing in cereals, forming, as a result, as much more sugars as there were formed soluble starches by the starch-dissolving diastase of taka-diastase. The following graphical illustration will make the explanation clearer:—

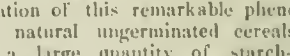
In case of malt diastase, I suppose that for every unit of starch-liquefying diastase (L) there is a corresponding unit of saccharifying diastase (S), as in Fig. 1:

Fig. 1.



while in the case of taka diastase, the ratio of L to S will be something like Fig. 2:

Fig. 2.



and the aqueous extract of cereals will be like Fig. 3:

Now if we combine Fig. 2 and Fig. 3, we shall have as it were three molecules of malt diastase, as in Fig. 4:

This fact seems to prove that there are at least two kinds of diastase.

Fig. 3.

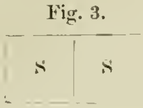
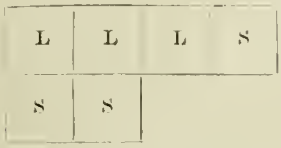


Fig. 4.



The fact that the aqueous extract of the cereals has saccharifying power, can easily be proved by the fact that if it is allowed to act upon the soluble starch previously prepared by the action of acid on starch, saccharification will at once take place. As far as I know, it was the accepted idea up to the present time that but one kind of diastase, which performs every stage of transformation of starch into sugar, is produced when the grains are subjected to germination.

From the above facts, and from other observations, I venture to say that the starch-saccharifying diastase necessary to convert all, or at least most, of the starch cell contained in each grain of cereals, exists in the grain already formed, and that the diastase formed during the germination of cereals, or the malting process, is chiefly, if not all, starch-dissolving or liquefying diastase. It seems to me that nature stores up the carbohydrate in the form of insoluble starch cells in the warehouse of the cereals with all the requisites until wanted, and when she wants the grain to grow, all she has to do is to generate starch-liquefying diastase.

The above-mentioned augmentation of the diastatic power, or the utilisation of the saccharifying property of the cereals, has been applied practically in the manufacture of alcohol from grains. In the early part of the history of the application of koji-diastase in distilling, the extract of taka-koji alone was used. But since this remarkable fact was found out, an extract from the mixture of equal quantities of taka-koji and wheat bran was used instead of taka-koji alone. By this means there was secured not only the economy in taka-koji, but also marked augmentation of the diastatic power.

The next step in the progress was the utilisation of the saccharifying power of cereals from which grains the mashes were going to be made. Heretofore in the alcohol distillery the grain, say corn (maize), has been ground to meal and cooked with water to form a cooked mash. During this cooking all the saccharifying diastase of the corn is destroyed by heat, and therefore wasted. Now in this new process the ground meal is put into percolators and cold water passed through it to extract the saccharifying diastase. Then the meal, after this extraction, is cooked in the usual manner, and the extract of the grain is mixed with the extract of taka koji, and used as a converting agent, thus still further economising the cost of wheat bran.

Having thus found the abundant supply of starch-saccharifying diastase ready-made in all the cereals, in all the practical applications of diastase, how to produce the most efficient starch-liquefying diastase will be the problem for us to solve. My paper, entitled "Diastatic Substances from Fungus Growths" will throw some light on the subject.

The study of the nature of the saccharifying diastase of the cereals, and further practical applications, will be treated in future communications.

### Discussion.

The Chairman said that all would feel grateful to the author for having brought to their attention this new and interesting development of diastase. Had the author or anyone working with him attempted to ascertain the constitution of diastase—whether produced from malt or by his microbe, and did it seem to consist of one definite substance. The author had further mentioned the liquefying property and the saccharifying property. Was there constant ratio between the liquefying and saccharifying properties? Did the ratio remain constant as the diastase became purer and purer?

Dr. L. Saarbach asked if the action of the koji could be regulated, as in the case of malt. In a distillery they wanted all starch ultimately converted into alcohol, but it was different in brewing; the wort resulting from the mashing process should retain some dextrin to form the extract of the finished beer. By using different temperatures, worts from the same malt showed a ratio of fermentable bodies from 100 : 30 up to 100 : 55.

In regard to the liquefying action, how did the author explain the very strong effect in this respect of green malt, compared to kiln-dried malt.

Mr. C. E. Pellew reminded the meeting that the test with guaiacum and hydrogen peroxide, referred to by Mr. Takamine, was an old test much used in medico-legal work as a test for blood. A minute trace of blood, whether fresh or old, would give the characteristic blue colour; but of late years it had been shown that it was not so much a test for blood as for the contents of cells, whether animal or vegetable. To illustrate this point to his medical students, he was accustomed to cut a raw potato in two, and make the guaiacum test upon the exposed surface. If there were any unsound portions in that surface they would turn a particularly deep blue. The test would also respond to milk, to spermatic fluid, and to many vegetable juices. This method of malting grain by the agency of a fungus growth was not confined to Japan. In studying last spring for some lectures on alcohol, he had to test some statements in temperance literature that the knowledge of alcohol was *not* universal, but that, among others, the North American Indian had kept his innocence in this respect until seduced by Henry Hudson with a generous dose of rum, in New York Harbour. A well-informed friend, however, told him not only about wine made from grapes by Indians along the Chesapeake, and about various concoctions brewed from masticated grains, but also about a curious drink made from maize which had been placed for some time in the soil of some particular marsh, and allowed, as his friend stated, to partially decompose there. He supposed that this was simply the familiar sprouting process of grain; but, remembering the special emphasis laid upon the particular locality, and that that one sacred spot was the only one to produce the desired effect, it has occurred to him that it might have been due to a special fungus growth localised in that one spot, as was the case with the mud of a swamp in New Zealand, impregnated with the tetanus bacillus, and used for many generations by the natives as an arrow poison.

Dr. H. Endemann said that one of the points that interested him most in the author's paper was that natural grain, and very likely all the natural substances containing starch, had a certain amount of diastase. An observation had been made, and was very likely known to all the members of the Society, that when potatoes froze and afterwards thawed they tasted sweet, and this change of taste was very likely due to the fact that the cells disrupted and the diastatic properties of the potato acted upon the starch of the potato and produced a certain amount of sugar. This old observation had even been used as a foundation for a Prussian patent. In a report of the Berliner Gewerbe Verein of the year 1823, which he had for the purpose of tracing a certain patent, a friend came across this peculiar patent, which consisted in allowing potatoes to freeze, and, after mashing them, fermenting them for the purpose of obtaining spirits. He did not know whether the patent had ever had any very serious results. At any rate a frozen potato contained sugar, which must be due to diastase in the potato and the starch coming in contact by the bursting of the cells, due to the freezing of the water.

Mr. R. C. Woodcock said he presumed, from what the author had said just now, that he used bran chiefly because of its being a high-feeding bed for the growth of this plant, and on account of the nitrogenous matter contained in it; and he asked whether, after the taka-koji was extracted the residual bran contained very much nitrogen. It looked as though the plant consumed the cellulose.

Dr. Hugo Schweitzer said with regard to that wonderful result of taking nothing, and adding one, and getting three, was it not possible to add nothing right at the start and to work with a diluted taka-koji, *i.e.* the ordinary taka-koji diluted with wheat bran, and get the same result. Were the products of the saccharification the same in saccharifying with the old diastase as with the taka-diastase, and was the taka-koji a commercial article and could it be bought?

Mr. J. Takamine, in reply to the Chairman, said that as to the constitution of diastase he was still in the dark. He had tried some few combustions with this substance and found every time he got different results; that is, every time he purified it. By reprecipitation, or by fractional precipitation, he had prepared a great many samples of different diastatic power, but he found just what had already been discovered in connection with the diastase; that is, the more you purify, the higher the amount of nitrogen, and to some extent the mineral constituents also: but his investigations in that line had been comparatively limited, and not only that, it had not thrown any additional light on the subject. Instead of consisting of one definite agent, it rather seemed to consist of two associated together.

As to whether the ratio remained constant as it was made purer and purer from the combustion point he was unable to tell anything about it, and that was probably the reason why no one could show that there were different kinds of diastase, and there was no opportunity of proving it. Of course it was possible that it was one substance, at the same time having different strengths of action in different directions; but it was more probable that there were at least two, especially when such actions as stated could take place. That was a point that still had to be investigated, and in fact there were several dozens of questions that occurred to him that he wanted to answer that could only be answered by investigation. He was investigating a few in which he was directly interested, and as he accumulated results he would take pleasure in laying them before the Society.

Taka-diastase was affected by heat pretty much the same as malt diastase; that is, at 70° C. the diastatic power was permanently destroyed. It did coagulate like any other albuminoid. If a solution of it were boiled, it would coagulate like the white of an egg. Alcohol precipitated it from its solution, simply from the want of a solvent.

In answer to a question of the Chairman as to whether there was any reagent that made an insoluble combination, he had tried all the usual mordants, lead salts, alum salts, tannin, &c. when he was trying to isolate the diastase, but for practical purposes of course alcohol was the simplest and best. The alcohol precipitated it without changing it or destroying its efficiency, while tannin also precipitated it, but the diastatic power was very much weaker. Whether this was due to the bulk of tannin, or whether a new combination was formed he could not tell. Salicylic acid and other antiseptics in moderation preserved the diastatic power, but in excess stopped the diastatic action.

In answer to the Chairman's question regarding the spores and how he obtained them, the author replied that that part of the description had been very incomplete, and that therefore he would briefly go over it again. In order to make this diastatic substance, they placed the seed and plant on steamed bran, and let it grow 48 to 50 hours when it reached the maximum point of diastatic power. That was called taka koji, and the spores were still very young, fit for diastatic purposes, but not fit for regenerating purposes. To regenerate itself, or in order to make a seed, they had to allow longer time that the seed might be thoroughly matured and healthy. For that purpose, take bran of wheat or any other suitable material, and artificially sterilise it. Get some potash salt and phosphate and a suitable amount of nitrogenous matter. In the case of wheat bran not much nitrogenous matter was needed, but it would be well to add some good soluble phosphate. Then after suitably preparing the soil, take a small quantity of seed just in a similar way as you make this taka-koji, and let it grow, with of course much care in every way. The room ought to be frequently cleaned and sterilised by different chemicals so as to keep away all other non-diastatic fungi, just on the same principle as was used in a bacteriological laboratory. With such care, the mass was allowed to grow about seven days, and during that time it required a little different manipulation, but on the whole the principle was just the same. Then, instead of being yellowish-white, it became greenish-yellow. The specimens shown were obtained by putting the mass into a sieve and sifting it, and the spores, being fine, passed through, and left the bran behind.

In answer to Dr. Saarbach, the same thing could be done with this koji by regulating the temperature of the mashing. If they used the koji green the action would be very similar to that of green malt; but dry taka-koji corresponded to dried malt, and with a little care and observation one could have any amount of desired extract, unfermentable extract. In his distillery sometimes unfermented dextrin was not required; but by proper observation and manipulation it could be regulated to any extent wanted.

In reply to questions by Dr. Ullman, the diastatic power was strongest at 111° to 116° F.; to a certain extent acid destroyed its action, and the extent depended upon the kind of acid. With some kinds of acid it would stand a great deal; hydrochloric acid about 1 in 1,000. Above that it weakened very rapidly. In a weak aqueous solution it did not keep very long. It was liable to decompose in a few days if kept in an ordinary room, so that if the diastatic power was to be used it ought to be dissolved there and then. The liquefying action was necessary before saccharification took place. As to whether this was not due to the diastase itself and without several diastases being present, he was trying to show that there were different kinds of diastase, and the fact that there were at least two was indicated by the mixture of bran and taka-koji. When one combined a substance of no diastatic power with a substance of one diastatic power, and the diastatic power was multiplied, that seemed to show that the extract of the cereals had saccharifying power. Starch must be liquefied first to make any practical use of this extract of cereals, and accordingly this solution was of no practical value until one found some starch-liquefying substance that could be used in conjunction with it, and for that purpose this taka-diastase derivative could be used with advantage.

He would like to say a word to Dr. Pellew's remark. All cereals were liable to sprout, and during the sprouting a diastase was naturally produced, and probably from that observation with a slice of potato the part that seemed rotten exposed the exact part where the diastase was generated. The part of the potato near the germs was liable to get rotten sooner than any other part, because the nearer to the germs the greater the amount of diastase and sugars, which were more prone to decomposition than starch cells. In regard to steeping corn in a marsh, some of the distilleries even at the present day, in order to give a certain flavour to a certain kind of whisky, used germinated corn along with the barley malt, and some used germinated rye. All grains, when kept in a damp place, began to sprout. The result obtained would not be due to any fungus growth, but due to germination of grain in the marsh.

In answer to Mr. Woodcock, this taka-koji, of which a tremendous quantity was used every day in a practical distillery or brewery, after the extraction of the diastase from it, could be used a good many times over to grow fresh plants on, as there was plenty of nitrogenous matter still left in the residue. In some cases he had used the same bran four times after making the extract. It was pressed in a hydraulic press and dried in a continuous revolving dryer, and mixed with a small quantity of fresh bran, and used over and over again; and, if properly handled, there was a great increase in nitrogenous matter. After using three or four times, it could be dried and used as a cattle food. Every time it was used, the richer it became in proteine. Ordinary bran contained about 12 per cent. of proteine, and its feeding quality was measured by that. After using three or four times, the percentage of proteine rose to 21 per cent. During the growth, about 20 per cent. of the weight of bran was lost: the plant must feed on something, and a large quantity of carbonic acid was evolved. If the room were shut up long, the gas would almost put out the light, so that carbohydrates, fibres not necessarily starch, were consumed during the growth of this plant. The carbohydrate was very cheap. The nitrogen was more expensive, and every time one grew this one concentrated the proteine in the feed. He once had an idea of getting a rich cattle food by planting some fungus on cheap feed.

In answer to Dr. Schweitzer, he had started with one portion of taka-koji to nine of the bran. In practice in a distillery, he had of course to use the bran, as flour was too expensive, and he extracted the soluble portion of the bran, and then mixed it with the soluble portion of the taka-koji. He wanted to economise his taka-koji as much as possible, and in the case of bran, half and half was the best for commercial purposes. On mixing these two, the product seemed to be identical with that of malt diastase. In the case of taka-koji diastase, this liquefying power was stronger, and if the conversion were allowed to take place by taka-koji alone, the product seemed to be slightly different—more dextrine in the one case and more maltose in the other. The taka-diastase could be bought in any quantity, but the taka-koji at the present moment was not on the market. Regarding the manner of working with it, it depended upon the investigations that were required. He would be pleased to supply the secretary with some of it if he needed it for investigation purposes.

## SAMPLING CARGOES OF ORE.

BY WILLIAM GLENN.

In another place (Trans. Amer. Inst. Mining Eng., Vol. XX., p. 155) the writer has ventured to say: "The taking of proper samples of crude ores seems to be less thoroughly understood, and less carefully practised, than its importance requires. We all know how often we encounter the reports of very accurate assays and analyses, the weight of which as evidence wholly depends on the method of obtaining the sample, and is very likely *nil*, because there has been no proper sampling. Passages might be cited from technical books and from current technical literature (all the work of writers whom we delight to honour) to show that even among high scientific authorities there is too little acquaintance with the practical art of the sampler. There is no metallurgical or chemical establishment which does not frequently receive samples truly representing nothing. They consist usually of bits of ore, or what not, selected because they are worse or better than the average of what they are meant to represent. And they are worthless, no matter who made the selection. Where conscious choice is permitted to enter into the operation a fair sample will not result, unless by a miracle."

Commenting on the foregoing, Dr. R. W. Raymond said that the importance of sampling had by no means been overstated, and with that assertion the average chemist will coincide. From points as far distant as California and New South Wales, from New York and London, from Yokohama and Constantinople, I myself have had sent me single stones as a preliminary basis for contract of delivery of thousands of tons of ore. And frequently such stones have come from those technically educated.

I propose to speak of one method of getting the final sample of a cargo of ore, which may be 2,000 to 4,000 gross tons, no matter whether the cargo consist of iron or manganese or other similar material. We will assume that it is essential to know quite exactly the quality of the cargo, either that it may be paid for justly, or that its after use may be intelligently directed, or for both of these reasons. And I desire to confess that the method to be stated is the only accurate one known to me.

Such a cargo almost universally is hoisted out of the ship in buckets of about one ton capacity, from which it is dischaged into small cars or into horse carts. Next it is transported to the place of storage. It matters not at which of these points the sample be taken, since the only alteration which could occur in the ore would be loss of moisture, which is without value. But the gross sample ought to be taken where the ore is weighed, because that very loss may happen. And thereafter the sample must not be permitted to lose any of its water.

My own custom is to store the gross sample in casks which hold about a thousand pounds of ore, and which are lined with sheet lead, because such casks happen to be available. But no doubt the same end could be attained by using casks which had been painted on the inside. Either of these would prevent absorption of water by the wood of the casks from the ore held within them. It avails not to suppose that the ore is dry because it looks to be so. Moreover, it often happens that part of the cargo is wet from bilge water, in which case it may be the last to come out of the ship; or a part of it may have suffered from seas taken on during stress of weather. In any case, the entire cargo must be sampled, and no moisture should be permitted to escape from the gross sample when once it is taken.

The sampling casks should be near the scale on which the ore is weighed. They should stand upright, with the upper heads removed, the open end of each of them covered by a gunny sack, and the loose heads laid upon the sacks. After the small car or the cart of ore has been weighed, take from it about 10 lb. for each ton in the load, using a shovel to do this. Put this sample in a cask and cover that with its sack and its loose head. When a cask is full, draw the sack closely over its open end, lay the loose head on that, put on that a heavy weight of some sort, and seal the head to the cask. This readily may be done with sealing-wax and bits of string. Do not subject the casks to hot sunshine.

When ore is hauled over a rough road in a cart, the finer parts of the ore tend toward the bottom of the contained pile; and although such is not likely, yet the finer part may be of constitution different from that of the large lumps. But an average sample may be had upon the shovel if the tail-board of the cart be removed first.

Proceeding in this way, about 20 casks of gross sample result from a cargo of 2,000 tons. Experience has taught me to respect a sample so taken and so treated. There has been almost no room, so far, for error to have crept in.

We have now to get from the crude sample (1) a manageable quantity fairly well representing its content of moisture, and (2) a finely ground powder which correctly represents its chemical qualities.

In my own practice, the crude sample is passed first through a rock breaker, then through Cornish rolls beneath the breaker, and then into a cylindrical revolving wire-screen having nine square meshes in each square inch of its surface. The ore which passes the screen is taken upward by an elevator, and delivered into a flat-bottomed iron trough which is 6 ins. wide and 4 ins. deep. The trough is inclined so that the crushed ore flows slowly down it. That part of the ore which does not pass the screen is elevated, and again delivered into the crushing chamber of the rock breaker.

Of appliances we have sampling barrels already described, an old canvas sail 14 ft. square, two wooden sample boxes of 150 lb. capacity each, common sweeping brooms, square-pointed shovels, and an ordinary fire-shovel, 4 ins. wide, meant for use about a stove. In addition, there are required six glass fruit-preserving jars of half a gallon capacity each.

After the crushing machinery has been freed from any ore contained within it, we pass through it about half a ton of the cargo sampled. Thereafter, it can contain no ore which might contaminate the sample now to be worked down, as follows:—

One after another, we open the sample casks, and at once pass their contents through the crushing machinery. As the crushed ore passes along the trough mentioned above, a man presses the square point of the small shovel against the flat bottom of the trough, and holds the shovel there until it is nearly filled with ore, when he empties the ore into a sampling barrel. This he repeats so long as any of the crushed sample flows along the trough.

We have supposed that we were dealing with a cargo of 2,000 tons, from which were taken 20 casks, or 20,000 lb., of crude sample. In that case, we would shovel from the trough about five casks of crushed ore while the entire sample was passing through it. We would now have about 5,000 lb. of crushed ore to represent the cargo. If the sampling casks were well guarded against loss of moisture, then we may be assured that but little of it was lost while the crude sample was passing through the crushing machinery.

The sailcloth may now be smoothed out upon a floor and the five casks of sample emptied on it, all in one pile and near one side of the cloth. Standing at one side of pile, let men shovel the ore toward the opposite edge of the sail, using square-pointed shovels to do the work. Then sweep all the scattered ore to and upon the conical pile. Halve and quarter the pile and cast out two of the quarters. Shovel the remaining quarters into a pile, shovel that over and upon itself to the farther edge of the cloth, then back to the centre of it, where again make up a conical pile and sweep the scattered ore upon it. Let three men take three of the glass jars, and while walking around the pile, dip up into them small quantities of the ore from many points on the pile, and until the jars are full. Then brush clean the open ends of the jars, put in place the rubber washers, and screw on the jar covers. In like manner fill and cover the remaining three jars. That six jars are for moisture sample. Label and seal them.

After the sample has been put upon the sail, to the time when the moisture sample has been gotten into jars and the covers screwed fast on them, work must be rapid. It is during mixing and quartering that moisture escapes from the sample. But once having secured the moisture sample, we are entirely at liberty to proceed slowly until the sample has been reduced in volume to between 200 and 300 lb. Put that in the wooden sample boxes already mentioned.

The top of our sampling table is a cast-iron slab 36 ins. square and 1½ ins. thick. It was planed smooth originally, and after many years' use it still is sufficiently free from indentations. Upon that we would empty the sample boxes, and would further mix the sample by moving it back and forth over the table, using thin trowels to do this. Then we would make up the pile, sweep up the dust with broad, thin paint brushes, halve and quarter, and reject two quarters. Next we would spread the ore as thinly as the size of the table permitted, and would break the larger lumps of it by means of blows from broad-faced steel hammers—bucking hammers. When there were no longer obtrusive lumps of ore, when the mass looked fairly uniform, we would make up the pile, sweep up dust, halve and quarter, and reject two quarters. And so we would continue until the sample had been broken until its grains looked like coarse and fine sand, and its volume was about 40 lb. This we would put on the steel table, which has a surface 36 ins. in diameter. It was made of ⅝-in. boiler plate, planed smooth.

So far, the lumps of ore would have been broken by blows of the hammers, and not at all by any process of rubbing them between the surface of the table and the faces of the hammers, for that kind of movement would abrade the table, affording particles of iron to vitiate the sample. Except so far as may safely be disregarded, this does not follow when grains of ore are rubbed between steel surfaces. Therefore, on the steel table the process of reduction of ore grains is by rubbing, or milling as it is termed.

We would spread the ore evenly over the table and would rub it with the bucking hammers. We would continue to mill the ore until its grains looked uniform, until none of them stood out obtrusively; then we would mix the sample, make up the conical heap, sweep up the dust, halve and quarter, and reject two quarters. And so we would continue to grind the ore and quarter the sample until only about a pound of it remained. When that volume had been reached, the mass would appear to be dust, but it would feel gritty when rubbed between one's fingers. If it did not look to be dust, we would grind it until it did; if it did so appear, we would quarter it once more, using spatulas for moving the fine ore when that state had been reached. And having gotten the sample reduced to half a pound, we would grind that to an impalpable powder and sweep the dust into a pile. From the pile we would fill as many and as large sample bottles as might be required of us.

The paper cited at the beginning of this account is entitled, "Sampling Ores without Use of Machinery." The reader may refer to it for a definition of what here is called mixing and quartering the sample.

As will have been noticed, the writer has said what his appliances are, and not what they best had be. As examples:—The sail cloth is no more than a substitute for a smooth and tight floor, which few of us possess; the cast-iron table could well be replaced by a larger one of steel.

As to the method, it is the best one known to me. Since we do not know of errors in it, and are willing to abide by it after many years of usage, it is clear that we think it just. If the reader can see that it is not, that it must fail at some point or at many of them, then will he be in duty bound to point out precisely where, and for what reasons, failure could come. And for his trouble he would receive the grateful thanks of many of us indeed. Even more would he merit our gratitude by publishing a better method of getting ore samples.

# Nottingham Section.

*Meeting held at Burton-on-Trent on Wednesday, January 26th,* 1898.

MR. JAS. O'SULLIVAN IN THE CHAIR.

## THE ESTIMATION OF CUPROUS OXIDE BY MEANS OF STANDARD POTASSIUM PERMANGANATE SOLUTION.

BY R. M. CAVEN AND ALFRED HILL.

(*This Journal*, 1897, 981.)

### Supplementary Note.

The authors have carried out the suggestion that an asbestos filter should be used in place of filter paper, and they find this a considerable improvement. Selected fibrous asbestos is cut into pieces an eighth of an inch in length, digested with strong sulphuric acid to destroy organic matter, then thoroughly washed, and mixed into a paste with water. For the preparation of the filter we have used a Hirsch's porcelain funnel with perforated filter plate; pouring the asbestos cream into the funnel, and applying suction by means of the filter pump until a mat of asbestos, suitable to receive the precipitated cuprous oxide, was obtained. After the removal of the beaker containing the precipitated cuprous oxide from the water-bath, the supernatant liquid is at once decanted through the filter, and the cuprous oxide remaining in the beaker is stirred up with hot water, transferred to the filter, and washed until free from alkali. The last traces of cuprous oxide need not be removed from the beaker, as these can be dissolved later on in a little of the acidified permanganate solution. The asbestos containing the cuprous oxide is transferred by means of a glass rod to a porcelain dish about eight inches in diameter, and the mass thoroughly broken up with water. The acidified permanganate is next poured into the dish, some of it being reserved, if necessary, in order to dissolve any cuprous oxide remaining in the beaker; and the liquid is stirred thoroughly for a minute or two, until solution of the cuprous oxide is judged to be complete.

We find it best, under these circumstances, to add excess of oxalic acid solution, after adjusting the temperature of the liquid, and then to titrate back with the permanganate. This process is very rapid, owing to the use of the filter pump, and we have found it to give consistent and accurate results.

### Discussion.

The Chairman said that the process was an interesting one. He thought that instead of removing the asbestos filter in each experiment, it would be better to pour the acidified permanganate through it, and so use the same filter more than once. The asbestos might even be shaken with the solution in the Soxhlet tube, in order to dissolve the cuprous oxide, and the asbestos mat would then be re-formed on pumping the liquid from the tube.

Dr. A. L. **Stern** remarked that the process could evidently be used only when the cuprous oxide was uncontaminated with organic matter. The method depending on the use of the Soxhlet tube (Jour. Chem. Soc. 69, 1691) was rapid and satisfactory.

Prof. F. **Stanley Kipping** said that he had had the pleasure of seeing the authors' process in operation, and could testify to its rapidity and simplicity. The operations themselves were exceedingly simple, and the end reaction delicate, since the blue colour of the cupric salt was so slight as not to mask the pink of the permanganate. The process should certainly be better than the one in which the cuprous oxide is weighed.

Mr. **Caven**, in reply, said that it was necessary for the acidified permanganate solution to remain in contact with the asbestos for a minute or two, in order for complete solution of the cuprous oxide to take place. He did not think that this could be done advantageously in the Soxhlet tube. The trouble of making the asbestos filter would be found to be so slight when the perforated funnel was used, that he did not think the use of the Soxhlet tube, as suggested by Mr. O'Sullivan, would be an improvement. The authors had already pointed out in their paper that the process could only be applied in those cases where it was permissible to weigh the cuprous oxide. They believed that it would then be found to be a saving of time upon the gravimetric method.

## NOTES FROM SOME OF THE TECHNICAL LABORATORIES IN COPENHAGEN.

BY JOHN GOLDING.

(Abstract.)

The author said that through the kindness and courtesy he received during a month's work in the summer of 1897 in Copenhagen, he had been enabled to gain an insight into the methods of work adopted in many laboratories of the capital, and had been much impressed by the extreme accuracy and care which characterised the scientific work of the Danes. Much of this work, together with the description of methods and apparatus used, had not been published at all, or had only found a place in local records.

Mr. Golding showed a number of lantern slides from photographs taken by himself in Copenhagen. The first of these showed apparatus in use in Herr Jorgensen's laboratory and special attention was directed to the inoculation chambers which are used by students employing liquid media for the cultivation of yeasts. These chambers resemble a large empty balance case, the interior of which can be easily rinsed with a 0·1 per cent. solution of mercuric chloride, thus ensuring a practically germ-free atmosphere in which to work. When in use, the front is raised sufficiently to admit the bare arms of the operator. Mr. Golding stated that since his return he had found these cases of great value for ordinary bacteriological work.

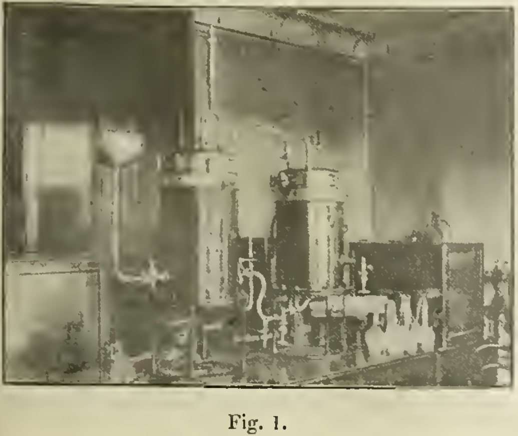


Fig. 1.

A slide of a model propagating machine (Fig. 1) was also shown, and it was pointed out that special advantages were obtained by having the wort cylinder and fermenting cylinder of equal size.

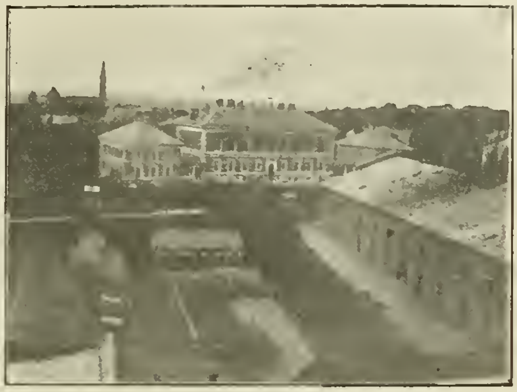


Fig. 2.

Slides were next shown from the Old Carlsberg brewery, the profits of which go to keeping up experimental laboratories, &c. A view of the new experimental laboratory (Fig. 2) from a tower in the old Carlsberg brewery, showed Prof. Kjeldahl's house to the left and Prof. Hansen's to the right, the large building in the centre being used for research work, chiefly in connection with chemistry and biology as applied to brewing.

The method of determining nitrogen, introduced by Kjeldahl, had not been altered since its introduction into his laboratory. Permanganate was still used when boiling up with strong sulphuric acid. A limited number of Englishmen are admitted for original work in this laboratory, free of charge.

Slides were also shown of the Agricultural Research Laboratory, where Prof. Sterch and his assistant, Herr Emil Holm, carry on their work; and explanations were given of apparatus used by them which is not in use in this country. This included a form of extractor of which the condenser is mentioned in the last number of this Journal, page 980, and which was exhibited by Mr. Golding.

Milk fat is determined by drying a weighed quantity of milk on prepared kaolin, and placing it when dry in the inside glass cylinder. Powdered feeding stuffs are previously dried in an atmosphere of coal gas before being placed in the gas cylinder. Either substance is then extracted by percolation of ether.

Another interesting feature of this laboratory was the use of a solution of lime water standing over lime in place of standard solutions of potash or soda, the advantages of which are that the solution remains of $\frac{1}{20}$ normal strength, and only varies to a very slight extent with the temperature, more lime going into solution if by chance any portion of it is removed through precipitation caused by the access of carbonic acid gas from the air. This standard lime solution, which can be made up without the aid of a balance, would be of special use in dairies, and would not be liable to an alteration in composition which could not be detected. A table of the experiments performed in this laboratory with this solution showed that the variations for temperature were constant and exceedingly small.

### Discussion.

Mr. **Charles Matthews**, who opened the discussion, said he had been extremely interested in Mr. Golding's paper, and should like to ask the reader whether any special precautions were taken to prevent or keep down contamination from the dust of the room, in addition to the use of the inoculation chambers.

Mr. Walter Odling asked what advantage was gained by using kaolin instead of paper. He had found paper thoroughly satisfactory.

Dr. Stanley Kipping said that in the Kjeldahl process a few pieces of paraffin put on the surface of the liquid prevented frothing. He alluded to the superior advantages of the Copenhagen laboratories as compared with those in this country, as far as the study of the scientific side of practical brewing was concerned, and expressed the hope that the excellent example set by the Danes would be emulated in the Burton district, and an endeavour be made to obtain increased facilities for this end at University College, Nottingham, which was situated in the heart of the chief brewing centre. He added that he would be happy to co-operate with the Burton brewers to bring about so desirable an end.

Mr. J. T. Wood said the use of a standard solution of lime water had obtained in the tanning laboratories for some years. Prof. Procter, of Leeds, first published it in 1885.

Mr. Golding, in his reply, said that all inoculation experiments were carried out in the inoculation chambers, all sources of contamination were removed from the laboratory, and the walls and floors were kept as clean as possible. The chief advantage to be obtained from the use of kaolin instead of an Adams coil was that it could be freed from every trace of fat by ignition. With regard to the lime solution, Mr. Golding said he was not aware that it was used in tanning laboratories. He had never seen it in use in agricultural laboratories or dairies in England, but thought that as it could be made up without the use of a balance, and as it corrected any contamination from the carbonic acid gas of the air, it could advantageously replace the standard alkaline solutions when such had to be purchased.

# Scottish Section.

*Meeting held on Tuesday, January 25th*, 1898.

DR. G. G. HENDERSON IN THE CHAIR.

## CYANIDE PRACTICE AS IN USE ON THE KOLAR GOLD FIELDS OF INDIA, WITH MORE SPECIAL REFERENCE TO THE MYSORE COMPANY'S PLANT.

BY LAURENCE PITBLADO.

The cyanide process up to the year 1894 had not been attempted on this field; but since the introduction then of a small 2,000-ton plant by the Mysore Company, it has advanced slowly but steadily.

In 1895, Balaghat started a 2,500-ton per month plant; the following year Champion Reefs followed suit with a 4,000, at present increasing to 8,000 tons capacity; and this year Nundydroog has introduced one of 3,000 tons. The Goldfields, and Nine Reefs, have also small plants of about 1,000 tons each.

The ores met with on the field present no difficulties in the use of cyanide, consisting mainly as they do of nearly pure quartz with only a small percentage of pyrites. In contrast therefore to the general rule, sun-drying and weathering the tailings—on this mine at least—causes a less consumpt of cyanide with better extraction than when treating the sands fresh from the settling pits or during the wet season.

The sands which first fell to be treated by the Mysore plant were an accumulation of concentrated tailings from the Pan Mills, averaging some 4½ dwt., and the treatment proved entirely successful, the extraction running about 65 per cent., with an average consumpt of cyanide equal to 1 lb. pure KCn per ton. An interesting point of the working of this material is the recovery every month of a large quantity of mercury from retorting the zinc-box precipitates, reaching as high as 175 lb.; but the average is nearer 100 lb.

A month's trial treatment of sands direct from the mill was tried; but owing to the use of a coarse 800 screen (28-mesh) in the battery, it was not a success. Last year another bulk trial of sands crushed through a 1,600 screen (40-mesh), with inside amalgamation in the battery, was tried, and proved most successful; 90·65 per cent. being extracted by chock-block and plates, and 74 per cent. from the tailings by cyanide, making a total extraction of 97 per cent. from the ore as delivered to the mill.

This experiment having demonstrated the advantages of fine crushing, arrangements were made for the new 120-stamp mill, at present in course of erection, to be inside amalgamation mortars with fine screens, for direct treatment of tailings by cyanide.

Another accumulation of material awaiting cyanidation, is the huge slimes heap, which will average about 2½ dwt. per ton. It was thought at first that this material, like the South African slimes, would be untreatable; but though 95 per cent. of the sands will pass a 90-mesh screen, it is quite tractable to the ordinary percolation, the last wash water only requiring to be drained off by vacuum to get the sands dry, and yielded a fair profit, even after allowing for the extra expense of tramming it a long distance to the present small plant, the cost then working out about 2*s*. 10½*d*. per ton, (exclusive of royalty and depreciation); but with a new 4,000-ton plant—shallow 4-ft. vats—being erected alongside the heap, I fully expect the cost will not greatly exceed 2*s*., thus yielding a good profit from an otherwise waste material.

This slimes sand is only treatable when dry and weathered, as on attempting the percolation of some recently discharged from the pits, they formed one mass impervious to the liquors, and had to be discharged untreated.

Champion Reefs (Mr. A. W. Jolly in charge) has been running mostly on tailings direct from the mills; but owing to the coarse screens in use (No. 5 needle, 20-mesh), the extraction is not so good as it would otherwise be with finer screens. It has averaged so far about 56 per cent., with a consumpt of 1 lb. pure KCn per ton; but it is likewise noted here that the dried and weathered tailings give a better extraction (up to 64 per cent.), with less consumpt of cyanide, than when treated fresh from the settling pits.

Nundydroog are treating sands direct from the mill; but owing to an increasing amount of pyritic material in the ores of the northern end of the field, the cyanide consumpt is somewhat greater, reaching as it does to 2½ lb. on the goldfields property.

The method of cleaning up and refining of the zinc-box slimes is such as is usually carried out elsewhere, *viz.*, screening through a 30-mesh or coarser screen, draining off the liquor by vacuum (steam ejector), drying, roasting with or without a small percentage of nitre, and fusing in No. 60 plumbago crucibles, generally two in a box furnace of firebrick with coke, and forced draught in the ashpit.

The exception, as mentioned previously, with the Mysore Company's precipitate, was the retorting of same and recovery of a large quantity of mercury. The presence of mercury in the zinc boxes generally leads to the production of much floured, and brittle, zinc, often chocking up the two top compartments, this being dissolved in sulphuric acid before retorting. When I arrived here first, in 1895, the usual practice, owing to want of a proper clean-up room and roasting furnace, was to mix the retorted slimes with fluxes and smelt direct, producing a large quantity of very base bullion, at the same time leaving several ounces, in shape of shots of metal, in the slag.

This, however, was always recovered by crushing and panning. Such a bullion as produced above gave an analysis of:—

| | Per Cent. |
|---|---|
| Gold | 56·40 |
| Silver | 3·00 |
| Lead | 2·40 |
| Copper | 19·60 |
| Zinc | 18·00 |
| Nickel | 0·10 |

The copper and nickel comes from the "Pyrrhotite in" the ore, one sample giving 0·25 per cent. of the latter metal.

Having had a proper roasting and smelting house built since, I found that roasting with nitre, while getting rid of nearly all the volatile zinc, still left the copper more in evidence than ever, but producing a slag very much more free from shots of metal, and lower in assay value. The bullion analysed :—

| | Per Cent. |
|---|---|
| Gold | 49·50 |
| Silver | 4·60 |
| Lead | 4·29 |
| Copper | 38·21 |
| Zinc | 2·10 |
| Nickel | 0·22 |

Roasting with excess (20 per cent.) nitre and trying to flux off the copper; with an acid slag, only resulted in bringing the bullion up to—

| | Per Cent. |
|---|---|
| Gold | 57·8 |
| Silver | 4·8 |

so I hit upon the following method, which entirely gets rid of the troublesome copper, at the same time it cheapens cost of clean-up, owing to the immense saving in fluxes, crucibles, &c.

The retorted and dried slimes are mixed with 10 per cent. of nitre and roasted at a bright red heat. When cold they are boiled in an enamelled iron boiler with dilute sulphuric acid (1 : 2), which in course of about 30 minutes removes all the copper as copper sulphate ($CuSO_4$); allowed to settle, the bulk of the copper sulphate siphoned off, and the residue washed with hot water, and drained on the vacuum filter. Pressing the slimy residue in a rough-and-ready-made screw press, gives a product at once ready to be gently dried on the roasting plate, and if care is exercised not to carry the drying too far, there need be very little loss from dusting. The former retorted slimes have now lost 69 per cent. of their weight, and are readily fluxed with about 35 per cent. borax, 15 per cent. soda, and 10 per cent. sand. Such a slag was absolutely free from shots of metal, and assayed 25 oz. per ton, or 6 dwt. on the total quantity of slag produced.

The bullion analysis gave : —

| | Per Cent. |
|---|---|
| Gold | 81·30 |
| Silver | 6·90 |
| Lead | 2·74 |
| Copper | 6·78 |
| Zinc | 0·40 |
| Nickel | 0·12 |

With a proper filter press, washing the gold mud under pressure, I am confident of getting rid of the whole of the copper, and bringing the bullion over 900 fine gold and silver, with a monthly saving in cost of production, against the old method, of :—

| | £ | *s.* | *d.* |
|---|---|---|---|
| Borax and soda | 1 | 18 | 0 |
| Crucibles and coke | 2 | 3 | 0 |
| Refining charges at 4*d.* per oz. | 3 | 9 | 0 |
| Melting at ¼*d.* per oz. | | 4 | 4 |
| | 7 | 14 | 4 |
| Less excess sulphuric acid used | | 5 | 4 |
| A total saving | 7 | 9 | 0 |

The bullion as produced at Champion Reefs runs about 75·0 per cent. gold and 10·0 per cent. silver, the principal contamination being zinc, with only small quantities of lead and copper; nickel has also been noted.

As Mr. Arthur C. Claudet fully points out in his discussion on "Notes on the Treatment of Zinc-box Precipitate" (by R. G. Brown, Inst. of Mining and Metallurgy, Vol. 4; also this Journal, 1897, 5), the necessity of obtaining as pure a bullion as possible must not be over-looked, "though it would not be of much importance if it were only a case of a few hundred ounces." Taking the output of this small plant, however, where the production at present is only about 300 oz. per month, a saving of 7*l.* 9*s.* monthly is a step in the right direction, and when we reach the thousand ounces the gain will be proportionately greater.

That the zinc in use on this mine is purer than the case quoted by Mr. Claudet is evident at a glance; but with still purer zinc, and the removal of all the copper by the foregoing process, that desideratum "pure bullion" would be an accomplished fact.

## NOTES ON THE TESTING OF GOLD ORES, &c., IN RELATION TO THEIR TREATMENT BY THE CYANIDE PROCESS.

BY CHAS. J. ELLIS, F.I.C.

In testing gold ores, &c., with a view to their practical treatment by any particular process, the main object is to arrive at a decision, firstly, as to whether that particular process is the most suitable and economical one for their treatment; and, if so, secondly, as to the best way to apply the process to secure the greatest profit from their treatment by it.

In the following notes I have endeavoured to give an outline of a course of testing in connection with the cyanide process, applicable to the general run of ores, &c., but without attempting to meet the requirements of really exceptional cases, as doing so would entail extending them far beyond the limits of a paper of reasonable length.

Whatever the material to be tested happens to be, before proceeding to the actual testing it is of course essential to find out by assay whether it contains sufficient gold or silver to pay the cost of treatment at all. It is unfortunately too often the case that so-called gold mines contain stone which would be much more suitable as road metal than to go to the mill for the purpose of getting gold out of it which it does not contain, as no process has yet been found good enough to effect this; though by means of modern methods of treatment, ores, and more especially tailings, which had formerly been considered too poor to be dealt with at all, can now be made to yield a more or less substantial profit. No hard-and-fast line can be drawn in regard to the minimum gold contents which will pay for treatment; this depends so much upon the character of the material to be treated; but I may mention that there are now cases where tailings, &c. containing little more than 2 dwt. of gold per ton are being treated by the cyanide process direct, without previous concentration, and yielding a fair margin of profit.

Referring now to testing specially in relation to the cyanide process.

If the assays have shown the material to contain a reasonable amount of gold and silver, or at least of one of them, the next step is to carry out a few preliminary tests, of which a determination of the quantity of acidity, if any, and the consumption of cyanide during short contact of the material with weak cyanide solution, with or without previous alkaline treatment according to the result of the acidity test, are perhaps the most important. The method of making these tests requires little explanation; for the former a few hundred grains of the ore is made into a pulp with some water, and the pulp tested with (say) blue litmus paper; if this is reddened, soluble acidity is present, and weak standard soda is run in until the neutral point is arrived at. After this point, however, though blue litmus is no longer reddened, red litmus may not be turned blue by a slight further addition of the soda solution, and in this case soda is again run in until the pulp does turn red litmus blue, even after standing for some time. This second quantity of soda is a measure of what goes by the general name of "latent acidity," and this is due to the presence of various constituents which, though not acid to litmus themselves, have the power of combining with and neutralising alkali. If the material is found to be free from acidity, 500—1,000 grains of it are placed in a bottle with a similar or greater quantity of weak cyanide solution of known strength, and shaken for 10 minutes or so, and the cyanide remaining undecomposed determined. If, however, acidity had been found, and unless this was in abnormal quantity, the material would be first shaken up, with free access of air, for a short time with a slight excess of soda solution, and then the cyanide added, and the test proceeded with as before.

In the case of excessively acid material, it is usual to try to wash out as much of the acidity as possible with water before proceeding to the above tests, testing also the acidity of the washings.

These tests having been made, it is then the practice to carry out a few small preliminary trials; for instance, agitating 1,000 grains or so of the material in each case, with or without previous alkaline treatment, for some hours (generally overnight) with various quantities of weak cyanide solution of different strengths, the material being ground to various degrees of fineness, or, if received ground, usually in the first instance just as received. From the results of a few such preliminary trials, one can generally at once decide to which of the two following classes the material belongs:—

A = Ores, &c. from which the gold is readily extracted by cyanide solution, with moderate cyanide consumption, and which are not exceptionally clayey.

B = Ores, &c. from which the cyanide process, applied to them direct under normal conditions, does not effect satisfactory extraction of the gold, or in the treatment of which the consumption of cyanide is abnormal, or which are exceptionally clayey, &c., &c.

Having broadly classified the ores, &c. as above, the following is an outline of methods usually adopted for their general examination:—

*Class A.*—If the material has been received unground, it is advisable to retain a portion of the sample in that condition for further reference, and to grind the remainder to several degrees of fineness; for instance, say, part through a 30-mesh screen, part through a 40, and part through a 60. A series of percolation tests are then carried out on these various portions, if necessary with preliminary alkaline treatment, using cyanide solution of the strength selected (the cyanide consumption in the preliminary trials serving as a guide for the selection). This is usually from about 0·1 per cent. to 0·3 per cent. for this class of material, and the quantity employed varies from about $\frac{1}{3}$ to 1 part for each 1 part of material to be treated. The course of the trials is followed by, in the first instance, carefully assaying an average sample of the material as charged into the percolator, and from time to time assaying and testing samples of the solution. The percolation may be by upward and downward flow, or entirely by downward flow. For small trials, however, especially on dry material which does not require preliminary alkaline treatment, it is perhaps best that the first passage of the solution should be upward and thereafter downward, say about once each 24 hours, for the remainder of the trial, as by this method channelling is best avoided, and a uniform flow of solution between the sand particles more or less insured. The normal time of treatment is about three to four days; but of course if the extraction is not fairly complete in that time, the treatment is prolonged until such is the case.

Sometimes it may be found that a better extraction is obtained by a double treatment, *i.e.*, after the treatment has proceeded for a certain length of time, the residues are washed, discharged, re-charged to the percolator, and treated a second time with fresh solution.

The apparatus required for making the above test is of the simplest description, and consists of a pair of small vats or vessels of suitable size (a sound barrel cut in half would do), one of which, (the percolator) is fitted with a false spar-bottom covered with filter cloth, and the other (the receiver), which may be smaller, without such a false bottom. A hole at the bottom of the side, and of course under the false bottom in the case of the percolator, is fitted with a rubber bung carrying a piece of glass or iron tube, to which is attached a piece of rubber tubing of suitable length, provided with a screw clip for regulating the flow. For upward percolation the rubber tubes of the two vats are connected by a piece of glass tubing, and the method of using this apparatus is so obvious as to require no explanation.

In addition to the above, a small zinc extractor box, or even a few suitable sized wide-mouthed bottles fitted up on the wash-bottle principle and filled with zinc thread, for use in precipitating and recovering the gold brought into solution, completes the apparatus.

If the material to be tested consists of tailings, or other material received ground, it is always advisable to try in the first instance to obtain satisfactory extraction from it in the condition as received, as to regrind more finely would generally add very considerably to the total expense of treatment of such material in practice, and in some cases even make it prohibitive. If, however, owing to unusual coarseness of the material, it is found to be impractical to reduce the gold and silver contents of the residues to the desirable small amount, the material, and more especially the residues, should be graded and the various gradings assayed to locate the gold and silver, and if, as is very frequently the case, this shows that almost the whole of it is retained locked up in the coarser particles of the residues, and a further trial proves that these yield it up readily on being more finely ground, the necessary data are available for calculating whether the expense of regrinding the material as a whole, or the coarser part separated from it, would be profitably incurred.

In the case of rich material such as concentrates the treatment, if by percolation, has often to be for a prolonged period, say, for as as much as a month or so, before the gold contents of the residues has been reduced to the desirable small amount. In the earlier days of the process it was usual to treat such material by the quicker agitation method, and this is still the practice in certain cases, but where the percolation treatment, even if prolonged, is found to extract the gold efficiently, it is in most cases preferred, as being the more economical to adopt. In testing such material as this, it is, however, always necessary, or at least advisable, to carry out a series of agitation trials, as well as those by percolation, in order that data may be obtained on which to base an opinion as to the relative merits of the two methods for the particular material.

In many cases it will be found advisable to further carry out one or more concentration tests in order to locate the gold, and to determine what proportion of the total exists in the pyritic concentrates, and what in the lighter sands, &c., &c.; and trials on the separated concentrates and sands might be carried out with advantage. The results obtained will serve as a guide regarding whether the material should be treated as a whole or concentrated, and the concentrates treated by themselves.

If the material has been correctly considered from the preliminary tests to belong to Class A, a carefully selected series of trials and experiments, as suggested above, can hardly fail to give sufficient data as a basis for a provisional decision as to what are likely to prove the best general conditions under which the process should be applied in practice for its treatment, and the most suitable form of plant to adopt, in order that the greatest profit may be secured, leaving any slight modifications to be decided upon from the results of actual practical working.

*Class A.* may be taken to comprise a very large proportion of the various kinds of gold ores, &c. more commonly met with, and experience in the examination and practical treatment of such material should be acquired in a comparatively short time by one possessed of a fair general knowledge of chemistry and metallurgy.

*Class B.* comprises ores, &c. which require a somewhat more searching investigation than those of Class A. before a decision can be arrived at as to the suitability or otherwise of the cyanide process for their treatment, or as to the best method of applying it.

It would be out of the question within the limits of a paper to attempt to enumerate and discuss all the possible characteristics of materials of this class which tend to cause the direct treatment of them by the cyanide process, applied under normal conditions, to yield more or less unsatisfactory results, in any case until they have first undergone some preliminary treatment to prepare them for it; the following may, however, be mentioned as examples of those more commonly met with:—

(1.) A considerable part of the gold exists irregularly distributed throughout the material in the form of comparatively large particles, or in the case of tailings a considerable portion of the total gold exists in the form of comparatively large particles of amalgam left in them.

(2.) Some base constituents of the material exist in a form which allows of their ready attack by cyanide, causing abnormal consumption, and simultaneously with this soluble sulphides are frequently found to be formed in the solution, and these alone, or possibly as well as the products of the action of the cyanide on the base constituents, seriously retard solution of the gold and silver.

(3.) A considerable part of the gold exists in a state of combination, more or less loose, with antimony, tellurium, bismuth, or other element of like nature.

(4.) The gold exists associated with a proportionately very large amount of silver in certain forms.

(5.) The material is so clayey or slimy as to prevent, or at least seriously retard, the filtration of solution through it.

Indications of these several characterictics will have been obtained during the course of the preliminary investigation, and the few remarks below in reference to each of them may perhaps serve as a guide in regard to the further investigation of the various ores, &c. in which they occur.

(1.) Material of this character is first treated with mercury to remove as far as possible the coarse and amalgamable gold, this may be done either by the ordinary methods of amalgamation, or by forced amalgamation, *i.e.*, grinding in a small wheeler, or similar, pan with mercury and weak caustic soda solution. The residues are then treated by either the percolation or agitation method of applying the cyanide process, and if the only difficulty to direct cyanide treatment has consisted of the one referred to under this heading, the effect of this double treatment will usually be a more or less satisfactory extraction of the gold, and a comparatively few trials under varied conditions carried out in this way, will generally indicate what are likely to be the most suitable conditions to apply in practical working.

In addition to double treatment trials as indicated above, a few might also be carried out by combined treatment simultaneously with mercury, or amalgamated copper plates, and cyanide. It will generally be noticed that in this latter case a less proportion of the total gold will be found amalgamated, and a greater proportion in the cyanide solution (owing to the solvent action of the cyanide on the gold amalgamated in the first instance) than when the double treatment is resorted to. In fact, the mercury appears to act as a carrier between the coarse gold and the cyanide.

It may in some cases be found preferable to treat first by cyanide and then the residues by amalgamation methods, but unless much float gold is present the other way about is the more general.

(2.) Apart from abnormal consumption of cyanide caused by the material being acid from the effect of weathering of the contained pyrites, and which in such a case can generally be prevented by preliminary alkaline treatment, the most usual cause of this trouble is the presence of certain compounds of copper, iron, &c., the physical condition in which they exist, as well as the chemical composition of the compounds, being an important factor in regard to the ease with which the base metal is attacked by cyanide; for example, marcasite is much more readily attacked than ordinary iron pyrites. If the preliminary trials have shown abnormal consumption of cyanide, usually coupled with more or less poor extraction of the gold and silver, and often with the formation of soluble sulphide, it is advisable to at once determine definitely the cause of the consumption, and perhaps also to make a qualitative analysis of the material.

The method of determining the cause of cyanide consumption is explained in a paper on "Residual cyanide solutions" which I read before this Section a year ago (see this Journal, 1897, 116). If, say, copper is found to be the cause of the trouble, attempts may then be made to remove the portion of it which is so readily attacked by cyanide by preliminary treatment with weak acid, or by ferric chloride solution, &c. Where this copper exists almost entirely as carbonate (examples of which are met with especially in the case of certain Chili ores) such treatment may be found to be quite effective and practical, and the material after this treatment to be in a satisfactory condition for treatment by cyanide, after removal of the acid, &c.; in most cases, however, it is found to fail, as, though this treatment may remove some of the copper, it tends to render that still left in the material even more easily attacked by cyanide than it was before such treatment.

For material of this character as a whole, however, the most generally effective course of treatment seems to be to roast it either partially or fully (in the former case leaching out any soluble compounds after roasting), and then to treat with cyanide, when the extraction of the gold will often be found to be satisfactory and the consumption of cyanide normal, or at least, greatly reduced.

If, however, a concentration test should show that the greater part of the gold and silver can be obtained in the concentrates, it may be found preferable to concentrate in the first instance, and to treat the concentrates only, either raw, or after roasting if this is found necessary or advisable.

In certain cases coming under this heading, it is found that, without the cyanide consumption being really abnormal, sulphide has been formed in solution to a sufficient extent to seriously retard or prevent satisfactory extraction of the gold and silver by means of cyanide; in such cases, the addition of certain salts such as those of lead, ferricyanide, &c., which decompose soluble sulphide, is often found to be very beneficial, and to cause the extraction to be satisfactory even from the raw material. Such addition is also sometimes found to increase the extraction of silver where this exists as sulphide.

(3.) Material of this character is somewhat frequently met with, and, especially of late, metallurgists have begun to turn their attention particularly to the question of finding out the most efficient method of dealing with it for the extraction of gold from it. Occasionally ores consisting almost entirely of stibnite are found to be rich in gold, and in such cases it is most desirable that a method of treatment should be available for separating out both the gold and the antimony efficiently from them. In some of the few examples of this sort which have come under my notice I have found that though the cyanide process, applied under exactly normal conditions, has failed to effect satisfactory extraction, yet if the ore has been exceptionally finely ground, and then agitated for a more or less prolonged period with solution somewhat stronger than usual, the gold has been fairly completely removed, and the consumption of cyanide moderate.

It is, however, more frequently the case to come across ores of this character containing only comparatively minute quantities of antimony, tellurium, &c., and in which only a portion of the gold appears to be locked up in combination with these, the remainder, say perhaps three-fourths of the total, being readily soluble in cyanide solution, and in such cases a preliminary roasting of the ore at a moderate heat is often effective in rendering the gold as a whole more completely soluble in this solution.

Arsenic, though in many of its properties an element of similar character to antimony, does not appear to have this property of combining with gold in at all the same degree, if at all, as antimony has, and the cyanide process is generally found to be just as efficient in extracting gold from arsenical pyrites as from ordinary iron pyrites.

(4.) Though in many cases the silver, as well as the gold, is able to be extracted satisfactorily from ores by means of the cyanide process, this process is certainly more generally suitable for the extraction of gold than of silver; and from true silver ores, or those in which the main value lies in the silver contents, it frequently is found to fail, as far as the silver is concerned, though it may succeed in extracting a large percentage of the comparatively small quantity of gold often present in such ores. Its use in relation to the treatment of such ores may perhaps be taken to consist mainly in dealing with the residues from them, containing at least part of the gold, after they have been in the first instance treated by some one of the well known processes for silver extraction. However, in certain cases it may be found advisable to treat such material after chloridising roasting by the single operation of leaching with cyanide solution for the extraction of both the silver and the gold where it is found possible to do this efficiently and economically by this means.

(5.) With material under this last heading the difficulty is often simply a mechanical and not a chemical one, the very fact of the extremely fine state of division in which it exists, and consequently also the gold it contains, rendering the gold as a rule most readily and quickly soluble in weak cyanide solution; in cases, however, where as well as being of a slimy character, the material is of a composition which introduces chemical difficulties, it would generally require to be roasted before treatment by cyanide.

In regard to the mechanical difficulty of filtration, in the early days of the application of the cyanide process attempts were made to get over this by the use of the filter press, but from the particular circumstances of the case this has been generally found to be a more or less unsatisfactory or cumbrous method, and it has, to a great extent at least, been now abandoned. The method now most generally adopted consists broadly of agitation of the slimes for a short time with cyanide solution by any suitable means and then allowing the solid particles to settle through a comparatively large volume of very weak solution, decanting the supernatant gold solution, and, if necessary, washing the pulp in a similar way by settling and decantation.

I first adopted this method five years ago for the treatment of a lot of some 30 tons of almost pure clay containing gold. Having in the first instance tried to treat this material by the ordinary percolation method and found it to be almost impervious to solution, even with the aid of suction, it struck me that, if solution would not pass between the particles of the clay in the usual way, the next best course to adopt was to allow the clay particles to descend through the solution, leaving the greater part of their gold in it, partly dissolved during their passage through it.

In the treatment of this 20 tons the clay was first agitated for a few hours with weak cyanide solution and then discharged into about four times its weight of very weak cyanide solution and allowed to settle, this taking about a day to a day and a half, after which from $\frac{3}{4}-\frac{4}{5}$ of the total solution was able to be decanted fairly clear, and this was passed through a zinc extractor. The creamy sludge was then stirred up with about two to three times its volume of water, or residual solution from a former charge and again allowed to settle, and the clear supernatant solution decanted and passed through the extractor, the sludge being then discharged. In some cases it might of course be advisable to give the sludge a second washing by decantation. In this way the greater part of the gold was obtained from this clay in which it was found to exist in such a fine state of division as to allow of its being dissolved very quickly by weak cyanide solution. For several of the charges I mixed a small quantity of slaked lime with them, and this to some extent aided the settling, and also gave a clearer solution on decantation.

As far as I am aware, that was the first time when slimes were treated on a practical scale by the settling and decanting method of applying the cyanide process; some two years later however, *viz.*, in 1895, this same method, with certain modifications to suit the greater extent of the operations, began to be practically adopted for the treatment of slimes, and is now largely in use in various parts of the world, and especially in South Africa; where, for example, it is employed by "The Jumpers Gold Mining Company," "The Crown Reef Gold Mining Company," and others for the treatment of enormous quantities of slimes.

The solutions from the treatment by this method are naturally, as a rule, very weak both in cyanide and gold, and for the precipitation of the gold from them the electrical method of precipitation appears to be generally preferred to the zinc method. There are still, however, differences of opinion on this point, especially if certain slight modifications are introduced into the ordinary method of precipitation by zinc to meet the altered conditions of the solution, for example, increase of the capacity of the zinc extractors, or, what is the same thing, decrease in the normal rate of flow of the solution for a given size of extractor. This subject is, however, outside the scope of the present paper and, I therefore, do not propose to enter upon a discussion of it here.

In testing clayey or slimy material it is first desirable to ascertain whether the clayey part, where it does not wholly consist of this, contains a really substantial amount of gold. This is done by making some of it into a pulp with some water and then washing away the slimes with a gentle stream of water, collecting, settling, and assaying these, and also assaying the more sandy part left behind. If this shows the slimes to contain only an insignificant amount of gold, they can be removed by any suitable wet or dry method, and the heavier sands then dealt with by the usual percolation method. If, however, this is not the case, the slimes, or the material as a whole, would have to be dealt with by the agitation, settling, and decanting method indicated above. The presence of lime, common salt, or other more or less inert salts, often greatly aids the settling of the sludge.

In certain cases, however, it will be found that a dehydrating heating brings the material into a condition which allows of its treatment being carried out by the ordinary percolation method within a reasonable time, with or without the aid of suction, and this may sometimes be found to be preferable to adopting the settling and decanting method.

In concluding this paper, I may remark that, in spite of having made it a more lengthy one than was my original intention, I have only been able within its limits to present a very imperfect sketch of the wide subject with which it deals, and have necessarily had to leave many more or less important points quite untouched. I have, moreover, not attempted to give any definite instructions regarding the methods of carrying out the various trials and experiments indicated, the details of most of which would be more or less obvious to anyone with a reasonable amount of general laboratory experience, and to have done so would have added very considerably to the length of this paper. These few notes may in fact be taken to be simply a programme or guide to a course of investigation of gold ores, &c. more commonly met with, and as such may, I hope, prove to be of at least some little use, especially to some of the younger members of this Society who may purpose specialising in the particular branch of chemical industry to a section of which they refer.

## TEN YEARS' PROGRESS OF THE CYANIDE PROCESS FOR THE EXTRACTION OF GOLD.

BY G. T. BEILBY.

It is not necessary to remind this Section of the Society of Chemical Industry that the cyanide process for the treatment of gold ores and tailings was invented and worked out in Glasgow, or that the first public description of the process and its results was given by Mr. J. S. MacArthur, one of the inventors, at the Sectional Meeting held at Glasgow on the 31st March 1890 (this Journal, IX., 267). The first MacArthur-Forrest patent is dated 19th October 1887, so that the process is now 10 years old. During these 10 years it has become such an all-important factor in the gold-mining industry, and the busy metallurgist of to-day takes it and its results so entirely for granted, that even those of us who remember its earliest beginnings are apt to forget the attitude of indifference or scepticism with which the new process was received.

Now that the cyanide process has proved itself an epoch-making invention, it is peculiarly fitting that it should be in this Section that a retrospect of the work and progress of the past 10 years should be made. Through the kind co-operation of the officials of the Cassel Company and its associated companies, I am in possession of a number of statistics as to the progress of cyanide extraction which have not before been collected and published. These I propose to lay before you as briefly as possible after I have sketched the main outlines of the development of the process.

### *Development of the Process.*

The primary idea of the process is extremely simple: it is to dissolve out the gold from the ore by a dilute solution of cyanide of potassium and to precipitate it again on a clean zinc surface. As an operation to be performed on a small scale, and with pure materials in the laboratory, nothing could be much simpler. The only theoretical point still in doubt 10 years ago was whether or not oxygen was necessary for the solution of the gold. This point may be said to have been finally settled by the researches of Maclaurin (J. Chem. Soc. **63**, 724) in 1893, when he definitely proved that oxygen is necessary for the solution of the gold.

But when complex ores have to be treated in apparatus in which the unit is not measured by grains, but by hundreds of tons, the altered conditions are found to have so completely altered the problem that the experience gained by experiments made with pure materials in beakers in the laboratory are found to carry the cyanide chemist only a very short way towards the successful working at the mines. The papers which immediately precede this (Pitbaldo and Ellis) supply ample illustration of this point. The large mass of ore or tailings, the great volume of solution dealt with, and the continuous use of the same liquors, with the consequent accumulation of impurities and decomposition products in the liquors, introduce so many variable factors that skill and attention are constantly necessary if good extractions are to be regularly maintained. As the process has made its place mainly through its application to low-grade tailings which were valueless before its advent, the cyanide manager has from the outset been faced by the necessity of obtaining maximum extractions of extremely small quantities of gold along with such moderate costs as would still leave a fair margin of profit on the turnover. The principal economies have come from two directions: chemically, by the reduction of the quantity of cyanide used; and mechanically, by improved handling of the tailings in larger plants.

In the early years of the process, the solutions used contained from 0·5 per cent. to 2 per cent. of cyanide, but gradually the strength has been reduced till 0·1 to 0·2 per cent. is commonly used, and, in some instances, even weaker solutions. The inventors began with the idea that agitation of the ore or tailings with the solution would be necessary, but it was soon found in practice that percolation was practically as efficient, as well as more cheaply carried out.

Where very large vessels can be used, it is economically possible to allow the solution to act for weeks if necessary, the only movement requiring power being the pumping of the solutions up from the sumps to top of the vats.

Percolation with both upward and downward flow through the tailings has been tried, but the downward flow is now generally adopted.

The application of a partial vacuum is sometimes used to quicken the flow of the solution through the mass of tailings, in cases where the tailings are of such a nature as to retard the flow of the solution unduly.

True slimes, owing to their fine pulpy condition, cannot be successfully treated by percolation; but in 1893, Mr. C. J. Ellis succeeded in treating certain slimes on a practical scale by mixing them first with normal cyanide solution till the solution of the gold was more or less complete, then turning the pulp over into a large volume of weak solution or water, and allowing it to settle. This process, slightly modified, is now pretty generally adopted where slimes have to be dealt with.

Double treatment is now sometimes resorted to, that is to say, the ore or tailings, after a first treatment in one vat, is drained and turned over into a fresh tank, and again treated with cyanide solution. Under this treatment, a 5-dwt. ore might give up 3 dwt. to the first treatment and 1½ dwt. to the second.

The turning over of the ore into another tank may have two beneficial effects: it may ensure that no portion of the mass shall escape the action of the circulating solution, and it may more effectually aerate the mass and supply the necessary oxygen for the reaction.

In the more economical handling of the ore or tailings, the increased size of the tanks has played an important part. In the earlier installations, the tanks were of 20 to 40 tons capacity; now they have been increased to 500 tons. With the increase of size, it became economical to apply labour-saving appliances for filling and emptying. While tanks were small, they were emptied over the side by shovels, but as the size and depth increased, side or bottom doors had to be provided; the latter are generally preferred.

Attempts have been made to improve the process chemically by the addition of other reagents to the cyanide solution. Among these may be mentioned:—

> Lead salts, for the removal of soluble sulphides.
> Oxidising agents, such as ferricyanide of potassium or peroxide of sodium.

These reagents have never had more than a limited and local application. If a cyanide is used which is free from alkaline sulphides, there is no need to apply correctives for sulphides; and if sulphides derived from the ore accumulate in the solutions, the chemist on the spot will have to deal with each case as it arises.

The bromo-cyanogen process of Messrs. Sulman and Teed, has been so recently described in the London Section (Vol. 16, p. 961), that it is only necessary to remind you that the object of that process is to render oxygen unnecessary for the solution of the gold by cyanide, by the addition of bromine to the solution.

Up till now the simple cyanide solution holds the field, and so long as every year continues to add to the economy of its application, it is not likely to be superseded.

For the precipitation of the gold from the cyanide solution, various new suggestions have been made. Aluminium has been proposed instead of zinc, but the formation of the gelatinous hydrated oxide interferes with the working of the precipitating boxes, and neutralises any other supposed advantages.

For the Siemens and Halske process of electrolytic deposition on lead anodes, it is claimed that a more perfect precipitation from very dilute solutions is obtained, and that the waste of cyanide of potassium through excessive solution of zinc is avoided.

With regard to the zinc precipitation, it has been proved that the gold can be perfectly precipitated even in the most dilute solutions if the precipitation has first been started by passing a solution of normal strength over the zinc, so as to produce the gold-zinc couple in the box. It is also necessary to pass the solution very slowly through the boxes.

Messrs. Sulman and Teed have proposed to revive the use of zinc fume for precipitation in a special apparatus (this Journal, Vol. 16, p. 961) as an improvement on the MacArthur-Forrest method; but a proper comparison of the two methods can only be made after the younger process has worked for a length of time on an extended scale.

### *Costs.*

The cyanide process made its first practical success in the Transvaal, and as that district has continued to take the lead in cyaniding up to the present time, the costs of working there may be taken as fairly illustrating the best work done.

In 1890, contracts were made by the Inventors' Syndicate to work tailings in the Transvaal at a cost not exceeding 20*s.* to 25*s.* per ton; the actual cost turned out to be 10*s.* per ton. In 1891, at the Robinson Mine, the working cost was 10*s.* per ton; a year later, in 1892, at the Langlaagte Estate, it was reduced to 4*s.* per ton; and in 1897 the record of 2*s.* 6*d.* per ton was reached. As has been already pointed out, these reductions have been brought about through reduced consumption of cyanide and through more economical handling of the tailings. In 1891 the average consumption of cyanide was about 2 lb. per ton of tailings; the consumption to-day is about ½ lb. per ton.

It is evident that the present very low cost per ton of tailings immensely extends the application of the process, so that tailings which formerly were absolutely unworkable have become an important source of profit. This is well illustrated in the case of the Indian mines, where tailings are being regularly worked for the recovery of 2 to 2½ dwt. of gold, or say 60 grains, at a cost of 3*s.* to 3*s.* 6*d.* per ton.

One consequence of the reduced consumption of cyanide per ton of tailings is that each ton of cyanide now does the work of four tons in the earlier days, so that the consumption of cyanide has not at all kept pace with the output of gold by the process. This effect was very noticeable in the Transvaal during the past few years, when the shipments of cyanide fell off considerably, while the gold output was steadily rising. This same effect is likely to be repeated in other parts of the world, where the consumption at present is extravagant.

As to the prospects of further reduction of costs, it is not easy to speak positively. It seems probable that what will happen will be that the general average of costs all over the world will approximate more and more to the present minimum, but that that minimum itself will not be much reduced. When only ½ lb. of cyanide per ton is used, any fall in the price of cyanide which is likely to occur cannot affect the cost by more than a few pence.

### *The Process in Africa.*

The existence of large accumulations of tailings in the Transvaal, together with their special suitability for treatment by cyanide, resulted in a more rapid and extensive adoption of the process there than in any of the other gold regions. So peculiarly favourable were these conditions to the process, that a few years ago there were experts who maintained that its success was due to the fortunate circumstance that suitable tailings had happened to exist in the Transvaal, and they predicted that as those accumulations became exhausted the process would sink into comparative insignificance again. The statistics before us this evening show conclusively that the cyanide process is neither a one-ore nor a one-district process, and the diagrams prepared from those statistics show that its extension and development are still proceeding so vigorously that we may safely predict for it many years of life and activity.

### *New Zealand.*

The process was introduced into New Zealand in 1889, but several years elapsed before it was taken up to any extent. Since 1893 the progress has been fairly rapid.

### *The United States.*

In the United States an important feature of the gold-mining industry has been the Customs mills which have existed at certain centres, and to which ores are brought by the mine owners for treatment and extraction of the gold. Prior to the introduction of the cyanide process, the ores and concentrates were treated at those mills either by amalgamation or chlorination. Of late years several large Customs mills have been started for the treatment of ores by the cyanide process, and the quantity of ore treated is steadily increasing. In a paper read before the American Institute of Mining Engineers in September 1896, Mr. George A. Pickard describes his experiences in visiting a number of these Customs mills. He states that a number of mills crush and cyanide the ore at a cost rather under 8*s.* per ton, the lowest cost he had heard of being 3*s.* 4*d.*, at the Mercur mill. He further estimates that nearly 200,000 tons of tailings were treated in 1895, producing bullion valued at 200,000*l.*

### *Australia.*

Owing to the enormous area over which the gold-mining districts of Australia are spread, it has been found impossible to collect accurate statistics in time for this evening's meeting. I can therefore only bring under your notice a somewhat general statement as to the progress in these colonies. The process is now successfully at work in Queensland, New South Wales, Victoria, and Western Australia. Western Australia, though the youngest of the fields, is already preparing to cyanide on a very large scale.

Queensland presents a feature of interest which calls for special mention. In most of the other regions cyaniding has been left to wealthy companies with extensive plant and a large turnover. In Queensland, on the other hand, cyaniding has been taken up as a poor man's process. In the district worked from Charters Towers there are at least 80 miners' syndicates, each consisting of four or five men who work with their own hands. Each of these little syndicates having pegged off its claim, erects a small cyanide plant, and thereafter proceeds to buy up and work tailings from the adjoining mine. Some of these small syndicates may happen to contain one man skilled in chemistry; but as a rule they have neither the skill nor the appliances for the practice of assaying or smelting, so they turn over the gold slimes from their zinc boxes to the head-quarters of the Australian Gold Recovery Company at Charters Towers, where they are worked up into bullion. As an illustration of the practical results of this system of working, I may quote one case which I heard of recently, in which five men, with a joint capital of 600*l.*, fitted up a small plant and started to work the tailings. Within six months of their raising the capital, they had produced 1,400*l.* in bullion and divided their first dividend of 300*l.* It is very interesting to find the most recent chemical process for the extracting of gold being worked on the same primitive system of combined effort as the original alluvial deposits in Victoria 50 years ago.

It is believed that there is a great future for the process for the extraction of such telluride ores as are not sufficiently rich to pay for carriage and smelting. Of such ores, containing, say, 1 to 2 oz. of gold, there are immense quantities in Western Australia which would pay handsomely, even if they only yield up 50 per cent. of their gold to cyaniding.

*Gold Production by the Cyanide Process in the Transvaal.*

| | Oz. |
|---|---|
| 1891 | 35,000 |
| 1892 | 175,000 |
| 1893 | 330,000 |
| 1894 | 600,000 |
| 1895 | 655,000 |
| 1896 | 770,000 |
| 1897 | 825,000 |
| | 3,390,000 |

*Gold Production by the Cyanide Process in the United States.*

| | | Oz. |
|---|---|---|
| 1895 | say | 75,000 |
| 1896 | „ | 135,700 |
| 1897 | „ | 190,000 |
| | | 400,700 |

*Gold Production by the Cyanide Process in New Zealand.*

| — | Oz. | Tons treated. |
|---|---|---|
| | | Approximate. |
| 1893 | 38,160 | 28,066 |
| 1894 | 80,728 | 50,816 |
| 1895 | 68,354 | 64,076 |
| 1896 | 119,091 | 63,967 |
| 1897 | 263,076 | 110,161 |
| | 575,109 | 317,056 |

*Gold Production by the Cyanide Process in Mexico.*

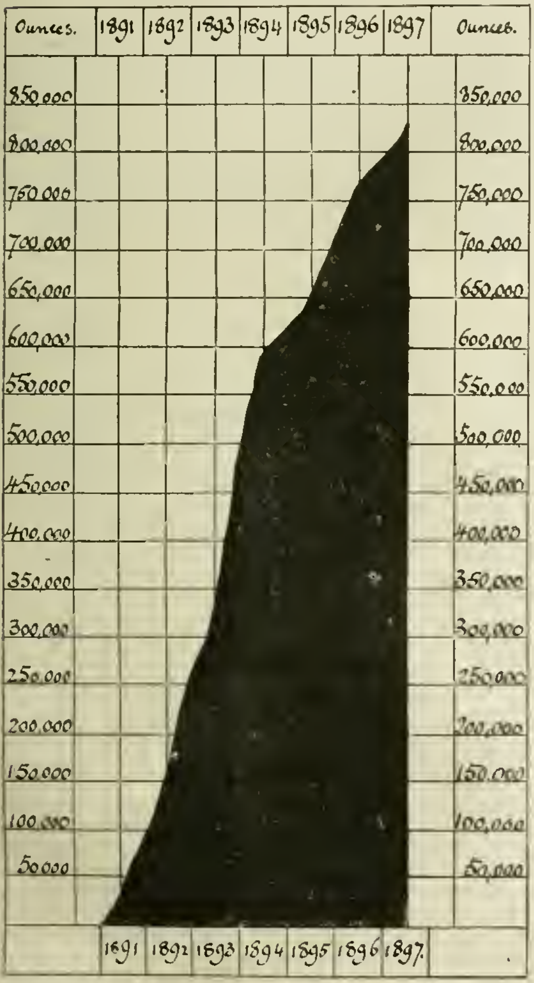
| — | Oz. | Tons treated. |
|---|---|---|
| 1895 | 4,053 | 18,730 |
| 1896 | 9,931 | 48,839 |
| 1897 | 10,207 | 45,670 |
| | 24,191 | 113,239 |

*Gold Production by the Cyanide Process in India.*

Process first put into operation at Mysore mine, November 1894.

| — | Quantity of Tailings treated. | Extracted. |
|---|---|---|
| | Tons. | Standard Oz. |
| Nov. and Dec. 1894 | 1,004 | 158 |
| 1895 | 18,065 | 3,177 |
| 1896 | 51,532 | 5,364 |
| 1897 | 132,871 | 18,798 |
| | 203,472 | 27,497 |

The diagrams which follow show the progress of the process in these countries.


The Transvaal.

The world's production of gold for 1897 is estimated at 11,678,000 oz.

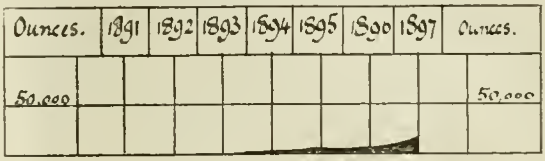
The total production of gold by cyanide for 1897 is estimated at—

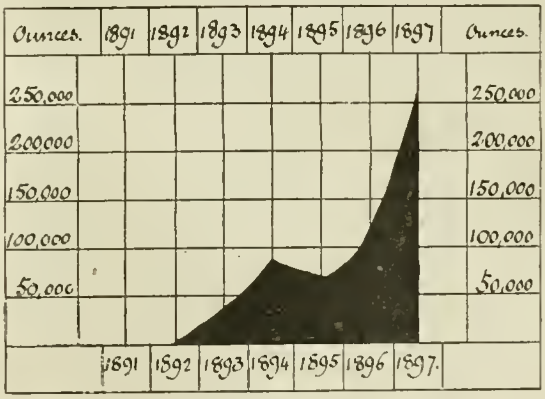
| | | | |
|---|---|---|---|
| Africa | say | 825,000 | oz. of bullion. |
| Australia | „ | 308,000 | „ „ |
| New Zealand | „ | 263,000 | „ „ |
| United States | „ | 190,000 | „ „ |
| India | „ | 18,800 | „ „ |
| Mexico | „ | 10,200 | „ „ |
| Other countries | „ | 5,000 | „ „ |
| | | 1,620,000 | „ „ |
| | = | 1,215,000 | oz. of fine gold, |
| | or | 10·4 | per cent. of the world's production. |

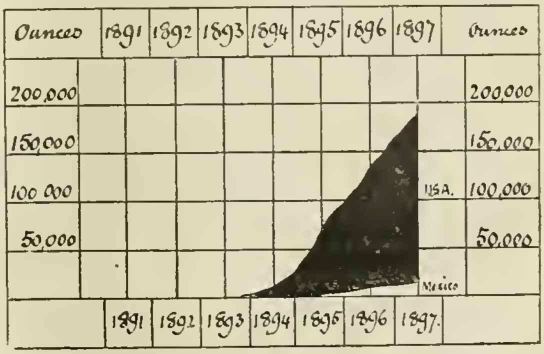
### *The Future of the Process.*

While the general experience of the past 10 years has set certain definite limits to the application of the process, the figures before us give strong indications that it has not yet reached its fullest development; in every country the annual output is still increasing.

In the Transvaal, the ratio of cyanide gold has remained fairly constant for several years at about one-fourth. In no other country has anything like this proportion been reached, so that with cheap cyanide and the spread of improved methods of handling, it would not be surprising if the output from the process were to be doubled within the next few years.


India.


New Zealand.


United States & Mexico.

### *Cyanide Production.*

The demand for cyanide of potassium for gold extraction has given rise to what is practically a new chemical industry. Prior to 1888, the only commercial outlets for cyanide were for the preparation of electro-plating solutions and for photographic and pharmaceutical purposes. The total consumption for these purposes did not exceed 50 to 70 tons a year, all of which was made by the smelting process from yellow prussiate of potash.

The pioneers of the gold-extracting process had therefore to provide for largely increased supplies of cyanide, and accordingly the Cassel Company erected works in Glasgow for its production from yellow prussiate. This new demand at once reacted on the prussiate industry, which had been languishing for some years, and the price of prussiate of potash rose within a short time from 6½*d.* to 10*d.* per lb.

Not only was the manufacture of prussiate from animal matters greatly stimulated, but the recovery of the cyanogen of crude coal gas assumed a new importance, and largely increased quantities of prussiate from this source came on the market.

The careful study, during the past 10 years, of the crude gases not only from gas retorts, but also from blast furnaces and coke ovens, has made us realise the very large stores of cyanogen which exist in these gases, most of which can be recovered at a moderate cost. I estimate that from these sources in Great Britain and the Continent of Europe at least 20,000 tons of yellow prussiate might be produced per annum if it were worth while to do so. To these sources of cyanogen must be added, as belonging to the same class, the gases produced by the destructive distillation of the nitrogenous residues from sugar refining. In one works in Germany, 300 tons per day of these residues are being distilled for the production of ammonia and cyanogen. 1,000 tons of prussiate per annum are said to be produced at this work.

The world's annual production of ferrocyanides and cyanide, calculated as yellow prussiate of potash, is estimated as 8,700 tons. The consumption of cyanide is estimated at 2,600 tons, equivalent to, say, 3,300 tons of yellow prussiate; this leaves a balance of prussiate production of, say, 5,400 tons, which has to be absorbed by the colour and dyestuff trades.

The production of 98–100 per cent. cyanides of potassium from the waste cyanogen of crude gases is necessarily a somewhat roundabout operation. As a rule the hydrocyanic acid in the gases is absorbed by a mixture of ferrous hydrate and an alkali, when a solution containing ferrocyanide of the alkali is obtained, which, on evaporation, gives a crude prussiate cake. The prussiate cake is purified by solution and recrystallisation till a perfectly pure crystallised ferrocyanide is obtained. The ferrocyanide crystals are carefully dried, and the anhydrous salt is then smelted either with metallic sodium or with alkali and charcoal. According to the well-known reaction, iron or carbide of iron separates, and a fused salt containing cyanide, cyanate, and carbonate of the alkali is obtained.

The necessity for the preparation of a white salt free from carbide of iron and alkaline sulphides complicates the process, and increases the consumption of raw materials as well as the working cost.

While the waste cyanogen of crude gases will always remain the cheapest source of yellow prussiate of potash and Prussian blue, it is doubtful if the more complicated process of producing high-grade cyanide for gold extraction from this source will be able to hold its own against the more direct synthetic processes.

In 1892, works were erected in Scotland and in Germany for the direct production of cyanide without the use of prussiates. As the output of these works increased from year to year the diminishing consumption of prussiates made itself felt in increasing stocks and lower prices, and during the year 1897 the prices of prussiates of potash and soda fell to the lowest recorded points, 4½*d.* and 3¼*d.* per lb. respectively.

The new demand for cyanide not only stimulated research into the recovery of cyanogen from waste gases, but also aroused the interest of chemical inventors. The usual result followed; the list of chemical patents applied for was swelled by an increasing number of new processes for the production of cyanides. During the past 10 years the annual numbers of cyanide patents applied for have been:—1888, 2; 1889, 4; 1890, 4; 1891, 5; 1892, 6; 1893, 12; 1894, 26; 1895, 30; 1896, 14; 1897, 5. This enthusiasm for cyanide processes reached its height in 1895, and appears to have now almost spent itself.

Among these 108 applications there is considerable variety in the sources from which it is proposed to draw the nitrogen of the cyanide; but on the whole the atmosphere remains the chief favourite.

The fascination which the problem of the fixation of atmospheric nitrogen has always had for chemical inventors is well known, and many of us have felt the influence of its spell. It is therefore with peculiar interest that technical chemists await the results of Dr. Readman's electrical furnace process which, it is well known, is being established in Scotland on a very large scale under the most favourable auspices. After a careful examination of the process and its working Prof. Wm. Ramsay reported in 1895 that it was possible by it to produce cyanide commercially at a cost of 5*d.* per lb. In order to produce cyanide at this price from yellow prussiate the price of refined prussiate of potash must not exceed 1¾*d.* per lb., so that obviously the manufacture of cyanide from prussiate will have to cease when high-grade cyanide is produced commercially at 5*d.* per lb.

Another of the indirect results of the cyanide process which ought to be appreciated by an association of chemists is that through its advent chemistry is now taking a more important place in the metallurgy of gold. The whole process is one which must be watched and regulated by chemical methods and tests. Quite a little army of chemists are now engaged all over the world at the gold mines, and it is gratifying to know that the enterprise of the Cassel Company in maintaining a laboratory for the training of chemists for this special work has given Glasgow men an important standing and representation in that army.

## Discussion.

Mr. J. S. MacArthur asked to be allowed to correct a little slip for which possibly he was himself responsible. Mr. Beilby had stated that the patentees had worked tailings in the Transvaal at 20*s.* or 25*s.* a ton. That was hardly the case. The patentees had guaranteed that the cost would not exceed 20*s.* or 25*s.*, but the cost actually came to be 10*s.*, so that they might take 10*s.* as the starting point of cost and not 20*s.* The Mercur cost was very low, and Mr. Beilby had said he did not think that included crushing. It did not include crushing. That ore was worked without being crushed, or with very little crushing; it was not ground in the same sense that gold ores generally were. In the case of a mine with which he was familiar, the management wished to try on how coarse crushing they could work, with a view to introduce direct cyaniding to the ore. They mined 200 or 300 tons, and without even putting it through a stone breaker, they applied cyanide direct, and got an extraction of 66 per cent. He named that incident only to show that one cannot expect all ores to work in the same way. Mr. Ellis had called their attention very pointedly to the necessity for fine grinding in many cases; and the grinding or state of division of each particular ore required the intelligent judgment of the man in charge. The action of soluble sulphides had been referred to, and he would like to hear an explanation of this action in retarding the solution of the gold. For himself he could not give a satisfactory explanation. When this process was first of all introduced, it was received with very cold courtesy. One great objection urged against the use of cyanide solutions was the terrible danger involved. It was said they were going to kill the whole of the population, and, what was worse, they were going to kill all the fish in the rivers. He was happy to say that the death roll in this enormous industry had, he thought, been lower than in any similar industry. So far as he was aware, in the whole gold industry, from the making of cyanide to the smelting of gold slimes, he was only aware of three human lives having been lost. A good many cows had tasted it, and had not survived, and probably some fish had been killed, but the exact number had not been reported. They now worked with cyanide as familiarly as any other ordinary commercial material. There had been one or two narrow escapes. One cyanide chemist had come in on a warm day, and seeing a glass of something looking very tempting, drunk it off. He had hardly taken it when he realised his mistake and, with astonishing presence of mind, stuck his fingers down his throat, and vomited the noxious dose. He was ill for a day or two, but he was now alive and well. He thought that the cyanide process had been the means of introducing accuracy and exact methods into gold mining and gold milling. Up till the advent of chemistry in the art, there was no proper account kept of the value of the ore or the value of the tailings. The management knew what they got out, but did not know how much they left in. Before the days of cyanide there was not one mine in ten which had an assayer to harass the manager, but he did it now. The assayer was a perfect thorn in the careless manager's side. The accuracy demanded in the chemical processes involved accuracy all through, because it was obvious that if a correct return was to be kept of the gold extracted, an exact return must also be kept of the tonnage treated. In the old days the tonnage was a matter of estimation, and the estimation was a matter of guess. They took out all the gold they could get and were thankful for it, and left the tonnage and tailings to look after themselves. The solutions that were used in the MacArthur-Forrest process had always been dilute, and now they were exceedingly dilute. Yet the consumption of cyanide was quite appreciable. In these early days they knew the value of dilution, and his partners in this invention, being medical men, were very much afraid of being found guilty of homeopathic heresy. They used solutions very weak indeed, but one well-known chemist had gone one better, and had extracted 25 per cent. from very refractory ore by the use of water only as a solvent! The cost now, at 2*s.* 6*d.* a ton, was very low, and made the possibility of improvement very remote. That it was capable of improvement they all admitted, but it would take some trouble to improve and economise much on 2*s.* 6*d.*, which was now the measure of future economy in ordinary cyanide practice. He hardly thought that improvements of very great consequence could be made to economise working on the present lines. It was a great pleasure to the Drs. Forrest and himself to know that this process with which they were so closely identified had been the means of creating a new industry. It had not come into competition with anything. Cyanide had been made to act on waste material, and had recovered gold which hitherto would practically not have existed. It had brought gold into the world in effect. That a process was wanted was better known by commercial men than by chemists. The cyanide process was a very simple thing in the laboratory, and it was very simple now that they all understood it, but it cost the Drs. Forrest and himself a good deal of work to get at it. Many a weary day they had had, and there was many a weary evening when they thought they had better throw the whole thing up. All through they were very much stimulated and compelled to work by the far-seeing wisdom of a friend, a commercial man, who said that the world wanted gold, and must have it. He insisted that if they went on they would find a process, if such were to be found, and said they were amassing knowledge all the time, and that if they did not use it, some other person would, or, worse than that, it would be lost. He thought that a very large share of the credit or the success of the cyanide process was due to the far-sighted policy and enterprise of that commercial man.

Mr. R. R. Tatlock said that the paper read by Mr. Ellis dealt with the methods of investigating the character, both physically and chemically, of gold ores, with the object of determining and designing the best plant for their treatment. It was not sufficient to assay the ores, or even to make experiments in the laboratory with the object of ascertaining the minimum amount of cyanide of potassium that would extract the gold, but it was necessary to ascertain whether it was not possible to treat the ore in some particular way which would be more economical than the more direct application of the cyanide to the crushed ore or the tailings from the ore. He recognised that the paper was of great value to technical chemists proper rather than to assayers, because he had always felt that the man who had the qualifications to devise plant and to apply processes, old or new, for any special purpose, in the gold industry particularly, should have, combined with that qualification, a knowledge of such work as Mr. Ellis had so aptly described in his paper. The two ought to go together. In regard to the Chairman's paper, it was a very happy thought of his to propose to read a paper on "Ten Years of Gold Extraction." He thought the ten years idea was a capital one, and he hoped it would very soon be followed by ten years of other things. Some one might give them a paper on ten years of chemistry applied to agriculture, ten years of the explosives industry, and ten years of minor processes which came very little to the front, but which collectively were of as much importance as some of the gigantic industies. With reference to the general question of new gold extraction processes, when a man contemplated devoting his time, skill, and money to devising a new method or to developing a new process of extracting gold, or some modification of a method which was of importance in the process of extracting gold, and when he successfuly evolved that process and it was likely to become a very important advantage to the industry, it was well that he should pause and consider whether the game was worth the candle. It was not good enough to secure patents in countries where they could only be enforced by harassing and expensive litigation, and in countries, he might say semi-barbarous countries, which were not amenable to the laws and customs of more advanced nations of the world. Unless a man wanted to emulate the self-sacrifice of the missionary, or the pioneer or the forlorn hope, in the industry, he would be far better, or unless he was ambitious to share the too frequent fate of these devoted gentlemen, to keep his money in his pocket and devote his brains to something else. In the light of past history he should say that a man would be very sanguine to hope that in some countries he would be successful in having his patent rights secured to him, when it was to impose an additional burden upon a mining industry already harassed to resistance by concessions and by taxation. All these things the inventor, as well as those who backed him up, had to face, and it would be well for inventors first to pause and consider, even if they had a process that would revolutionise the gold industry and build up a new Johannesburg, whether it was worth while to go on with it. If they looked at it in the light of past history, what did they see? They saw men of skill and experience, able chemists, men of practical knowledge, devoted and courageous men, going to countries far away, to darkest Africa, he might say (as far as up-to-date knowledge of gold extraction was concerned), and there forcing down the throats of an unwilling mining community, a process which they were very much in need of, and which they knew they were in need of, and yet would not have, forcing down their unwilling throats, so to speak, the food that was to become their life-blood afterwards, and what was the result? The result was that they were told, not only by the beneficiaries of that process, but by the legislators of the country, that the thing was all known before, and had all been done before, and yet they all knew that not a single ounce of gold had been extracted before that process was introduced into that country, and urged upon these unwilling miners. It was most discouraging to investigators in the gold industry, not only as regards foreign countries, but even here, that such trouble and expense were entailed in having patent rights secured; and it was much more difficult in a country like the Transvaal where patent laws did not seem to be known or understood even by the framers of them, if there were such.

Mr. H. A. Jones said that it had often been a question in his mind whether the cyanide process would have become generally recognised and adopted if it had not, by the command of capital, the application of time, patience, and technical skill, been specialised and forced into prominence. Without these advantages it seemed to him possible and even probable that it might have remained, like so many other patents, an item of curious scientific lore, lost among the crowd so to speak, and regarded as of little practical utility. The reasons for these impressions were principally twofold, namely—First: the opposition, hostility, incredulity, and indifference which the process had to encounter; and second: the specialised technical knowledge required for

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its successful working. With regard to the first of these heads, the difficulties that had to be encountered by the Cassel Company in introducing the process were enormous. The idea of treating unroasted ores with a simple cyanide solution was laughed to scorn, and was generally looked upon as another of the numerous "wildcat" tribe. In South Africa a challenge was publicly published by a well known metallurgical chemist, boldly denying that the process could do what was claimed; and Mr. Tatlock was well within the mark when he said that the process had to be forced down the throats of the mining community. Nor were these difficulties confined to one locality. The monotony of such experiences was wearisome and disheartening. The first Transvaal patent was dated 28th November 1888. In the same month the Cassel Company sent their first expedition to the Transvaal. The agreement with the original Syndicate for the promotion of the African Company was entered into in November 1889. The second expedition to further publicly demonstrate the process was dispatched by the Cassel Company in November 1889, and its operations extended over a large part of 1890, and the public demonstration and continuous working, though successful, met with scant encouragment and much incredulity. The expenditure for the two expeditions, plant, staff, public demonstration, &c. was very heavy. The African company was floated in March 1891, but, notwithstanding that the ores of the Transvaal were said to be specially suitable for the cyanide process, the total output in the Rand for that year, 1891, was only 17,000 ounces. In 1892 the upward tendency set in; so one might say that even in South Africa, with the most favourable conditions, namely, tailings ready to hand and easy to treat, it had taken four years of uphill work and heavy expenditure to produce the necessary impression. In like manner the first expedition to New Zealand was despatched by the Cassel Company in November 1888. The expenditure for plant, staff, &c. was over 5,000*l.*, and the expenses accumulated before any impression was made or return received, and though the operations were imminently successful, it was not until 1893 that the process began to be seriously taken up, and 1894 was reached, or six years from the start, before any substantial return began to be reaped. There has been the same experience in Australia. Indeed it was for a long time claimed by the opponents of the process that it had only been successful because of the specially favourable conditions in South Africa, and that it would not succeed elsewhere for the direct treatment of ores instead of tailings. This theory had now been entirely exploded, but it had taken time, work, and further expenditure to disprove it. Only last year, 1897, might the cyanide development in Australia be said to have taken a really large and substantial shape, and now the rate of progress had become rapid, but nine years had passed since a start was made by the Cassel Company's staff at Ravenswood in Queensland. Similar remarks applied almost identically to the United States, though the process was first demonstrated there in 1890. One might well ask, in looking at these facts, where the process would have been without persistent and patient pushing, demonstration, and consequent heavy expenditure of capital.

The second reason for his doubt as to whether the process might not have lain dormant unless it has been specialised and exploited was, that "specialised technical knowledge was required for its successful working," and such knowledge was not to be had at the time the patents were secured, but had to be created and trained. Practically nothing was known about the action of cyanide on ores. It was an unexplored country, knowledge of which could only be acquired by original investigation and research. Such knowledge as then existed of the action of cyanide solutions on metallic gold did not even naturally lead to the conclusion that they would be successful in the treatment of ores, but rather the contrary. What chances were there then that, in the absence of qualified experts to show the way, miners with their rough and ready methods, would have so much faith, intuition, and specialised knowledge as to successfully adopt cyanide treatment? The idea seemed most unlikely. As a matter of fact, in the earlier days of the cyanide process there were numerous failures where attempts were made to put it into practice without the direction of trained experts; and it may be stated as a general truth, that success was only fully achieved when experts originally trained in the Cassel Company's laboratory were in charge; of course this had been and would be modified as the cyanide operations extended and experience increased, but the original fact remained all the same. When the Cassel Company took out the cyanide patents they had also to train a staff of cyanide experts ready to be sent all over the world, and one of the greatest services, among many others which they rendered to the cyanide industry—in fact, a service of incalculable benefit—was the formation of the central training school for cyanide experts at their works in Glasgow.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Mechanical Filters, Impts. in.* I. S. McDougall, Didsbury, Lancashire. Eng. Pat. 29,224, Dec. 19, 1896.

In mechanical filters in which a granular, porous, fibrous, or felted material is employed, the filtering material is placed between two sieve plates, the upper one of which has a screw adjustment for exerting pressure on the medium, so that it may be held in compression and offer equal resistance throughout its mass to the passage of the liquid. The apparatus is also constructed to facilitate the removal of the spent filtering medium and the introduction of fresh material.—R. A.

*Filtering Apparatus, Impts. in.* W. R. Renshaw, Phœnix Works, Stoke on-Trent. Eng. Pat. 2326, Jan. 28, 1897.

Filtering apparatus for water, &c., is formed with passages for washing water or liquid between layers or portions of the filtering material, so that the whole, or any particular layer or layers can be cleansed, as desired.
—R. A.

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* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Purifying Fluids, Impts. in Apparatus for.* R. G. Brooke, Macclesfield, Cheshire. Eng. Pat. 2931, Feb. 3, 1897.

The apparatus comprises a casing consisting of a stationary portion and a removable portion, and having an inlet and outlet for the fluid to be purified, a discharge pipe or outlet for the water or other matter separated from the fluid, one or more slotted or perforated cylinders arranged within the casing so as to form one or more annular spaces, and means for supporting the cylinders in their proper positions in or on the stationary portion of the casing after the removal of the other portion. The slots or perforations in the cylinders are so arranged, relatively to the fluid inlet and outlets, &c., that the fluid to be purified will be caused to pass around or along and through the walls of the cylinders, and will have its direction of flow changed in passing through the walls of the cylinders. When used for the filtration of water, &c., means are also provided for enabling the apparatus to be cleansed by reversing the direction of flow of the liquid.—R. A.

*Concentrating, Cooling, and other Purposes; Improved Evaporating Process and Apparatus applicable for.* J. B. Alliott, Bloomsgrove Works, Nottingham. Eng. Pat. 3482, Feb. 9, 1897.

The evaporation of the liquid to be treated is carried out in a vessel or vessels wherein a more or less perfect vacuum is established, and the condensation of the vapour is effected in a condenser or condensers in connection with such vessel or vessels, by means of water, &c., which is brought into direct contact with the vapour, the air being previously extracted from the condensing liquid, by exposing it to the action of a vacuum in a separate vessel. A jet condensing arrangement is employed, and the air-pump used for producing the vacuum in the vessels is made to discharge into a second vacuum vessel or air-pump, the arrangement being such that the primary air-pump will discharge against a much lower pressure than that of the external atmosphere. —R. A.

*Cooling and Evaporating, Improved Methods of and Apparatus for.* J. B. Alliott, Bloomsgrove Works, Nottingham. Eng. Pat. 3483, Feb. 9, 1897.

In the cooling of liquid by evaporation, the liquid is treated successively in two or more vacuum vessels, working in conjunction with vacuum pumps and condensers, in such a way that the cooling of the liquid is effected in stages, liquid previously deprived of air being employed for condensing the vapour given off in the last vacuum vessel. Strong sulphuric acid may also be employed, either alone or in conjunction with condensing liquid, for effecting the condensation of the vapour. Another method consists in effecting the cooling of a solution by making the solution originally of a greater strength than is required for use, cooling such solution in stages by evaporation in vacuum vessels, and afterwards further reducing the temperature of the solution by the addition to it of such a quantity of a colder liquid as is needed to bring it to the strength and temperature actually required.—R. A.

*Filters and Arrangements and Apparatus or Plant connected therewith, Impts. in.* F. Candy, Surrey, and The International Water and Sewage Purification Co., Ltd., 7, Victoria Street, Westminster. Eng. Pat. 3966, Feb. 13, 1897.

In filters acting upwards and provided with arrangements for cleansing the filtering material by downward passage of the filtered liquid, the filter-bed is placed within a receptacle of smaller sectional area at the lower part than at the upper part, for the purpose of causing the cleansing liquid to act more forcibly in the part of the bed where the separated matter accumulates, than at other parts. Provision is also made for the escape of air or gas from the liquid prior to its being filtered. The cleansing of the filters is effected automatically by the descent through them of liquid which has been filtered, the automatic action of the apparatus being determined by the obstruction to the upward passage of the liquid through the filter, caused by the collection in the filter of separated matter.—R. A.

*Valves more especially adapted for Use in Self-discharging Saturators used in the Manufacture of Sulphate of Ammonia from Gas-Liquor or Ammoniacal Liquor, Impts. in.* H. Howell, Donnington, Shropshire. Eng. Pat. 4095, Feb. 15, 1897.

The valve is a casing having one opening connected to the saturator and another opening constituting the outlet, in which casing is mounted a piston capable of movement by a screw or otherwise, to put the openings into communication, or to close them. The piston is provided with sharp edges, to cut away or scrape off deposited matter. The screw is worked by a hand-wheel operating in a nut fixed to the casing by rods.—E. S.

*Condensing and Cooling Vapours and Liquids, Impts. in or connected with Apparatus for.* T. Fairley, East Parade, Leeds. Eng. Pat. 4536, Feb. 19, 1897.

The apparatus consists of a cone or conical chamber, through which a supply of the cooling agent is passed, in combination with an inlet over the cone for the passage of the vapour, &c. to be condensed or cooled, and a jacket for confining the vapour, &c. to the cold surface of the cone. —R. A.

*Acid-Proof Jars, Bottles, Reservoirs, Cylinders, and the like; Impts. in the Construction of, and in their Manufacture.* W. P. Thompson. From J. Marx, Berleberg, Germany. Eng. Pat. 22,640, Oct. 2, 1897.

The acid-proof vessel, utensil, or article, is enclosed within a protecting envelope or cover of a material capable of resisting fracture, an intervening layer of a homogeneous material, such as cement, &c., being inserted between it and the said envelope, and subjected to pressure until solidification takes place. When the exterior of the article is acid-proof, it may be similarly lined with an inner covering or core of strengthing material, with an intervening layer of homogeneous material. The invention is described in the specification as applied for constructing salt-cake vessels, autoclaves, &c., and vessels or objects for treating phosphoric acid.—R. A.

## II.—FUEL, GAS, AND LIGHT.

*Calcium Carbide Works at Geneva.* Eng. and Mining J. 1897, **64**, [22], 641.

It has been decided by the authorities who control the electric light plant at Vernier, to utilise it for the manufacture of calcium carbide. The dynamos are driven by turbines operated by abundant water power, and, in order to reduce the labour charges, the pulverising of the coke, mixing the latter with lime, &c., are performed mechanically. Most of the operations are automatic, and only one man's labour is required from the time the crude material is received, until the product comes from the furnace. Special attention is paid to the use of good raw materials; the coke used contains only 5 per cent. of ash, and the lime is very pure, containing 99—99·5 per cent. of calcium oxide. Each furnace will take 500 electrical horse-power (6,000 ampères at 57 volts), and is composed of a large cylindrical crucible, 59 in. in diameter and 32 in. high. The electrode is made of compressed carbon, and is in six pieces, each 60 in. long and 5·2 by 9·2 in. in section. The total weight of the six carbons is over 858 lb. The crucible is fed from above by means of iron chutes.—A. S.

*Acetylene, Purification of.* E. Odernheimer. Chem. Zeit. **22**, [4], 21.

A substance for this purpose, which has recently been put on the market, was found to be chiefly composed of sawdust and oxides of iron, and is identical with the compound used in Laming's process for purifying coal-gas. By its use, the smoking of the acetylene flame is prevented, but poisonous phosphoretted hydrogen compounds are not removed. These latter, however, may be eliminated by first passing the acetylene over moist lumps of chloride of lime, and then over lime, to absorb any liberated chlorine compounds.—W. P. S.

*Cyanides in Gas Manufacture.* H. Poole. J. Gas Lighting, **70**, [1897], 1231—1236.

An enormous quantity of cyanides in gas is now wasted, in spite of modern attempts at recovery. How they are formed has not yet been decisively settled. Ammonia passed over incandescent carbon, or with carbonic oxide over heated surfaces, is known to produce hydrocyanic acid and ammonium cyanide; if the temperature be high, or an excess of hydrogen be present, only the free acid is found. The addition of sulphur to these products, or the mixture of carbon bisulphide with the ammonia, yields the more stable ammonium sulphocyanide. The yields increase with the temperature and retardation of the flow of gas, and in the presence of fixed alkalis; but the reactions never occur singly, and sulphur, if present, always enters into the combination.

In the gas retort, these fundamental reactions are modified by the presence of a quantity of free hydrogen, and it is a question whether the small percentage of cyanide in crude gas is due to the presence of hydrogen *per se*, or to the high temperature. Bergmann, however, showed that the percentage of the nitrogen in a mixture of carbonic oxide and ammonia converted, on heating, into cyanogen, was 4 at 800°, 24 at 1,000°, and 60 when illuminating gas was also present.

Part of the sulphur of the pyrites in coal is found in the gas as sulphocyanide, but the iron of the pyrites does not form ferrocyanide, as the latter has not been traced with certainty in the gas leaving the retort. Ferrocyanide and sulphocyanide occur in ammoniacal liquor, but cyanide does not—perhaps because stronger acids expel the hydrocyanic acid. The gas suffers little change so far as cyanogen compounds are concerned until it reaches the purifiers. The ferric oxide in these is usually supposed to form ferrous cyanide with the cyanogen. Ferrous cyanide is yellowish-orange, turning to bluish grey by slight oxidation, and the compound in the purifiers is of the latter tint. It is not easy to write a formula for the formation of ferrous cyanide from ferric oxide, but the following may explain the formation and subsequent partial oxidation:—

$$Fe_2O_3 + 4HCN = 2Fe(CN)_2 + 2H_2O + O.$$
$$9Fe(CN)_2 + 3O = Fe_2O_3 + Fe_7(CN)_{18}.$$

The Prussian blue thus formed tints the remainder of the ferrous cyanide. If ammonium cyanide were present in excess, the ammonium ferrocyanide formed could be traced in the lixiviate on extracting the oxide with water, as no other soluble ferrocyanides could be there. After thorough extraction with water, treatment of the oxide with weak soda would give a liquor in which the cyanide existing as Prussian blue in the oxide could be detected. Cyanogen can also be fixed by the ferrous sulphide formed in the purifiers; thus: $FeS + 2HCN = Fe(CN)_2 + H_2S$.

This equation is supported by the fact that sulphur reappears at the outlet of a purifier containing ferrous sulphide from which the "extra" sulphuretted hydrogen has been removed by inert gas, when gas containing cyanogen is passed in.

Sulphocyanogen, formed from ammonia and carbon bisulphide, occurs in the crude gas, but the amount therefrom is small compared with that formed in the purifiers, where cyanogen in simple or compound form meets free or loosely combined sulphur. Nevertheless, in order to reduce the amount of sulphocyanides in the purifiers, ammonia should as far as possible be prevented from entering the latter. Sulphocyanides are more stable than cyanides or ferrocyanides, and are formed wherever possible.

The facts may be summarised as follows:—(1.) Cyanogen exists in crude gas as hydrocyanic acid, ammonium cyanide or sulphocyanide, but not as ferrocyanide. (2.) Prussian blue is formed in the purifier under ordinary circumstances only in small quantities; on the admission of air, it is formed from the oxidation of the ferrous cyanide. (3.) Sulphuretted hydrogen may be taken up by gas in passing through an old purifier. (4.) Sulphocyanides may form in the purifier when no sulphocyanogen is in the gas, and ammonia favours their formation.—I. A. B.

## PATENTS.

*Carbide, A Process of Manufacture of a, which, by its Decomposition by Water, produces Acetylene Gas.* A. Kiesewalter, Limburg-on-Lahn, Germany. Eng. Pat. 27,741, May 6, 1896. (Under Internat. Convent.)

Baryta, magnesia, or potash is added to the lime to render it more "fusible." and a metal which is volatile, such as zinc, is also added to assist in removing and carrying away the oxygen from the lime, so that the reaction with the carbon to form the carbide, is rendered more easy of accomplishment. Two suitable mixtures are mentioned.

The process is carried out as usual, the inner walls of the furnace being lined with carbon plates, and the carbide is produced at a temperature of from 1,300° to 1,500° C. It is not pure calcium carbide, but a mixed carbide, which, however, yields acetylene gas with water. The first mixture yields a carbide of calcium and potassium.

The author states that the real formula of calcium carbide is $CaOC_2$, which with water produces carbon dioxide in addition to acetylene.—A. W.

*Gas, Automatically Indicating, Recording, and Regulating, in a controlled Manner, the Percentage Volume of a Particular Kind of, in a Gaseous Mixture [Carbonic Acid in Furnace Gases]: Process and Apparatus for.* M. Arndt, Aachen, Germany. Eng. Pat. 27,757, Dec. 5, 1896.

The automatic indication and record of the percentage volume of a gas in a gaseous mixture is the object of this invention. The mixture is conducted through a suitable absorbing apparatus, the working of which, as also that of the recording apparatus, is controlled so that the gas vessel is first charged with the gaseous mixture and then shut off, the separation of the gas, the quantity of which it is desired to ascertain, being effected by means of a chemical absorbing medium. After the completion of the absorption, the attenuation of the gaseous mixture corresponding to the percentage volume of the separated gas is indicated and recorded, and an electric current-distributing mechanism is actuated, so that upon alteration in the percentage of the particular gas taking place, working parts are correspondingly influenced to regulate the supply of air to the furnace, &c. Finally, the inlet and outlet apertures of the gas vessel are again opened for the purpose of recharging it with fresh gaseous mixture and repeating the operations. Several forms of apparatus for carrying out the process are fully described in the specification.—B. A.

*Hydrogen Gas, and in Apparatus therefor; Impts. in the Production of.* R. G. Leventhorpe, Hyde Park, London. Eng. Pat. 28,721, Dec. 15, 1896.

Relates to the known method of producing hydrogen by passing steam through heated retorts charged with iron borings. The retorts are arranged horizontally in two or more tiers within a chamber adjacent to and heated by a producer furnace. The furnace narrows towards the bottom, and is provided with a perforated arch, through which the gases pass to a combustion chamber above, whence they pass through a side opening to the retort chamber. The hot gases afterwards heat the secondary air-supply flues, and next pass to the boiler for producing steam, to be passed successively through the tiers of retorts. The gases evolved from the retorts, consisting of hydrogen and undecomposed steam, are passed through a condensing coil, and thence to horizontal storing vessels or gasholders. An automatic gas pressure regulating gasholder is described for the hydrogen storing apparatus, and is used in combination with the water outlet from the main gasholders.—R. S.

*Artificial Fuel, Impts. in the Manufacture of.* J. Steiger, Blackwall, London. Eng. Pat. 19,741, Sept. 7, 1896.

The fuel is formed by mixing "petrifite," made as described in Eng. Pat. 17,142, 1893 (this Journal, 1894, 255), with oil waste and water. To the "petrifite," before mixing with the oil waste, &c., may be added filling materials, such as coal refuse, coal dust, lignite coal, wood waste, husks, bran, chopped straw, resin, town refuse, and other inflammable material.—R. S.

*Petroleum and other Matter, Admixing and Preparing in Compound Form, with a View to its Final Use as a Combustible; An Improved Mode of.* J. W. Leadbeater, Leeds. Eng. Pat. 3891, Feb. 13, 1897.

The following ingredients are mixed together with as little heat as possible, and in approximately the following proportions, the proportions varying according to the consistency required in the product:—Petroleum, crude or otherwise (including astatki), 128 parts; animal or vegetable stearin, 1 part; resin, 12 parts; unslaked lime, 8 parts; and sawdust, 8 parts.—R. S.

*Briquettes or Blocks of Fuel, Impts. in the Manufacture of, from Waste Products or otherwise, enriched by Petroleum Compounds.* J. W. Leadbeater, Leeds. Eng. Pat. 3892, Feb. 13, 1897.

The briquettes are formed by mixing about 5 or 10 per cent. of the petroleum compound (prepared as in the preceding case) with small fuel, which may consist of waste products, such as town's refuse, dried sewage sludge, and the like, or other carbonaceous matter, unsuitable as fuel, such as coal dust, peat, &c. The petroleum compound may be added in a granulated or divided condition, or in a fluid state. To bind the materials together, an agglutinant, such as pitch, starch, and the like, is added.—R. S.

*Electric Furnaces [Carbides, Hollow Electrodes, &c.], Impts. in or relating to.* W. W. Hughes, London. Eng. Pat. 29,576, Dec. 23, 1896.

"It is the object of the present invention to ensure that the whole of the materials delivered into the furnace shall come under the direct influence of the arc." "The hearth of the furnace [for carbides to be prepared from peat charcoal and lime, and also for general purposes] is used as one electrode, and an annular arc or its equivalent is set up between the hearth and the other electrode or electrodes, which are so disposed as to constitute or surround a supply tube, through which material for treatment is delivered on to the hearth," so that "all the material delivered to the furnace must pass through the field of the arc."

Claims:—1. . . . . "the combination with a solid hearth, forming one electrode of a second [hollow] electrode, through or about which the material to be treated passes." 2. . . . . "the combination with an electrode, through or about which the material to be treated passes, of a solid hearth, forming the other electrode, and adjustable in relation to the first electrode." 3. "The combination and arrangement of parts constituting the electric furnace . . . as described and illustrated."—J. C. R.

*Electric Furnaces [Utilising Gases, &c.], Impts. in and relating to.* W. P. Thompson, London. From La Societá Italiana dei Forni Elettrici, Rome. Eng. Pat. 14,247, June 11, 1897.

"Relates to an improved construction of electric furnace, specially adapted for the carrying out of the process set forth in Eng. Pat. 13,297 of 1896 (this Journal, 1897, 231), and applicable also to other purposes."

Claims:—1. "In an electric furnace the arrangement of a heating chamber around the hearth of the furnace, into which the heated gases produced at the hearth pass and circulate before leaving the furnace, thus assisting in maintaining the temperature of the hearth." 2. "In an electric furnace, the arrangement for the utilisation of the gaseous products to heat the materials to be treated in the furnace, consisting in passing the furnace gases through a flue in which they return a proportion of their heat to the furnace, then passing them through suitable purifying and collecting apparatus, and finally returning them to the furnace to be ignited beneath the inclined tube through which the materials are delivered to the hearth of the furnace." 3. "The improved electric furnace, constructed substantially as described, with reference to the accompanying drawings."—J. C. R.

*Furnaces for Evaporating Pans and other Purposes, Impts. in.* R. H. Yeomans, Middlewich, Cheshire. Eng. Pat. 29,757, Dec. 28, 1896.

The furnace is formed with a downwardly inclined hearth, leading from the front nearly to the back, at which place the cinders fall to the floor. Above is an arch, perforated or not, while beneath, or at the sides, is an air-passage leading from the front, and passing to openings in the back, leading into the furnace above the hearth.—R. S.

*Electrical Furnaces [for Carbides, &c.], Impts. in and connected with.* F. J. Patten, New York. Eng. Pat. 17,081, July 20, 1897.

The inventor illustrates several forms of electric furnace, for the production of carbides and such like; but his invention consists in extending the function of the arc in such furnaces by making it move about.

Claim 1. "In an electrical furnace, a furnace wall, a pair of carbon electrodes within said wall, one of the electrodes being circular in cross-section, means for rotating the arc between and relatively to the electrodes, and passages to convey material through the annular arcing space." . . . . Other nine claims include a ring of magnetisable material surrounding the arcing space, means for creating a rotating magnetic field in the ring and its winding from the arc; adjusting the upper carbon, &c. A carbon pencil of relatively small cross-section primarily connected to electrodes, in combination with a transverse rotating magnetic field surrounding the pencil, &c. "Electrically heating a homogeneous mass of material of low and approximately uniform conductivity, which consists in passing an electric current through the mass, establishing around it a magnetic field having its lines of force transverse to the line of current flow, and causing the magnetic field to rotate about the line of current flow," &c.—J. C. R.

*Bunsen Burners for Burning Petroleum or other Hydrocarbons, Impts. in or relating to.* A. J. Boult, Hatton Garden. From G. Barthel, Dresden-Striesen, Germany. Eng. Pat. 26,324, Nov. 11, 1897.

The improvements relate especially to soldering lamps which burn petroleum, and the apparatus—air-pump, reservoir, vaporiser, &c.—" is substantially arranged in the well-known manner." The novelty described and claimed is the arrangement on the inner surface of the vaporising ring in the burner tube, of long projections or teeth, at short distances apart, "projecting so far towards the centre that a free central opening is left, which is very small in comparison with the inner diameter of the annular vaporiser."
—H. B.

*Incandescent Lighting, Impts. in Means for.* C. Colas, Rheims. Eng. Pat. 29,736, Dec. 24, 1896.

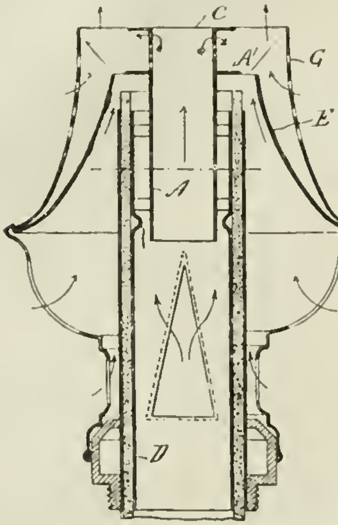
"The core or base of the solid mantle or device . . . . which is a suitable fabric of the ordinary character," is repeatedly dipped into a cream (prepared with water from oxide of thorium, "kaolin and plastic clay, alkaline silicates, infusible and fusible salts, cements, plaster associated with the salts or metallic oxides, and even with foreign bodies"), dried at 25° to 26° C., and then baked in an oven. The "solid mantle" thus formed is to be used in the same way as an ordinary mantle.—H. B.

*Incandescence Lamps for use with Petroleum and the like, Impts. in.* A. Graetz, Berlin. Eng. Pat. 2295, Jan. 28, 1897.

In a petroleum burner, having a circular wick and a central air supply, the top of the wick tube is surrounded by two concentric conical rings, which form two annular flues, the rings being of equal height. Apertures at the base of the rings give rise to three air currents—one passing upwards between the wick tube and the first ring, one passing up between the two rings, and a third passing between the outer ring and the chimney. A cylindrical cap, closed at the top, extends upwards from the inner wick tube, and is provided near its upper end with a circular flame disc; the sides of the cap are perforated both above and below the level of the disc, and the diameter of the cap is smaller than that of the wick tube. The mantle, supported from above, slips over the outermost conical ring, and when the burner is lit, the various air currents give rise to a non-luminous flame. The burner gallery, carrying the chimney, mantle, and conical rings, is attached by means of hooks and corresponding slots to a ring provided with vertical guide-rods, and the whole may be raised or lowered by means of the usual device of curved levers. The whole combination of parts is claimed.—H. B.

*Incandescence Oil Burners, Impts. in or relating to.* W. P. Thompson, London. From The Continental Gas Glühlicht Gesellschaft "Meteor," vormals Kroll, Berger, and Co., Berlin. Eng. Pat. 1583, Feb. 20, 1897.

[See also this Journal, 1897, 1007.] Referring to the previous specification, the patentee states that the inner tube A may be dispensed with if it is replaced by a device



consisting of a support rising centrally from within the wick tube, and having at its upper end an ordinary disc baffle (corresponding to C), to the under side of which is fastened a short perforated tube of diameter approximately equal to the diameter of the wick tube. The lower end of the perforated tube is open, and an annular ring (corresponding to the flange A¹) projects from its lower edge over the wick. The new device and the burner embodying it are claimed.—H. B.

*Acetylene, An Improved Method of Storing, for Lighting and other Purposes.* G. Claude, Saint Mandé, and A. Hess, Paris. Eng. Pat. 29,750, Dec. 24, 1896. (Under Internat. Convention.)

Acetylene is dissolved under pressure in certain liquids in which it is soluble to a considerable extent. The solubility increases somewhat in the ratio of the pressure. The following are among the suitable solvents, which may be used either alone or in mixtures:—Fatty and aromatic hydrocarbons, and their chloro, bromo, iodo, and nitro derivatives; the lower fatty acids and oxy acids; aldehydes, mono-hydroxyl, and unsaturated alcohols of the fatty series; ethers, phenols, and aromatic amines; trimethylene, furfurane, thiophene, furfurol, &c., and their derivatives. The solution is stored in a receiver, from which the gas is evolved when required for use.—J. A. B.

*Acetylene and other Gases, An Improved Generator, Condenser, and Holder for.* H. R. Bean, Plaistow, Essex, and H. Ringwood, Poplar. Eng. Pat. 2428, Jan. 29, 1897.

A carbide receptacle and waste container are suspended from the crown of a bell gasholder, which floats in a double-walled tank. The gas generated, passes by a pipe to the bottom of the annular space between the walls, which acts as a condenser, and it is withdrawn for use from the top of that space. An annular enlargement of the top of the tank accommodates displaced water.—J. A. B.

*Acetylene Gas, Impts. in or relating to the Production of.* H. Sefton-Jones, London. From P. P. H. Macé, Paris. Eng. Pat. 14,905, June 19, 1897.

In the manufacture of carbide, 5 to 15 per cent. of binoxide of manganese is added to the usual charge of lime and carbon. A mixture of carbides of manganese and calcium is thus obtained, of which the cost price is about the same as that of calcium carbide. The manganese carbide is said to act on water with the production of methane and an equal quantity of nascent hydrogen, which acts upon the carbon which tends to be deposited at the moment of the combustion of acetylene, and forms with it a combustible hydrocarbon. The reaction is stated to be:—

$$CaC_2 + 3MnC + 8H_2O =$$
$$2C_2H_2 + CH_4 + CaO, H_2O + 3MnO, H_2O.$$

The object of the invention is to prevent carbonaceous deposits at the burners, arising from polymerisation of acetylene at the moment of combustion.—J. A. B.

*Acetylene Gas, Impts. in or relating to Apparatus for the Manufacture and Use of.* A. J. Boult, London. From F. Alexandre, Paris. Eng. Pat. 26,325, Nov. 11, 1897.

The upper compartment of a gasholder communicates with the lower compartment by a siphon tube through which water may pass. In the upper compartment is supported a generator containing carbide, to which water from an adjacent reservoir gains access through porous plates at different levels, so that the carbide is attacked from the bottom upwards. The gas generated, passes through sponge in the top of the water reservoir to the lower compartment, whence it forces water to the upper compartment, and thereby operates a ball float, which closes the gas exit from the generator. The pressure of gas in the latter forces back the water to the reservoir, and the action ceases until gas has been consumed.—J. A. B.

*Acetylene Gas Generator, Improved.* J. H. Schülke, Berlin. Eng. Pat. 27,767, Nov. 25, 1897.

Generating vessels, each consisting of a perforated carbide container within a vessel open at the top and attached to a superposed bell, are arranged in a tank of water. Telescoping pipes convey the generated gas through the bottom of the tank to a main pipe which leads to a condensing coil, which discharges the gas within a floating bell, from which it passes to a pressure regulator. The pressure within the bell determines the height to which water rises in the bells of the generating vessels, and the water inlets to the carbide receptacles are at different heights in each of these, so that they are brought into operation in succession. The pressure on the bell controls the operation of the apparatus.
—J. A. B.

*Acetylene Gas Generator, An Improved.* W. Tyree, Wellington, N.Z. Eng. Pat. 28,094, Nov. 29, 1897.

A bell floating in the annular space of a double-walled tank has attached to its crown a trunk, the bottom of which seals in the water in the tank, and is furnished with a slide or trap operated from the top of the bell. Carbide is dropped from the trunk as required. It may fall on a distributing cone in the tank or into a crate. A sludge cock permits removal of sludge, while fresh water is introduced through the trunk without escape of gas.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* M. Wagner, Nogent-on-the-Marne, France. Eng. Pat. 28,102, Nov. 29, 1897.

A tube closed by screw plugs comprises three superposed compartments, the lower of which contains a cage of carbide, to the bottom of which water is supplied from the middle compartment, until the gas pressure checks the supply. The generated gas passes by a pipe through the middle to the upper compartment, which is filled with purifying agents. Surplus gas passes from the bottom of the generating compartment to the space above the water in the middle compartment, and thence through a small orifice to the air.—J. A. B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*α-Acetylfurfuran in Wood Tars, Presence of.* L. Bouveault. Comptes Rend. 1897, **125**, [26], 1184—1186.

The raw material in this research was a light oil, boiling at from 150° to 200°, from an Alsace works where beech and a little oak only were distilled. After washing with a solution of caustic soda and dilute hydrochloric acid, the oil was distilled under a vacuum, a rectifying column being used. The fraction distilling between 60°—70° C., under 10 mm. pressure, was treated with excess of hydroxylamine, with which it partially combined, yielding a mixture of oximes boiling between 105° and 115° C. under 10 mm. On separating the oximes from the unattacked oil, and allowing them to rest for some time, crystals separated, and proved on examination to consist of the oxime of the ketone $C_6H_8O$ discovered by Looft in wood oils (Annalen, **275**, 366; this Journal, 1893, 919). The bulk of the mixture of oximes having remained liquid, it was treated with acetic anhydride, and an acetate boiling at about 135° under 10 mm. obtained. From this acetate the oxime boiling at 110—111° C. under 10 mm. was obtained by decomposition with alcoholic potash. It crystallised from ether in regular dodecagonal prisms melting at 104° C. The ketone was prepared from it by treatment with dilute sulphuric acid, and boiled at 67° under 10 mm., and formed crystals melting at 29·5° C. It appeared to be connected with the furfuran series, and to have the composition of α-acetylfurfuran. This body was prepared synthetically by warming ethyl pyromucate with ethyl acetate and a molecular proportion of sodium for 12 hours, isolating the ethyl pyromucylacetate formed, and decomposing it by boiling with dilute sulphuric acid. The α-acetylfurfuran thus prepared was identical with that extracted from the wood oil.—J. A. B.

*Phthalic Acid, Oxidation of Naphthalene by Permanganate for the Production of.* J. Procháska. Ber. **30**, [19], 3108—3109.

The observation that naphthalene is soluble in boiling water is utilised for its oxidation into phthalic acid by means of permanganate. Almost theoretical results are obtained, phthalonic acid being produced in the first place, and from the latter, phthalic acid. The reduction of permanganate is very rapid, and the best results are obtained when an excess of naphthalene is employed, the separation of which is easily effected. 100 grms. of commercial permanganate yielded 19 grms. of orthophthalic acid.—D. B.

*Hydrocarbons, High-molecular, Decomposition of, by Moderate Heat.* C. Engler. Ber. **30**, [18], 2908—2920.

Three classes of hydrocarbon oils were experimented upon, *viz.*, oils resulting from the decomposition of high-boiling petroleum oils by simple distillation; those formed during the "cracking process"; and oils produced by distilling heavy mineral oils under high pressure.

In the case of simple distillation, a Galician oil was employed, the fractions below 200° C. being removed, and the remainder—consisting chiefly of unsaturated hydrocarbons (this Journal, 1896, 191)—distilled and rectified A considerable difference was found to exist between the distillates formed under ordinary atmospheric pressure and those resulting from distillation *in vacuo*: in the former case the distillates became more fluid beyond a certain point, whilst in the other, the higher fractions were progressively thicker and of greater density. On rectification, too, it was found that the initial boiling point of the fractions from the ordinary-pressure distillation receded by more than 200° C., the fractions that came over at 315°—330° and 330°—340°, for example, commencing to boil at 80° and 82° C., and yielding 25—30 per cent. of hydrocarbons boiling below 200° C.; whereas the decrease in the boiling point of the fractions from the vacuum distillation did not exceed 27° C. The conclusion drawn by the author is that, under ordinary pressure, decomposition commences at temperatures above 200° C., and that as the temperature thereafter increases, so dissociation progressively exceeds any polymerisation that may occur, the originally heaviest fractions thus giving the greatest proportion of light hydrocarbons. He is also of opinion—based on experimental grounds—that the assumption put forward by Thorpe and Young, that the dissociation of paraffin of high melting point by distillation under pressure is solely due to the complex structure of the molecule, cannot hold good for the hydrocarbons now under consideration, but that the temperature of the distillation exercises a decisive influence on the results.

Further experiments with a high boiling (400° C.) petroleum from Java, showed that the decomposition-oils, resulting from distillation under pressure, contained (like the above-named Galician distillates) about 40 per cent. of unsaturated hydrocarbons, and that the percentage of these latter increased, in all the separate fractions, from 23 to 48 per cent., in proportion to the rise in the boiling points of the fractions.

In the second series a crude cracking-oil (from Galician petroleum) of sp. gr. 0·8769 was taken, and the distillate therefrom under 200° C. subjected to examination. For determining the olefines, two methods were employed: treatment with 2½ per cent. of fuming sulphuric acid, and bromination, the results showing that the former is sufficiently accurate for approximate determinations. The fraction below 85° yielded 10·8 per cent. of hexylene; that between 85° and 110°, 12·7 per cent. of heptylene (or 14·5 per cent. of octylene); and 17·0 per cent. of octylene (or 19·1 per cent. of nonylene) was obtained from the fraction 110°—135°, 23·6 per cent. of nonylene from that at 135°—160°, and 29·1 per cent. of decylene from the fraction 160°—185° C. Suitably concentrated fractions were examined for benzene hydrocarbons, benzene being detected in the fraction 77°—85°, toluene in the fraction 107°—114°, metaxylene (and probably orthoxylene) in the fraction 134° — 146°, and mesitylene and pseudocumene in the fraction 160 —185° C.

The paraffin series was isolated by removing the ethylenes, benzenes, &c., the following members being detected: hexane in the fraction 68°—71°, heptane in the fraction 95°—101°, octane in the fraction 124°—126°, nonane in fraction 150°—152°; and decane in fraction 173°—175° C. The naphthenes were extracted by the Heusler method, and the following identified: in fraction 78°—80°, hexanaphthene; fraction 100°—102°, heptanaphthene; fraction 117°—121°, octonaphthene; fraction 134°—137°, nononaphthene; fraction 160°—162°, decanaphthene; and fraction 180°—182°, undecanaphthene.

The third set of experiments was performed with an oil prepared from brown-coal oil by the Krey pressure-distillation process. Its specific gravity was 0·8301, and the elementary composition, C, 84·67—84·83; H, 13·93—14·30; S, 0·80 per cent. The following constituents were identified:—*Paraffins:* pentane, b.p. 37°—39° C.; hexane, b.p. 68°—69°; heptane, b.p. 97·5°—98·5°; octane, b.p. 124°—125°; nonane, b.p. 148·5—149·5. *Olefines:* fraction 35°—40° C., amylene 44 per cent.; fraction 67°—70°, hexylene 41 per cent.; fraction 96°—99°, heptylene 43 per cent. The probability of the presence of naphthenes was indicated by the modification of the specific gravity and refraction of the individual fractions. Of benzene hydrocarbons the fraction 75°—90° contained benzene, and the fraction 160°—180° pseudocumene and mesitylene. Here also the formation of paraffins from hydrocarbons poorer in carbon must be assumed to occur during distillation under pressure.—C. S.

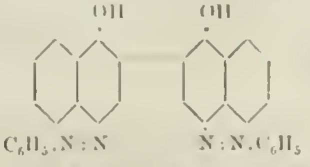
*Native Asphalt, Detection of Coal-Tar Distillation Products in.* J. Bengough. Mitt. d. k. k. tech. Gewerbe-Museums in Wien, 1897, **7**, [9, 10, and 11], 217—218.

*See under* XXIII., *page* 185.

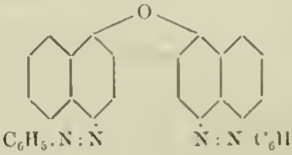
# IV.—COLOURING MATTERS AND DYES.

*Benzene-azo-α-Naphthol.* O. N. Witt and J. Dedichen. Ber. **30**, 2655—2667.

The authors point out that benzene-azo-α-naphthol is generally incorrectly assumed to be analogous to hydroxyazobenzene They refer to the differences in alkylation, and show that it has more the properties of an amido acid, since it gives salts both with metallic oxides and with strong acids. Moreover, the ethers have still basic properties, whilst those of hydroxyazobenzene are almost neutral substances. The preparation of benzene-azo-α-naphthol succeeds best in alcoholic solution, since when prepared in aqueous solution it is always accompanied by varying amounts of the disazo compound, from which it is only separated with difficulty. A mixture of 93 grms. of aniline, 200 c.c. of fuming hydrochloric acid, and 250 c.c. of water, is cooled externally with ice and diazotised with the theoretical quantity of sodium nitrite dissolved in as small a quantity of water as possible. This solution is then run into 155 grms. of α-naphthol dissolved in 2 litres of alcohol. The whole is allowed to stand 24 hours, when the hydrochloride of the benzene-azo-α-naphthol separates out in almost theoretical yield. The free base separates from acetic acid in reddish-brown crystals melting at 206° C. Prolonged boiling with acetic acid gives rise to the formation of a red insoluble substance, and the product is therefore purified preferably by conversion into the potassium salt. The decomposition product referred to is an oxidation product, and can be obtained by the action of nitrosodimethylaniline in acetic acid solution. The yield is, however, poor, and a better result is obtained as follows:—110 grms. of the potassium salt are dissolved in 1 litre of hot water, and the solution filtered into 1 litre of alcohol. The whole is then stirred into 1,500 c.c. of alcohol, 250 c.c. of concentrated hydrochloric acid, and 500 c.c. of water containing 250 grms. of commercial ferric chloride. The product, which separates out after heating the mixture 12 hours on the water-bath, is filtered off and washed with alcohol and water, when it is sufficiently pure for most reactions. For analysis it is converted into the acetyl derivative (reddish needles melting at 265° C.), and this is then hydrolysed with alcoholic potash. The substance is insoluble in the ordinary solvents, and melts at 246° C. It no longer possesses any basic properties, but is a dibasic phenol. It has the composition $C_{32}H_{22}N_4O_2$, and is formed by the condensation of two molecules of benzene-azo-α-naphthol with the elimination of two atoms of hydrogen. In view of the fact that the Badische Anilin and Soda Fabrik has taken out a series of patents (Eng. Pats. 6697, 12,303, and 17,260 of 1895; this Journal, 1896, 588) on the oxidation of simple azo dyestuffs resulting in the formation of diphenyl derivatives, the authors compared the product with that obtained from tetrazodiphenyl and α-naphthol, and also reduced their substance. No similarity could be detected, nor was benzidine one of the reduction products. With stannous chloride, in addition to aniline, a highly unstable di-*p*-amidonaphthol hydrochloride was obtained, which on oxidation with nitric acid gave a quinone similar to, but not identical with, α-naphthoquinone. It melts between 270 and 280° C., and gives on distillation with zinc dust a colourless hydrocarbon melting at 186° C. and identical with Watson Smith's iso- or β-dinaphthyl (J. Chem. Soc. 1877, 551; 1879, 220). The new dyestuff therefore has the following formula:—



A similar product was obtained from Tropæolin O O O, but was not further investigated. By warming the sulphuric acid solution of the new dyestuff on the water-bath, until a sample no longer gives a red colour with alcoholic potash, and then pouring the melt into water, another product is obtained, which, after crystallisation from xylene, separates in shining orange-red crystals melting at 291° C., and is a derivative of dinaphthylene oxide, having the formula—



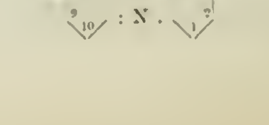
This, and the compound from which it was obtained, appear to have been already prepared by Fischer and Hepp (Ber. **25**, 2731; this Journal, 1893, 31), although in an impure state, and incorrect conclusions were drawn as to their constitution.—T. A. L.

*The Saccharеïns: Reply to Sisley.* P. Monnet and J. Koetschet. Bull. Soc. Chim. 1897, **17**, 1030—1031.

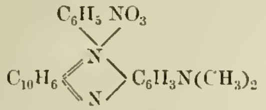
Sisley has made a claim for priority (Bull. Soc. Chim. 1897, 821) in the discovery of the sacchareïns described by the authors (this Journal, 1897, 728). Sisley's work was published after the conclusion of that of the writers, and, moreover, deals with colouring matters obtained from resorcinol and saccharine in the presence of excess of sulphuric acid. This reagent decomposes saccharine below 100° C.; the colouring matters obtained must therefore be derivatives of *o*-sulphobenzoic acid.—A. C. W.

*Phenylisonaphthophenazonium Salts, and Action of Amines thereon.* F. Kehrmann and W. Helwig. Ber. **30**, 2629—2636.

The action of β-naphthoquinone on phenyl-*o*-phenylene diamine (Ber. **29**, 2318; this Journal, 1896, 802), yields two condensation products. One of these—amounting, however, to only about 2 per cent.—is identical with the product obtained by removing the amido group either from rosinduline or from Nietzki-Otto's isomeride (Ber. **29**, 2969). The chief product is phenylisonaphthophenazonium hydroxide, which can also be obtained from a previously described blue isorosinduline (Annalen, **290**, 270; this Journal, 1896, 536; see also 708). The investigation of certain salts of this substance, and the action of amines on it, has led to the discovery of a fourth isomeride of rosinduline, of the 30 which are possible. The ferric chloride double salt $C_{22}H_{15}N_2ClFeCl_3$, crystallises from alcohol in orange-yellow plates melting at 200·5° C. The nitrate can be obtained by precipitating the iron with ammonium carbonate, filtering into dilute nitric acid, and salting out with sodium nitrate. It can also be separated directly from the product of condensation of β-naphthoquinone and *o*-amidodiphenylamine by salting out the solution with sodium nitrate, and crystallising the precipitate from alcohol. The first fractions consist of pure iso-salt, whereas the mother-liquor contains phenylnaphthophenazonium nitrate. The presence of this substance was detected by boiling a mixture of the nitrates with an excess of alcoholic dimethylamine. Under these conditions, the salts of phenylnaphthophenazonium are converted through dimethylrosinduline into rosindone, whereas the salts of phenylisonaphthophenazonium yield stable salts of dimethylisorosinduline. Phenylisonaphthophenazonium nitrate forms reddish-yellow crystals similar to potassium bichromate, and dissolves easily in water and alcohol with the same colour. It melts at 229° C., and dissolves in concentrated sulphuric acid with a violet colour, which turns yellowish-red on the addition of water. Amongst other salts, $(C_{22}H_{15}N_2)_2PtCl_6$ forms an orange-red crystalline powder, $C_{22}H_{15}N_2AuCl_4$ melts at 240° C., and $(C_{22}H_{15}N_2)_2Cr_2O_7$ is a scarlet powder sparingly soluble in cold water. By the prolonged action of alcoholic ammonia at the ordinary temperature on the ferric chloride double salt, the new isorosinduline chloride 4 above referred to is formed. It has the formula—



and, apart from its colour—violet-red needles dissolving to a magenta-coloured solution—it is very similar to rosinduline. Dimethylamine, as already mentioned, forms dimethylisorosinduline, of which the nitrate has the formula—

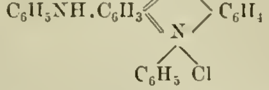


This salt forms coppery blue prisms melting with decomposition at 220° C., and is easily soluble in water and alcohol to blue solutions. Concentrated sulphuric acid dissolves it to a brown solution, which on dilution passes through green to blue. The gold chloride salt $C_{21}H_{20}N_3AuCl_4$ separates in violet-blue crystals melting at 203° C. with decomposition. By employing aniline in place of dimethylamine, phenylisorosinduline is formed, the chloride of which is a heavy coppery crystalline powder almost insoluble in cold water. With regard to the shade of the isorosindulines described, it is interesting to note that whilst the amido derivative itself is red, its dimethyl and phenyl derivatives are pure blue dyestuffs. Most of the monamidoazonium compounds hitherto known have had a red, or at most a reddish-violet colour.—T. A. L.

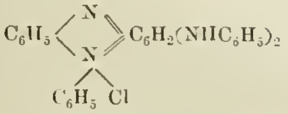
### *Phenylphenazonium and Phenylnaphthophenazonium Salts, and Action of Alkalis and Amines thereon.* F. Kehrmann and W. Schaposchnikoff. Ber. 30, 3620—2628.

Salts of phenylphenazonium and of phenylnaphthophenazonium have already been described (Ber. 29, 2967), and reference has also been made to the action of amines and alkalis on them (Ber. 29, 2316; this Journal, 1896, 802). The present paper, describing certain other salts, refers more particularly to their decompositions. Thus, the action of ammonia on phenazonium yields aposafranine, and naphthophenazonium gives rosindone with soda-lye. The latter reaction takes place in such a manner that, simultaneously with the rosindone, the hydrazonium compound is formed; this is reconverted by the atmospheric oxygen into phenylnaphthophenazonium, which again undergoes decomposition. It appears probable that Nietzki's formula for rosindone is correct. This involves the consideration that the indones exist as inner salts connecting the strongly basic azonium hydroxyl and a phenolic hydroxyl in the ring, or, in other words, as salt-like anhydrides of oxyazonium bases. This view has received some further confirmation, since Fischer and Hepp have shewn that the indones are converted by phosphorus pentachloride into the chlorides of chlorinated azonium bases. It is, however, also possible that the indones exist as *p*-quinonoid anhydrides, although perhaps rosindone and aposafranone do not belong to this group.

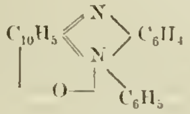
With regard to the phenylphenazonium salts, the ferric chloride double salt $C_{18}H_{13}N_2ClFeCl_3$ forms shining light reddish-brown needles melting at 186° C., easily soluble in water, boiling alcohol, and acetic acid with a yellowish-red colour, sparingly soluble in cold acetic acid and alcohol, and almost insoluble in saturated aqueous ferric chloride, 20 per cent. hydrochloric acid, and ether. When a concentrated alcoholic solution of the ferric chloride double salt is treated with an excess of soda lye, filtered, and air is passed through the solution, a yield of 50 per cent. of aposafranone is obtained on evaporating the alcohol, salting out and crystallising the residue from benzene. As already described by Jaubert (Ber. 28, 275; this Journal, 1895, 564), it forms golden-red plates melting at 242° C. The action of alcoholic ammonia in place of soda lye yields aposafranine chloride, whilst dimethylamine gives dimethylaposafranine nitrate, if sodium nitrate be used to salt out the dyestuff. By the action of aniline on the double salt, two products are formed, the one being phenylaposafranine chloride—



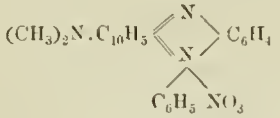
identical with that obtained by Fischer and Hepp by the action of aniline on chlorophenylphenazonium chloride (Ber. 30, 1831; this Journal, 1897, 1010), and the other anilidophenylaposafranine chloride—



The latter, being insoluble in hot dilute hydrochloric acid, can be separated by this reagent, and after crystallisation from alcohol was characterised by the usual tests. The salts of phenylnaphthophenazonium behave in a similar manner. The alcoholic solution of such a salt is treated with concentrated soda lye, and air is passed through the liquid, which rapidly turns red. After evaporating the alcohol, the residue is washed with hot water, and finally recrystallised from benzene, when it yields shining golden plates of rosindone—



melting at 259° C. By the action of alcoholic ammonia on the alcoholic solution of a naphthophenazonium salt, the latter is converted into rosinduline chloride, whilst dimethylamine yields dimethylrosinduline, of which the nitrate has the formula—



This salt crystallises in shining greenish prisms with $0 \cdot 5H_2O$ from dilute nitric acid, and dissolves in water to a yellowish-magenta shade without any appreciable fluorescence. When boiled with alkalis or alkaline carbonates, dimethylamine is given off, and rosindone crystallises out.
—T. A. L.

### *Tartrazine.* R. Gnehm and L. Benda. Annalen, 299, 100—130.

The authors in a previous paper (Ber. 29, 2017; this Journal, 1896, 707) have described the preparation of dyestuffs by the action of diazo compounds on tartrazine, and, in further investigating this reaction, have arrived at the following conclusions:—(1.) The formula proposed by Anschütz for tartrazine (Annalen, 294, 219; this Journal, 1897, 324) receives further confirmation, since the products obtained by the action of diazo compounds on tartrazine are unsymmetrically constituted substances and not osazones, and, moreover, a "mixed tartrazine" thus obtained contains only one carboxyl. (2.) Different hydrazines behave differently towards dioxytartaric acid. Thus, under similar conditions to those in which phenylhydrazine gives monohydrazone together with a little osazone, *m*-nitrophenylhydrazine gives mainly osazone, whilst *p*-nitrophenylhydrazine gives it exclusively. (3.) The ease with which the transformation into the pyrazolone derivative takes place, varies with the different hydrazines. For instance, diphenylizindioxytartaric acid can be warmed in an aqueous hydrochloric acid solution without change, whereas the *p*-nitrated osazone is thereby converted quantitatively into the pyrazolone, and the *p*-sulphonated product undergoes the transformation even on standing in the cold. (4.) Diphenylizindioxytartaric acid is, however, more easily converted by the action of alkalis into the pyrazolone than the *p*-nitro derivative. (5.) The imide hydrogen of either osazone cannot be replaced by heating with acetic anhydride. (6.) Both the osazones yield isomeric anhydro bodies—one a pyrazolone, the other an acid anhydride. (7.) The statement that the red substance from dioxytartaric acid phenyl osazone and acetic anhydride is an acetyl derivative (Ziegler and Locher), or a pyrazolone (Anschütz), is incorrect.—T. A. L.

*Ethyl-o- and p-Sulphobenzylaniline and Disulphobenzylaniline.* E. Jolles. Mitt. d. k. k. tech. Gewerbe-Museums in Wien, 1897, 7, [9, 10, and 11], 238—245.

Ethylbenzylaniline is obtained by the action of benzylchloride on ethylaniline, and forms a viscid oil boiling at 286° C. Sulphonation in the cold gives the ortho-, and, on heating, the para-sulphonic acid. The *o*-sulphonic acid is formed by dissolving the base in $2\frac{1}{2}$ times its weight of sulphuric acid monohydrate in the cold, and adding fuming sulphuric acid until a sample gives a clear solution with dilute ammonia. After liming, the calcium salt is converted into the easily soluble sodium salt having the formula $C_6H_5N(C_2H_5)CH_2.C_6H_4.SO_3Na[1:2]$. A neutral or alkaline solution gives a reddish-brown precipitate with ferric chloride, which becomes darker on boiling. When cooled, a dark green precipitate is obtained, which again turns red on boiling. The potassium salt is also easily soluble, whilst the barium salt is sparingly soluble, and crystallises with 3 mols. of water. On treating the sodium salt with dilute sulphuric acid and the calculated quantity of nitrite at 20° C., a dark greenish-brown solution of the nitroso compound is obtained, which is exactly neutralised and then precipitated with barium chloride and salt. The product, on condensation, gives the following reactions:—With resorcinol, a deep bluish-violet; with β-naphthol, a dark bluish-green; with gallic acid, a reddish-blue-violet; with pyrogallol, a dull blackish-green. A characteristic reaction of the nitroso compound is obtained by treating the solution with ferric chloride and passing in sulphuretted hydrogen. With a small quantity of ferric chloride, a reddish-violet coloration is obtained, whilst with an excess, the colour is bluish-violet. The *p*-sulphonic acid is obtained by heating ethylbenzylaniline with 4 parts of sulphuric acid monohydrate to 180° C. It is distinguished from its *o*-isomeride by the sparing solubility of the free acid in cold water. The sodium and potassium salts are easily, the barium salt, sparingly soluble. The nitroso compound also gives condensation products. Both *o*- and *p*-acids were condensed with benzaldehyde, and the leuco compounds separated. That of the *o*-acid is easily soluble in cold water whilst the *p*-compound is sparingly soluble. On oxidation with lead peroxide, the *p*-dyestuff was more easily salted out, and gave very slightly bluer shades than the *o*-compound when dyed as 1 per cent. solutions from an acid bath.

Dibenzylaniline is sulphonated by dissolving it in the cold in $3\frac{1}{2}$ parts of sulphuric acid monohydrate, and adding about twice the weight of fuming sulphuric acid. The reaction is complete when a drop dissolves to a clear solution in water. The sodium salt is easily, the barium salt, sparingly soluble in water, whilst the nitroso compound also forms a relatively sparingly soluble sodium salt. The combination of dibenzylaniline disulphonic acid in an alkaline solution with diazosulphanilic acid gives a yellow dyestuff which is very sensitive to acids, turning reddish-violet. The change is more marked than in the case of Methyl Orange, but it is not sufficiently sensitive to enable it to be used as an indicator for organic acids.—T. A. L.

*Naphthoresorcinol, Azo Derivatives of.* S. Blumenfeld. Mitt. d. k. k. tech. Gewerbe-Museums in Wien, 1897, 7, [9, 10, and 11], 236—237.

Kostanecki showed (Ber. 22, 3163; this Journal, 1890, 491) that the oxynaphthoquinonephenylhydrazide obtained by Zincke and Thelen (Ber. 17, 1811) was a derivative of naphthoresorcinol, and was in fact phenylazonaphthoresorcinol, $C_6H_5.N:N.C_{10}H_5(OH)_2[1:2:4]$. Naphthoresorcinol was obtained by Friedlaender and Rüdt (Ber. 29, 1609; this Journal, 1896, 531), and by combining diazobenzene chloride with this substance, the author obtained a product identical with the above. It forms a brick-red precipitate, crystallises from acetic acid, melts at 220° C., and gives an acetyl derivative melting at 176° C. and a diacetyl derivative melting at 123° C. The following table gives the dyestuffs obtained and some of their reactions by combining various diazo compounds with resorcinol and naphthoresorcinol:—

| Diazotised | Combined with | Dyestuff obtained. | Behaviour towards | | |
|---|---|---|---|---|---|
| | | | NaOH | Conc. HCl. | Acetic Acid. |
| Sulphanilic acid ........ | Resorcinol | Yellow | Brick-red | Brownish-yellow | Unchanged |
| | Naphthoresorcinol | Brownish yellow | Yellow | Brownish-red | Unchanged |
| Benzidine .............. | Resorcinol | Brown flocks | Red | Brownish-violet | Yellow |
| | Naphthoresorcinol | Dull bluish-violet | Dull bluish-red | Bluish-violet | Violet |
| Tolidine .. ............ | Resorcinol | Reddish-brown | Bluish-red | Brownish-violet | Yellow |
| | Naphthoresorcinol | Bluish-violet | Maroon | Blue | Violet |
| Dianisidine ............ | Resorcinol | Violet | Carmine | Brownish-violet | Unchanged |
| | Naphthoresorcinol | Insoluble | .. .. | .. .. | .. .. |

—T. A. L.

*Paranitrotoluene Sulphonic Acid, Oxidation of.* A. G. Green and A. R. Wahl. Ber. 30, 3097—3101.

In view of the ease with which *p*-nitrotoluene sulphonic acid is converted at the cost of the nitro group into dibenzyl and stilbene derivatives, it seemed possible that a similar oxidation might be brought about by the use of oxidising agents in an alkaline solution. The best reagent appears to be sodium hypochlorite, which converts *p*-nitrotoluene sulphonic acid first into dinitrodibenzyldisulphonic acid, and subsequently into dinitrostilbenedisulphonic acid. This latter product, on further oxidation with potassium permanganate in neutral or alkaline solution, splits up into *p*-nitrobenzaldehyde *o*-sulphonic acid. *Dinitrodibenzyldisulphonic acid* is prepared by dissolving 100 grms. of sodium *p*-nitrotoluene sulphonate in 1 litre of warm water, adding 500 c.c. of caustic soda lye (30 per cent. NaOH), and running in with constant agitation at 40°—50° C., 220 c.c. of a sodium hypochlorite solution (7 per cent. of available chlorine), avoiding the formation of any coloration. When all has been added, the stirring is continued, and the mixture is tested with starch iodide paper. When only a slight iodine reaction is apparent, the mixture is cooled, diluted with 2 litres of cold water, and the sparingly soluble sodium salt is filtered off and washed with brine. In order to purify it from any dinitrostilbenedisulphonic acid, it is dissolved in water and treated with permanganate so long as this latter is decolorised. After filtering from the manganese dioxide, the sodium salt is salted out, dissolved in water, and decomposed with an excess of hydrochloric acid. On cooling, the free acid separates in colourless plates. The acid, when dissolved in cold soda lye, does not give any coloration with reducing agents, such as zinc dust or phenylhydrazine; on boiling with caustic soda lye, it yields directly yellow dyestuffs without the formation of a red intermediate compound. Further oxidation with sodium hypochlorite converts it into dinitrostilbenedisulphonic acid, whilst it is not attacked by potassium permanganate in a neutral or faintly alkaline solution. *Dinitrostilbenedisulphonic acid* is obtained by dissolving 100 grms. of sodium *p*-nitrotoluene sulphonate in 2 litres of warm water and 200 c.c. of caustic soda lye, adding 500 c.c. of the sodium hypochlorite solution as above. The mixture is warmed at 50° C. until the sodium hypochlorite has almost disappeared. On cooling, the sodium salt of dinitrostilbene disulphonic acid crystallises out. The free acid is obtained by decomposing the sodium salt with an excess of hydrochloric acid. It crystallises in colourless needles. The acid is only slowly changed by heating with caustic soda lye alone, but in presence of reducing agents, forms at once yellow and orange dyestuffs. If treated in the cold, the formation of the dyestuff is preceded by that of a brilliant safranine coloration. This unstable substance appears to be identical with the red intermediate product obtained by the cautious action of soda lye on *p*-nitrotoluene sulphonic acid. Both red substances give pure blue lead salts. The reduction of the acid in an acid solution yields diamidostilbenedisulphonic acid. The so-called dinitrostilbenedisulphonic acid obtained by Fischer and Hepp (Ber. **26**, 2234; this Journal, 1894, 29) by oxidising their dinitrosostilbenedisulphonic acid, appears to have a different constitution, more especially as it is stated to be a dyestuff. *p-Nitrobenzaldeyde-o-sulphonic acid* is formed by dissolving 20 grms. of sodium dinitrostilbenedisulphonic acid in 500 c.c. of cold water, cooling to 10° C., and running in a solution containing 8·75 grms. of potassium permanganate in 175 c.c. of water. After removing the manganese dioxide, the filtrate is neutralised with hydrochloric acid and concentrated, when the sulphonic acid crystallises out on cooling in almost theoretical yield.—T. A. L.

*Fustic Extract, An Improved.* S. Kapff. Färber Zeit. 1897, **8**, 327—329.

The firm of G. Eberle and Co., of Stuttgart, have recently brought into the market a new variety of fustic extract under the name of "Santiago New Yellow." This dyestuff is produced by an oxidation process analogous to the ageing of logwood, and must be applied in dyeing with the aid of a non-oxidising mordant. According to experiments made in the dyeing school at Aix, the new product gives deeper shades than ordinary fustic extract when applied to cotton with acetate of iron, alum, &c. as mordants, but when a "saddening" process with potassium bichromate is employed, ordinary fustic extract gives the better result.

There are two brands of the dyestuff, E and K; the former is suited for wool dyeing when non-oxidising mordants—*e.g.*, potassium bichromate and tartaric acid—are employed; whilst the K brand is less readily over-oxidised, and is recommended for use on cotton together with logwood for mode colours and dead blacks.—R. B. B.

*Schiff's Reaction applied to some Substituted Magentas.* Bull. Soc. Chim. 1897, **17**, 998.

*See under* XXIII., *page* 186.

## PATENTS.

*Methyleneditannic Acids from Formaldehyde and Tannin, Impts. in the Manufacture of.* E. C. H. Dürkopf, Darmstadt, Germany. Eng. Pat. 6686, March 1, 1897.

*See under* XVIII. C., *page* 178.

*Blue Mordant-dyeing Colouring Matters, Impts. in the Manufacture of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 1962, Jan. 25, 1897.

The specification refers to an electrical method for the production of Anthracene Blues from dinitro-anthraquinones. Such dyestuffs have already been described in Eng. Pat. 13,029 of 1892 (this Journal, 1893, 921). The method now described consists in dissolving the dinitro-anthraquinone in fuming sulphuric acid at the ordinary temperature in a cell containing the cathode. The anode is placed in concentrated sulphuric acid (95 per cent. $H_2SO_4$), separated from the cathode cell by a suitable diaphragm. The following example gives the quantities employed:—About 10 kilos. of 1.4′-dinitro-anthraquinone are mixed with 200 kilos. of fuming sulphuric acid (40 per cent. $SO_3$) in the cathode cell, whilst the anode dips into concentrated sulphuric acid. An electric current of about 80 ampères per square decimetre of electrode surface is passed through the cell, and the temperature is kept at about 100° C. until the dinitro-anthraquinone has disappeared and a sample of the melt dissolves in sulphuric acid with a red, and in water with a brown, colour. The whole is then rapidly cooled, poured into ice-water, boiled up, filtered, and the dyestuff is precipitated by salt from the red filtrate, filter-pressed, and dried. By continuing the action of the electric current for a longer time, until a test dissolves with a brown colour in sulphuric acid, a dark violet precipitate, which is also a mordant dyestuff, is obtained on pouring the melt into water. A similar sparingly soluble dyestuff may be obtained by heating the soluble dyestuff obtained as above with 10 times its weight of concentrated sulphuric acid (95 per cent. $H_2SO_4$), in the manner described in Eng. Pat. 19,589 of 1891 (this Journal, 1893, 258). In place of the 1.4′ dinitro-anthraquinone, other nitro-anthraquinones or mixtures thereof can be used, such as α- and δ-dinitro-anthraquinone, or mixtures of these with one another or with the 1.4′ derivatives such as are obtained by nitrating anthraquinone.
—T. A. L.

*Colouring Matters of the Rhodamide Series, Impts. in the Manufacture of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 2655, Feb. 1, 1897.

The dyestuffs described are obtained by sulphonating monobenzylrhodamines and monobenzylrhodols, which can be produced by fusing an alkyl-benzyl-*m*-amidophenol with phthalic anhydride, according to Eng. Pat. 7258 of 1891 (this Journal, 1892, 345), at a temperature of 120°—140° C., and condensing the product with a *m*-amidophenol or *o*-amido-*p*-cresol (either being alkylated or not). The benzylrhodols are obtained by using resorcinol instead of *m*-amidophenol. These products may then be sulphonated either with ordinary sulphuric acid at the temperature of the water-bath, or with fuming sulphuric acid in the cold. The former series dye tannined cotton, and have little affinity for wool or silk: whilst the latter dye wool and silk from an acid bath in shades which exhibit, even on wool, the characteristic fluorescence of rhodamine. About 23 kilos. of ethyl-benzyl-*m*-amidophenol and 15 kilos. of phthalic anhydride are stirred together for about 1 hour at 125° C., until the melt solidifies to a crystalline mass, when about 16·5 kilos. of diethyl-*m*-amidophenol are added, and the temperature is raised to 130° C. The fluid mass has then added to it 30 kilos. of potassium bisulphate, and the temperature is raised to 175° C., until no further increase in the formation of colouring matter is observed. This takes about 1 hour. The melt is then cooled, powdered, washed free from potassium sulphate, and the residue, consisting mainly of the sulphate of triethyl-benzyl-rhodamine is dried. One kilo. of this product is dissolved in 7 kilos. of concentrated sulphuric acid, and warmed on the water-bath until a test dissolves in dilute soda solution. The melt is then poured into 10 litres of water, when the sulphonic acid separates, and is purified by dissolving in water, adding sodium carbonate, and salting out. A different sulphonic acid is obtained by dissolving 1 kilo. of the triethyl-benzyl-rhodamine sulphate in 5 kilos. of fuming sulphuric acid below 15° C. When dissolved, the melt is poured into a small quantity of ice and water, and the separated sulphonic acid, which is more soluble than the previous one, is converted into the sodium salt as above.—T. A. L.

*New Disazo Dyes [Black] on Cotton, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 2874, Feb. 3, 1897.

The patentees claim the production of black shades on cotton fibre by dyeing the fabric with 4 per cent. of Primuline, diazotising, and developing in a bath containing about 0·13 per cent. of the hydrochloride of 1.1′- or of 1.4′-naphthylene diamine, or a mixture of the two. The 1.1′ isomeride gives a more greenish shade of black, whilst the mixture produces a neutral black. The preparation of the dyestuff has already been referred to in Eng. Pat. 18,317 of 1893 (this Journal, 1894, 879), but when prepared on the fibre according to the present specification, it is very fast to fulling, and is not dissolved even by boiling with dilute soda solution.—T. A. L.

*Disazo Colouring Matters [Cotton Blues and Violets], The Manufacture or Production of Substantive.* W. E. Heys, Manchester. From "The Chemical Works formerly Sandoz," Basle, Switzerland. Eng. Pat. 4703, Feb. 22, 1897.

The disazo dyestuffs of Ger. Pat. 58,076, obtained by combining one molecular proportion of the tetrazo compound from a *p*-diamine with two molecular proportions of 1.3.3′.1′-naphtholtrisulphonic acid, although giving bright shades, are not suitable for dyeing unmordanted cotton. The patentees have discovered that cotton dyestuffs can be obtained by combining equimolecular proportions of a tetrazo compound and the naphtholtrisulphonic acid above mentioned, and further combining the intermediate compound so formed with one molecular proportion of β-naphthol. The following is a typical example of the method employed :—24·4 kilos. of dianisidine are dissolved in 300 litres of water, together with 58 kilos. of hydrochloric acid (21° B.), diazotised with 13·8 kilos. of sodium nitrite at a temperature not exceeding 5° C. To this is then added, slowly and with continual agitation, a solution (cooled to 0° C. in 800 litres of water) of 45 kilos. of the trisodium salt of 1.3.1′.3′-naphtholtrisulphonic acid. The whole is then made alkaline with 35 kilos. of soda-ash dissolved in 250 litres of water, the intermediate compound in the form of a bluish-red solution being formed immediately. After agitating for 3—5 minutes, a cold solution of 14·4 kilos. of β-naphthol and 4 kilos. of caustic soda in 100 litres of water is run in. The colour thereupon changes to a pure blue. In the course of a few hours, salt is added, the solution is heated under pressure at 110° C., filter-pressed whilst hot, and dried. The dyestuff dissolves to a pure blue solution, which is not affected by sodium carbonate, but turns reddish-violet with caustic soda. Strong sulphuric acid dissolves it with a greenish-blue colour, and on adding water, the violet sulphonic acid of the new dyestuff is precipitated. The new dyestuff gives bright blue shades on unmordanted cotton between Diamine Blue 2 B and 3 B when dyed from a boiling bath containing salt or sulphate of soda, which may be made slightly alkaline by adding sodium carbonate, soap, or sodium phosphate. By employing benzidine in place of dianisidine, pure violet shades are obtained, whereas tolidine gives violet-blue shades.—T. A. L.

*Diphenylethane and of Diphenylethylene, The Manufacture and Production of Derivatives of.* A. G. Green, A. R. Wahl, and The Clayton Aniline Co., Ltd., Manchester. Eng. Pat. 5351, Feb. 27, 1897. (Ber. **30**, 3097 ; see Abstract above.)—T. A. L.

*Paradioxythiazin and Paramidoxy Thiazine, Impts. in the Preparation of.* H. R. Vidal, Paris, France. Eng. Pat. 5691, March 3, 1897.

When *p*-amidophenol is heated with sulphur, only a small quantity of *p*-dioxythiazine is obtained, the ammonia formed in the reaction reacting with this substance, and yielding *p*-dioxytetraphene trithiazine. An almost theoretical yield of *p*-dioxythiazine can be obtained by heating together *p*-amidophenol, hydroquinone, and sulphur, the ammonia liberated by the action of the sulphur on the *p*-amidophenol reacting with the hydroquinone, and in this way giving *p*-dioxythiazine also. Heat together, in an iron vessel provided with an agitator, 11 kilos. of *p*-amidophenol, 11 kilos. of hydroquinone, and 3·2 kilos. of sulphur, until the melt becomes too thick to stir. It is purified by dissolving in sodium carbonate, and consists almost entirely of *p*-dioxythiazine. In a similar manner, *p*-amidoxythiazine can be obtained by reacting with sulphur on a mixture of equal parts of *p*-phenylenediamine, *p*-amidophenol, and hydroquinone.—T. A. L.

*Ink [Invisible and Indelible Writing].* J. Moeller, Hamburg. Eng. Pat. 21,991, Sept. 25, 1897.

100 parts of alum are dissolved in water, the solution boiled and 100 parts of white garlic juice added. The writing is invisible till the paper is heated ; then the ink cannot be removed by water.—F. H. L.

## V.—TEXTILES : COTTON, WOOL, SILK, Etc.

*Silk, Weighting, with Stannic Chloride Solutions.* A. Fraenkel and J. Fasal. Mitt. d. k.k. tech. Gewerbe-Museum in Wien, 1897, **7**, [9, 10, and 11], 230—231.

It is frequently found in weighting silk, by immersing it in a bath of stannic chloride solution (about 30° B.), washing, and fixing with sodium phosphate, that after repeatedly using the tin chloride solution, although brought up to the same degree Baumé each time by the addition of solid stannic chloride, the silk exhibits differences in the quantity of tin contained, *i.e.*, in the weighting. With a view of investigating the cause of this phenomenon, the authors have titrated the solutions for tin and chlorine, the following table giving the results obtained, I. being the original solution, II. after using three times, and III. after using nine times :—

| Tin Chloride Solution. | Tin. | Chlorine. | Combined Chlorine calculated. | Chlorine Excess present as HCl. |
|---|---|---|---|---|
| I. | 14·73 | 18·31 | 17·72 | 0·59 |
| II. | 14·80 | 17·95 | 17·81 | 0·14 |
| III. | 15·22 | 18·49 | 18·31 | 0·18 |

The numbers are expressed in grms. per 100 c.c. of solution, and the results show that the differences found in the weighting are not due to the absorption of basic salts by the silk, since this would have made the solution gradually more acid.—T. A. L.

*Textile Fabrics, Waterproofing.* Leipziger Färber- und Zeugdr. Zeit. 1897, **46**, [12], 538—539.

Textile fabrics are frequently rendered waterproof by the precipitation on them of aluminium hydrate. This method is unsatisfactory, as the fabrics in time lose the alumina, which falls away as dust, and with it they lose their water-repelling power. Another method, consisting in the impregnation of the fabrics with ammoniacal copper oxide is more rational and better, as a permanent surface-gelatinisation of the fibres is produced by it, but it has not hitherto been much employed, owing to the diminution in strength which results in the first instance from excessive action of the ammoniacal compound on the fibres, and then from the continued action of the copper oxide which is left in them, and which is affected by the atmospheric moisture and other agencies.

Breitenfeld has discovered (German patent) that when tissues are impregnated with tannin substances, albumin, or ferrocyanides, and are then treated with ammoniacal copper oxide, or are first treated with the latter and then with any of the former compounds, the action of the ammoniacal copper oxide upon the tissues is considerably moderated, and coloured compounds are formed, which, besides being inert and odourless, are stable under ordinary atmospheric conditions.—E. B.

*Silk, Artificial ; Recognition of.* P. Truchot. Ann. de Chim. Anal. ; through Leipziger Farber- und Zeugdr. Zeit. 1897, **46**, [12], 535.

*See under* XXIII., *page* 186.

### PATENTS.

*China-Grass and other Fibres, A New or Improved Process of and Apparatus for Treating.* H. Lane, Blockley, Worcester. Eng. Pat. 29,727, Dec. 24, 1896.

Hanks of china-grass or other fibres, are placed in frames, made of wire bent into two or more loops, and are then packed loosely into a cylinder made of wire or perforated metal. The cylinder is made up in sections, with doors attached, so that any sector can be removed if desired. The whole is situated in a tank, and concentric with the diameter of the cylinder is a steam pipe with jets along its length.

The tank is filled with soft-soap solution, 1 lb. of soap to 1 lb. of fibre being a convenient strength for very refractive fibres; the cylinder is then rotated, and the steam turned on. After removing the gummy substances, the liquor is run off, and the fibre, still in the cylinder, treated with a dilute solution of chloride of lime, to bleach it. Finally, the fibres are thoroughly rinsed with water, and removed.

—C. M.

*Textile Materials, Impts. in Mercerising.* A. G. Boubon, Troyes, France. Eng. Pat. 3218, Feb. 6, 1897.

It is well known that, during the process of "mercerisation," fabrics shrink very considerably, and, moreover, further shrink when being finally washed.

In this invention, the material, placed on a roller after having been cleansed of all grease, and still moist, is passed round two other rollers, and, as it leaves, the mercerising liquid is allowed to drop on to it in the form of a spray. Immediately, the fabric is passed on to a fourth roller, where it is wound tightly, and left for about 12 hours. At the end of this time there is no shrinkage on unwinding and washing, and the material can be bleached and dyed in the ordinary way. It is preferable to use alkaline mercerising agents, as, owing to the long time the agent acts on the material, acid would probably destroy the fibres.—C. M.

*Waterproof Fabrics and Ropes, and other Waterproof Articles, Improved Manufacture of.* A. L. Kennedy, Philadelphia. Eng. Pat. 22,205, Sept. 28, 1897.

Small threads of pyroxylin, or acetate of cellulose, are woven into fabrics and ropes, and the materials, when finished, are treated with solvents of these substances, so that they diffuse and impregnate the raw fibres and ordinary threads. In this manner the inventor claims to produce waterproof materials, which are not changed in structure or appearance, inasmuch as they are uncoated and unglazed.

—C. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Oxycellulose, Note on.* J. Allan. J. Soc. Dyers and Colorists, **14**, [1], 6—8.

The author states that much of his work has been confirmed by Vignon (this Journal, 1897, 908). However, he thinks that the method of oxidation used by Vignon yielded a considerable proportion of hydrocellulose as well as oxycellulose. Hence he questions the figures and analyses of Vignon, until it can be shown that his method of preparation gives oxycellulose only. The effect of several oxidising agents was tried, but the best results were obtained with alkaline permanganate. The strength of the oxidising agent must be carefully regulated, since oxycellulose is much more sensitive to oxidising agents than is cellulose, and therefore any endeavour to push the oxidation too far, results in the destruction of the oxycellulose first formed.

*Preparation.*—35 grms. of purified cotton yarn were heated with 5 grms. of $KMnO_4$ and 25 c.c. of caustic soda (10° Tw.) in 500 c.c. of water until the pink colour of the $KMnO_4$ was destroyed. The residue, which was black, owing to the deposition of peroxide of manganese, was well washed with hot pure water, and then extracted with warm NaHO (35°—40° Tw.).

The clear yellow liquid was acidified with hydrochloric acid, which deposited a bulky white precipitate of oxycellulose. This precipitate was washed with alcohol and then dried in a desiccator over $CaCl_2$ to a hard semi-transparent mass, which broke with a well-marked conchoidal fracture. On weighing the mass it gave a yield on the original cotton of only 7·3 per cent.

Oxycellulose does not dissolve in caustic soda solution of less than about 15° Tw. In stronger solutions (25° Tw. and upwards) it dissolves only on prolonged contact; in hot solutions it dissolves much more easily to a yellow solution.

Hitherto, when cellulose has been treated with oxidising agents until seemingly completely destroyed, it has been considered to have been converted wholly into "oxycellulose." This is wrong, because on repeatedly extracting such "oxycellulose" with NaHO, it showed a continually decreasing affinity for basic dyes, whilst the substance obtained by acidifying the caustic soda extracts, absorbs such dyes freely.

Two trials, to determine the use of a solution of oxycellulose in caustic soda, as a mordant for basic dyes, gave results of negative value.—J. E. H.

*Imitation Crêpons, The Manufacture of.* L. Caberti. Färber Zeit. 1898, **9**, [1], 1—2, and [2], 18—21.

The chief difficulty in the manufacture of crêpons lies in the limitation of the mercerising action to the parts intended. This is done by (1) Printing the alkali directly, or (2) Printing first a substance which prevents the mercerising action. In the first case the unprinted parts make the crêpon due to the shrinkage of the mercerised parts. In the second case the reverse effect is usually obtained, since the printed parts, protected from the influence of the alkali, are drawn by the surrounding mercerised part, and thus form the crêpon.

After a few experiments the author directed his attention to method (2), although method (1) undoubtedly presents certain advantages, *viz.*:—

(*a.*) The work is done more rapidly, since the pieces only need washing after printing.

(*b.*) Much less consumption of alkali.

(*c.*) More easily carried out, since the alkali bath, which is necessary in method (2), is omitted.

Its disadvantages are:—

(*a.*) The alkali has to be thickened to get good printing colours.

(*b.*) It is absolutely necessary to avoid any contractile effect upon the pieces during and after printing. This is not easily avoidable in the case of the usual printing machine.

Method (2), depending on the protection of the fabric in different places from the influence of the alkali, possesses the following advantages:—

(*a.*) Great facility in preparing the printing-colour.

(*b.*) The omission of any special precaution during printing.

(*c.*) The possibility of steaming the printed pieces and consequently obtaining a much faster colour.

The author has always used a solution of gum arabic. He found no advantage in adding such salts as alum.

Gum alone is quite sufficient for a good crêpon.

However, the gum can be replaced partly or even entirely, with good results, by yellow dextrin.

Solutions of gum tragacanth do not act as a resist for the alkali, so that they cannot be employed along with gum arabic. To obtain fast bright shades, substantive colours printed with egg albumin are used. These resist the action of alkali and the acid bath which usually follows the washing out of the alkali.

Certain tannin-colours have been proposed, but the author finds they are all more or less sensitive to alkalis, and stain the white much more than the substantive colours.

Practically all substantive colours are adapted for this method. The author used with good results, Diamine pure Blue, Benzo pure Blue, Benzocyanine, Naphthyl Blue B, Diamine Rose B D, Geranine, Erika, Thioflavin S, Cotton Yellow G and R, Chrysophenine, Chrysamine, &c.

Several of the substantive colours can be applied with chrome mordant—*e.g.*, chromium acetate, printed and steamed—such as Chrysamine, Cotton Yellow G and R, Geranine, Diamine Orange D, &c. These colours, after steaming 1—1½ hours, are very fast to soaping at 40° C. The author gives a few recipes for the printing-colours used by him.

The general procedure is to print the colour upon the light fabric, dry, steam by passing once or twice through the Mather-Platt steaming chamber, treat with alkali, rinse with water, pass through an acid bath, and dry.

Special precautions are to be observed in carrying out the operations mentioned.

(1.) The fabric must be well and completely bleached. The author only obtained poor results on half-bleached material.

(2.) In steaming, dry steam must be used, for the printed resist must be very dry in order to obtain the best effect. The author finds it advantageous to run the steamed pieces at once over a strongly-heated drying cylinder, and to pass through the alkali whilst still hot.

The passage through the caustic alkali is the most important part of the whole process, and must be performed with great care.

(*a.*) The bath must be 38°—42° B., and free from $Na_2CO_3$, which has no action on the cotton. The temperature of the bath should be as low as possible.

(*b.*) This is done in the usual padding machine. The piece is entered into the machine in folds, where it has plenty of room for expansion. It is at once seen whether the crépon effect has been successfully produced or not. If this inflation does not occur, it is noticed in the washing that the crépon is only very incompletely shown, if at all.

(*c.*) The squeezing rollers must be very heavy. If too much caustic soda remain in the fabric, it softens the resist and stains the fabric. In such cases the crépon is partly or wholly absent. The author used two copper rollers, one of which was covered with stout linen cloth (3—4 m. for a roller of 11—15 cm. diameter). This was wound with a long piece of linen (120—130 cm. long) of triangular shape, beginning at the base. This piece, again, was wrapped with 3—4 m. of linen.

(*d.*) Since the caustic soda which is upon the piece previous to squeezing, easily attracts $CO_2$ from the air, only so much alkali is put into the liquor trough of the machine as is necessary to cover the piece which runs under the small rollers.

The alkali is continually replenished from a vessel fitted with a tap.

(*e.*) The caustic soda solution is filtered, and stored in steppered bottles.

After the passage through alkali, the pieces are allowed to stand 15—20 minutes, then washed, care being taken not to twist the pieces so as to injuriously affect the crepon.

After the first washing, pass through $H_2SO_4$ (2°—3° B.), and finally wash in clean water, hydro-extract, and dry.

The author prefers to dry the cloth in the open air, and then to dry it on the tentering machine in order to make the borders entirely uniform. Of course the workmen must use india-rubber gloves.

The author finally mentions one or two methods of obtaining various effects, such as printing mixed stripes or lines upon a white ground, or upon a ground printed with aniline black.—J. E. B.

### *Cerium Lakes, compared with those of Iron and Tin.* G. Matschak. Mitt. d. k.k. tech. Gewerbe-Museums in Wien, 1897, 7, [9, 10, and 11], 232—235.

The table below gives the results obtained with various dyestuffs on the three mordants. In the case of the cerium, the mordanting of the thick woollen cloth was carried out by heating nearly to boiling point, for 1 hour, with 4 per cent. of cerium sulphate and 3 per cent. of tartaric acid; the wool was mordanted with tin in the usual manner, with 4 per cent. of tin salt and 2 per cent. of oxalic acid, whilst the mordanting with iron was effected with 12 per cent. of ferrous sulphate and 12 per cent. of tartaric acid, the wool being subsequently exposed to the air in a damp condition, in order to thoroughly oxidise the ferrous oxide. After dyeing, the samples were washed with distilled water, and afterwards twice soaped at 60°—70° C. with a 1 per cent. soap solution. The acid test consisted in boiling the sample for 15 minutes with dilute sulphuric acid (1 grm. per litre). With regard to the fastness to acids, the tin lakes, with the exception of those of Alizarin and Alizarin Orange, are least changed. Of the cerium lakes, Cochineal, Persian berries, and Alizarin Yellow are very fugitive, whilst Alizarin, Alizarin Orange, and logwood are entirely altered. The fastest is the cerium Sandal-wood lake, which only becomes somewhat lighter, whilst the most fugitive are the iron lakes, with the exception of that from Persian berries.

| Dyed with | On Tin. | | On Cerium. | | On Iron. | |
|---|---|---|---|---|---|---|
| | Colour. | After Acid Treatment. | Colour. | After Acid Treatment. | Colour. | After Acid Treatment. |
| Logwood.. ... | Matt violet | Slightly redder | Reddish-black | Dark chamois | Bluish-black | Reddish-violet |
| Alizarin ...... | Orange red | Orange | Dark reddish-violet | Brownish matt yellow. | Very dark bluish-violet. | Dark chamois |
| Cochineal..... | Bluish-scarlet | Almost pink | Reddish-violet | Faint pink | Greyish-violet | Light pink |
| Alizarin Orange | Dark orange | Orange | Dark brownish-red | Pinkish-yellow | Bluish-red brown | Pinkish-red |
| Persian berries | Orange | Yellow | Yellowish-green | Almost colourless | Yellowish-brown | Yellow |
| Sandal-wood.. | Matt brownish-red | Somewhat redder | Reddish-brown | Lighter | Violet-brown | Dark pink |
| Alizarin Yellow | Reddish-chamois | Light chamois | Dark greenish-yellow. | Very light chamois | Grey | Chamois |

—T. A. L.

### *Wool Hat Dyeing.* C. A. Otto. Färber Zeit. 1898, 9, [1], 5—6.

The hat bodies must be boiled for half an hour with water to free them from adhering soap and soda, which would render the obtaining of a level bright colour a difficult matter. Then mordant with 3 per cent. of sodium chromate and 3 per cent. of tartar (or 3 per cent. of sodium chromate and 2 per cent. of "Redarin" in case of uncarbonised hats). Enter the goods at 70°, heat slowly to boiling point, and boil 1—1½ hours. After mordanting, the bodies are at once thrown into cold water, and are well stirred.

Dyeing takes place in a separate bath. The goods and dyestuff are entered at 30°—40° C., and turned for a quarter of an hour. The bath is raised slowly to boiling point, and boiled half an hour; the goods are then taken out. 1½ litres of acetic acid (45 per cent.) for every 1,000 litres of water are added, and boiling continued for from ½—1 hour. Alizarin colours are used, and are introduced into the dye-bath through a fine hair-sieve or thick cloth. Patent Blue, Fast Acid Violet, Orange II., Fast Yellow, &c., are used for shading.—J. E. B.

### *Suède Glove Leather, The Dyeing of.* Der Gerber, 1898, 24, [560] and [561].

Formerly the only colour known for suède leather was a shade of brown produced by dyeing with a decoction of oak bark, but because of the high percentage of tannin in this material, the leather was not satisfactory. At a later date the dyeing was done with aniline colours, but the brilliant shades produced did not meet the taste of the public, and the leather became unpopular, until it was found possible to use the dyewoods with alum as the mordant.

Alum appears, in fact, to be the only suitable mordant in the dyeing of suède leather, because (1) it is colourless, and does not interfere with the shade of the dye; (2) it can be used in any proportion according to the depth of shade required; (3) it unites with all colouring matters, thus giving an equality and evenness of shade, which is all important for this class of leather; (4) it penetrates the fibre of the skin as no other material does, making it possible to dye the leather an equal shade right through, and not, as with other leathers when dyed, having the colour only on the surface; (5) there is no known substitute (which makes it all the more valuable); other so-called mordants, such as urine, ammonia, potash, &c., would make of suède leather a harsh, horny substance, which could scarcely be called leather, and would be certainly unsuited for glove purposes.

It is best, with each addition of the dyestuff, to add a small quantity of a saturated alum solution as mordant. How much, must be ascertained by experience, acquired by observing the effects produced.—J. G. P.

*Aniline Black, Experiments on the Resist Action of Calcium Carbonate on.* R. J Flintoff and E. Ormerod. J. Soc. Dyers and Colorists, **14**, [1], 2—6.

The authors have determined the effects of certain lime salts as resists for Aniline Black (Ferrocyanide Black). They found calcium carbonate was a much better resist than calcium acetate or hydrate. They also assert that $CaCO_3$ gives better whites than the soluble salts of sodium now in use. This superiority they attribute to the insolubility of the lime salt, which prevents the Aniline Black mixture from saturating the cloth to the same extent in the parts where the resist has been printed, so that any tinting of tho cloth is more superficial and consequently more easily removed by soap.

Similar experiments made with other insoluble carbonates showed that their resist actions varied inversely as their atomic weights. (1.) $MgCO_3$. (2.) $CaCO_3$. (3.) $ZnCO_3$. (4.) $BaCO_3$.

The printing pastes in the experiments were made by thickening, in the form of pulp, the respective carbonates with British-gum solution, *e.g.*, 1 part $CaCO_3$ (30 per cent.) and 4 parts gum solution, gave good results.

The carbonates were prepared by mixing aqueous solutions of the corresponding chloride and carbonate of sodium. On mixing solutions of $CaCl_2$ and $Na_2CO_3$, a thick gelatinous precipitate was formed, and at the same time, bubbles of gas (found to be $CO_2$) were noticed forcing their way through the viscid mass. This gelatinous mass quickly changed to a white amorphous precipitate of $CaCO_3$. The gelatinous mass was so unstable that they were unable to isolate it. However, they believe it to be a basic carbonate corresponding to the formula $CaCO_3[Ca(HO)_2]_n$. The authors were unable to find any account of their observations in current chemical literature, and consequently performed their experiments with every possible care and frequent repetitious, so as to eliminate all uncertainties and any possible inaccuracies.

Similar changes took place in the preparation of the other carbonates enumerated above.—J. E. H.

*Aniline Black, An Interesting Formation of.* E. Knecht. J. Soc. Dyers and Colorists, **14**, [1], 9—10.

The author in this paper draws attention to the fact that in order to obtain an aniline black it is possible to have the oxidising agent in actual combination with the fibre. To this end he prepared trinitrocellulose, which, on suitable treatment with aniline salt, gave a black which would neither green nor rub. Although he does not pretend to advocate the use of such a dangerous material as trinitrocellulose for obtaining an aniline black, he thinks it might be possible to nitrate cotton slightly, and produce a good black.—J. E. H.

## PATENTS.

*Dyeing Yarns, Apparatus for.* J. W., J., and H. Moorhouse, Wakefield. Eng. Pat. 26,816, Nov. 26, 1896.

This apparatus is a machine by means of which earthenware, metal, or wooden bobbins are forced on to and taken off hanks of yarn for the purpose of keeping the dye from the covered parts in the dyeing of spotted or "random" yarns. The machine consists of a frame with a sliding beam, cut and shaped to hold the bobbins, which are forced by the depression of the beam by means of geared wheels on to the hanks, the hanks being suspended from the top of the frame.—R. B. B.

*Dyeing a Thread in Different Colours, New Process for.* A. Leven, Cologne, Germany. Eng. Pat. 27,409, Dec. 2, 1896.

The apparatus employed is in two parts: a flat plate upon which the material is placed, and a framework containing a number of partitions, but open at the top and bottom. When the frame is screwed down on the plate, several water-tight compartments are formed, the material acting as packing, and the material in each compartment may then be dyed a different shade.—R. B. B.

*Tapestry or Ornamented Fabrics, The Imitation by Impression in Fatty Inks upon Paper of.* E. Courbet, Paris, France. Eng. Pat. 27,552, Dec. 3, 1896.

By printing with fatty inks upon the variety of paper known as "velveted" or "flock" paper, very close imitations of tapestry, &c., may be produced. In order to print without detaching the fluffy layer of textile substance with which the paper is covered, the paper is treated, before printing, with a preparation consisting of white lead, linseed oil varnish, and a drying agent.—R. B. B.

*Dyeing Process, An Improved.* T. Ingham, Manchester. Eng. Pat. 1629, Jan. 21, 1897.

The process consists in passing the yarn or fabric through a dyebath of logwood extract, with or without the addition of fustic, &c., and after squeezing between rollers, passing into a cold concentrated solution of a mordant. On emerging from this bath, the goods are again squeezed, then steamed without pressure, and finally washed.—R. B. B.

*Coloured Discharge Patterns on Indigo-dyed Cotton Fabrics, Impts. in and connected with the Production of, in Calico Printing.* W. Watson, Manchester, and E. Bentz, Salford. Eng. Pat. 2620, Feb. 1, 1897.

Indigo-dyed cotton fabrics are impregnated with β-naphthol and then printed with a diazotised solution of *p*-nitraniline, *m*-nitraniline, *o*-nitro-*p*-phenetidine, or certain other aromatic amines, together with a suitable thickening agent and a soluble lead salt. After drying, the fabric is passed through a solution of sodium or potassium chromate, in order to convert the lead salt into lead chromate, and on subsequently passing into an acid bath, chromic acid is liberated, which discharges the indigo in the printed parts. If it be desired to remove all lead from the fabric, it is finally passed through hot dilute hydrochloric or hydrofluoric acid.—R. B. B.

*Dyeing Half-Wool with Basic Azo Dyestuffs in an Acid Bath, A Method for.* O. Imray, London. From "The Farbwerke vorm. Meister, Lucius, and Brüning," Hoechst a/M., Germany. Eng. Pat. 5119, Feb. 25, 1897.

This patent relates to the application of the colouring matters described in Eng. Pat. 27,896 of 1896 (this Journal, 1897, 1011). These dyestuffs are basic polyazo compounds having as components an aromatic ammonium base or amido-benzylamine or its derivatives; or basic azo compounds obtained by the action of diazosafranines upon phenols, naphthols, amines, or diamines. Cotton and wool union fabrics are dyed in a bath prepared with 2 per cent. of sulphuric acid and 5—20 per cent. of sodium sulphate. The addition of a neutral salt, *e.g.*, $Na_2SO_4$ or NaCl, increases the attraction for the wool fibre. The material is entered at 40°—50° C., the dyebath brought to the boil, and boiling continued for one hour.

The basic azo colouring matters may be employed in combination with acid dyestuffs in a bath acidified with an organic acid. The material is first dyed at the boil with the acid dyestuff only, the dyebath is then cooled, the basic azo colouring matter added, and dyeing continued at 70° C. In this way it is possible to obtain the so-called "cross-dyed" effects.—R. B. B.

*Fast Dyeings upon the Fibre, Producing, by Means of Direct Dyeing Colouring Matters.* S. Pitt, Sutton, Surrey. From L. Cassella and Co., Frankfort-on-Main, Germany. Eng. Pat. 5122, Feb. 25, 1897.

According to this invention, those direct cotton dyestuffs which contain free amido groups are combined with diazo bodies upon the fibre, the process thus being an inversion of the well-known method of diazotising direct-colouring matters on the fibre. The colours produced by the new method are stated to be absolutely fast to milling, and a considerable increase in the intensity of colour is the result of this developing process.

The process consists in dyeing with the direct colour in the usual manner and afterwards entering the goods into either an alkaline or an acidulated solution of the diazo body. Since the diazo bodies are very stable in acid solutions, the employment of an acidulated bath is a useful feature of the process.

The dyestuffs derived from *m*-phenylenediamine and analogous bodies are particularly suited for this method of dyeing, and the most useful developers are diazonitrobenzene, diazo-azobenzene, the diazo compound of dehydrothiotoluidine, &c.

The following table indicates some of the principal shades which may be produced in the manner described:—

| Dyestuff. | Developed with the Diazo Compound of | | | |
|---|---|---|---|---|
| | *p*-Nitraniline. | *m*-Nitraniline. | Amido-azobenzene. | Dehydrothio-toluidine. |
| Diamine Black R | Reddish-black | Violet-black | Black | Reddish-black |
| " " B | Black | Blackish-blue | " | Black |
| " " B H | Blue-black | Blue-black | " | Blue-black |
| Diamine Blue 3 B | Bluish-green | Bluish-green | Blackish-green | Greenish-blue |
| Diamine Blue-Black E | Black | Blackish blue | Black | Blackish-blue |
| Oxamine Violet | Blackish-violet | Violet-brown | Brownish-violet | Blackish-violet |
| Diamine Brown M | Dark brown | Dark brown | Dark brown | Brown |
| " " V | " " | Violet-brown | Violet-brown | Dark brown |
| " " B | " " | Brown | Dark brown | Brown |
| Diamine Cutch | Violet-black | Brownish-violet | Brownish-violet | Brownish-violet |
| Diazo Blue | Violet-brown | Brown | Violet-brown | Brown |
| Diamine Jet Black S S | Blackish-brown | Blackish-brown | Blackish-brown | Blackish-brown |
| " " R B | Dark brown | " " | " " | Dark brown |
| " " Cr | " " | " " | " " | " " |
| " " O O | " " | " " | " " | " " |
| Columbia Black R | Blackish-brown | " " | " " | Blackish-brown |
| " " 2 B | Brownish-black | Brownish-black | Brownish-black | Black |
| Direct Blue-Black | Blackish-brown | Blackish-brown | Blackish-brown | Blackish-brown |
| Diazo Brilliant Black | Dark brown | Brown | Dark brown | Brown |
| Benzo Chrome Black | Blue-black | Jet black | Black | Black |
| Oxydiamine Black S O O O | Brownish-black | Brownish-black | Brownish-black | Brownish-black |
| " " N | " " | Black | Black | " " |
| Diaminogene | Greenish-black | " | Jet black | Black |
| Cotton Brown N | Dark brown | Brown | Brown | Brown |
| Benzo Brown | " " | " | Yellowish-brown | " |
| Toluylene Brown G | " " | Yellowish-brown | Dark brown | Dark brown |
| Toluyene Orange | Chestnut-brown | Brown | Yellowish-brown | Brown |
| Primuline | Yellow | Greenish-yellow | Reddish-yellow | Golden-yellow |

—R. B. B.

*Dyeing Cotton, New Process for.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 5573, March 2, 1897.

This patent describes a method of applying dyestuffs, such as Cachou de Laval, Katigen Black-Brown, &c., which are produced by the action of sulphur or alkaline sulphides on different organic substances. The method consists in dyeing in very strongly alkaline baths in the cold, and the greatly increased depth of colour produced is ascribed to the greater attraction of the cotton for colouring matters, due to the "mercerising" action of the alkali. When the material is stretched during dyeing, the cotton also exhibits a silky lustre.

To illustrate the process, the following example is given: 50 parts by weight of Katigen Black-Brown are dissolved in a small quantity of hot water, and the resulting solution is added to a cold bath containing 500 parts of water and 150 parts of sodium hydroxide. 50 parts of the well-moistened cotton material are worked in this bath at the ordinary temperature for 6—10 hours, care being taken that the fabric remains covered by the liquid. The material is then wrung out, rinsed, and dried. It exhibits a deep black shade.—R. B. B.

*Fixation of Pigments or Metallic Powders in Printing on Textile Fabrics or similar Surfaces, Impts. relating to the.* W. E. Kay and The Thornliebank Co., Ltd., Glasgow. Eng. Pat. 6112, March 9, 1897.

The pigment or metallic powder is mixed with a suitable thickening containing dissolved gelatin, and this mixture is printed on the fabric, which is then subjected to the action of formaldehyde. The gelatin is thus rendered insoluble, and the pigment becomes permanently fixed. This improvement may also be employed in conjunction with the mercerising process, the insoluble gelatin acting in this case as a resist to the caustic alkali.—R. B. B.

*Dyeing certain Colours on Vegetable Fibres, Yarns, and Fabrics; Impts. in or in connection with.* F. A. Gatty and Co., Ltd., and V. H. Gatty, Preston. Eng. Pat. 7011, March 18, 1897.

Vegetable fibres which have been dyed by the fixation upon them of the oxides of iron and chromium are passed through a solution of a soluble silicate, and by this treatment, it is claimed, the colours are rendered much faster to the action of acids. For the fixing bath, a solution of silicate of soda at 2° Tw. is recommended, an immersion of 15 minutes in this bath at the boil, effecting the desired result.—R. B. B.

*Indigo White, Improved Method of Using, for Printing.* J. Grossmar, Manchester. Eng. Pat. 13,215, May 28, 1897.

The printing colour consists of a suitably thickened mixture of indigo-white, with an amount of caustic alkali largely in excess of the quantity theoretically required. Since this mixture is readily oxidised, a stable reducing agent is also added, and this may be glucose, zinc dust, aluminium dust, &c., but preferably a mixture of glucose and zinc dust.

The colour is printed in the ordinary way, and the printed pieces are rapidly and carefully dried just sufficiently to harden the thickening material. The pieces are then steamed for a few minutes, with exclusion of air, and passed into the ageing chamber, where they remain until the blue colour is developed.—R. B. B.

*Dyeing, Bleaching, and otherwise Treating Cops of Yarn: Impts. in Apparatus for.* W. Beaumont, Stockport. Eng. Pat. 27,690, Nov. 25, 1897.

The perforated tubes bearing the cops are attached to a perforated plate, which forms a false bottom to the bleaching or dyeing vessel. By means of a circulating pump, the liquor in the vessel is continuously drawn off from below this plate, and returned to the upper part of the vessel.

—R. B. B.

*Silk for Dyeing and Bleaching, Impts. in the Treatment of.* C. Spindler and C. Göhring, Spindlersfeld, Germany. Eng. Pat. 28,115, Nov. 29, 1897.

The process consists in treating any kind of silk, such as wild silk, waste silk, raw silk, or fibres made from these, &c., with the usual bleaching agents, such as hydrogen peroxide, but having in the bleaching bath sufficient alcohol, aldehyde, acetone, or similar bodies, so that the sericin and fibroin—the constituents of silk which make it valuable for technical purposes—are not destroyed. The proportion of 40 parts of alcohol to 60 parts of 3 per cent. hydrogen peroxide solution has been found to work well.—C. M.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Bauxite, Constitution and Formation of.* M. G. L. Bourgerel. Monit. Scient. 1898, 21.

Bauxite is found in the south of France, chiefly in the Var Department. It occurs in many varieties, grouped as red or white bauxite, according to the colour imparted by the proportion of ferric oxide present. The rock is compact and hard, and does not disintegrate on wetting, but the outcrop of the better qualities, exposed to frost, air, and sunshine, breaks up into small fragments in the form of oblique prisms with very variable angles. Such bauxites, when of the red variety, have a minimum of silica and a maximum of alumina, the white variety having also much alumina and very little iron. The fracture is conchoidal. Analyses give, for red bauxite, from 50 to 62 per cent. of alumina, from 24 to 28 per cent. of ferric oxide, from 1 to 7 per cent. of silica; and from 0·1 to 4 per cent. of titanic acid, with traces of vanadium, chromium, &c., besides from 12 to 13 per cent. of water. The white varieties contain from 65 to 74 per cent. of alumina, from 0·25 to 3 per cent. of ferric oxide, and from 12 to 18 per cent. of silica, with about 14 per cent. of water.

Bauxite is found between calcareous deposits in a sedimentary area, and reasons are given for assigning its origin to the precipitation of gelatinous alumina, together with silica and ferric hydrate. The greater part of the alumina in bauxite is monohydrated, whereas artificially precipitated alumina is at least trihydrated. Gelatinous trihydrated alumina heated to 350° C. is transformed into the hydrate present in bauxite. From such and other considerations it is conjectured that this mineral has been formed in a vast, highly heated basin, in which water charged with aluminium and ferric chlorides came into contact with decarbonated limestone, thus giving the precipitates that ultimately solidified as bauxite, while the accompanying lime was subsequently washed out by pluvial infiltration conveying carbonic acid.—E. S.

*Liquid Ammonia, Methods of Analysis of.* A. Lange and W. Heffter. Chem. Ind. 1898, **21**, [1], 2—7.

*See under* XXIII., *page* 183.

*Stannic Salts, Estimation of Tin in.* A. Fraenkel and J. Fasal. Mitt. des k. k. tech. Gewerbe-Museums in Wien, 1897, **7**, [9, 10, and 11], 227.

*See under* XXIII., *page* 185.

### PATENTS.

*Ammonia, A Method of and Apparatus for the Continuous Manufacture of, from Nitrogenous Coke.* H. H. Lake, London. From C. Pieper, Berlin, Germany. Eng. Pat. 29,455, Dec. 22, 1896.

Heated coke is brought into contact with steam so long as may be necessary to remove the nitrogen as ammonia, together with the other gaseous products produced. In one form of apparatus, the coke is continuously lifted by a conveyor screw, working in a vertical tube, to near one end of a long, narrow chamber, so that the coke, on entering the chamber, slides down an inclined bridge into the hopper-shaped and deep continuation of the chamber, from the bottom of which it is removed by a horizontal conveyor into an air-tight receptacle. Steam meanwhile is admitted through a nozzle, so as to travel in the same direction with the coke. The gaseous products issue at the opposite end of the apparatus, and are taken for absorption of the ammonia to suitably charged receivers. The heating apparatus described, is located in a furnace, in which its walls are suitably heated externally, the greater heat being applied to the fore part, where the coke and steam first come into contact. A modified apparatus is shown in which the feed chute is arranged to introduce the raw material from the top on to an inclined bridge, a steam nozzle being provided opposite the bridge, so arranged that the steam passes in an opposite direction to that taken by the coke.—E. S.

*Sulphuric Acid or other Acids or Liquors, Impts. in Apparatus for the Concentration or Rectification of.* J. E. Campbell, Manchester. Eng. Pat. 3592, Feb. 10, 1897.

A concentrating apparatus constructed with double pots, is claimed, one of which is placed to rest upon a sand bath inside the other. There is thus an outer pot, in which the concentrating vessel rests, constructed with a pipe or nozzle leading to the interior of the furnace, for recovering any liquor that may be spilled, and preventing the same from running into the flues. Finally, the combination of the metal pipes placed to support the pots, with the brickwork, &c., is claimed.—E. S.

*Detergent Fluids, Impts. in the Manufacture of.* R. Stewart, Glasgow. Eng. Pat. 4145, Feb. 16, 1897.

Finely divided slaked lime and sodium carbonate in about equal quantities (say one pound of each) are boiled with water (say five quarts) until clear. The supernatant liquid is drawn off, filtered if necessary, and mixed in equal or variable proportions with a fluid obtained by mixing alum and pearl-ash, approximately in the proportion of an ounce of the former to a pound of the latter, keeping in a closed vessel until liquefied, and then straining. The mixture is boiled until clear, and when cool is ready for use. For some purposes borax is added. About a teacupful of the detergent is added to the water in the copper at a temperature slightly below the boiling point, and the clothes are then boiled for about 30 minutes, which is said to cleanse them without the necessity of rubbing. The preparation, as described, and use of this detergent fluid are claimed.
—C. A. M.

*Nitrites, Manufacture of.* A. Knop, Rheinau, Germany. Eng. Pat. 4747, Feb. 22, 1897.

Finely divided coke is stirred into fused caustic soda, and the mixture is poured out and allowed to cool. Sodium nitrate is then melted with fresh caustic soda, and the first mixture of coke and soda is added by small pieces to the latter mixture, with stirring. The reaction is expressed by the equation—

$$2NaNO_3 + 2NaOH + C = 2NaNO_2 + Na_2CO_3 + H_2O,$$

but the proportions prescribed for practice show a considerable excess of soda over that involved in the equation, so that the melt consists of sodium carbonate, caustic soda, and nitrite, "and the last-named, it is added, can easily be obtained pure." The caustic soda is recovered for re-use by causticising the sodium carbonate.—E. S.

*Washing and Cleansing Compound, Improved.* F. L. Bartelt, Keynsham, Somerset. Eng. Pat. 4830, Feb. 23, 1897.

An ammonium salt, preferably the sulphate, is combined with an anhydrous alkali, preferably sodium carbonate or compounds containing sodium carbonate, in order that the latter may "crystallise" the water contained in the former, and that the compound, on being dissolved in water, may liberate nascent ammonia, which is claimed to have a superior cleansing power. If required, soap or fatty matter may be added to the compound.—C. A. M.

## VIII.—GLASS, POTTERY, ENAMELS.

### *Porcelain Body, Casting, with Carbonate of Soda.* Sprechsaal, **30**, [41], 1279, [45], 1311.

In preparing the slip with sodium carbonate for casting, the action of the latter ingredient on the thick mass must not be under-estimated, since otherwise the slip will turn out to be too thin and practically spoilt. Whilst the actual optimum amounts of sodium carbonate and water can only be determined by experiment, the rule that only a minimum of each should be used, is generally applicable, deviation from this leading to unsatisfactory results.

To prepare the moulds, the simplest plan is to model the inside of the article, make a gypsum casting of this, then mould the ware in modelling clay on the latter, and make a gypsum casting of the outside, fitting this casting with a wide aperture for pouring in the slip-body, and, in the case of large pieces, with ventilating holes as well.

A frequent defect in pieces cast from slip-body is greyness of colour, due to an excess of sodium carbonate, which, by increasing the fusibility of the body, causes it to readily become "over-fired" in the glazing-kiln; and as the use of more fusible glazes is inadvisable, these being more liable to form bubbles than the harder kinds, the soda must be reduced in quantity. However, if this procedure would prejudice the fluidity of the slip, the same end can be attained by increasing the proportion of quartz and kaolin, or by reducing the amount of felspar in the body.

Another inconvenience arising from an excess of sodium carbonate is the occurrence of roughness at the edges and projecting points of the ware. This is due to an efflorescence of sodium carbonate during the drying of the cast pieces, and to the subsequent fusion of the incrustation, whereby the absorptive capacity of the ware for the true glaze is reduced and the surface remains rough. Should a diminution in the amount of added sodium carbonate fail to remedy the defect, then the incrustation must be removed by cleaning, by well-known methods, the surface of the ware whilst still in the state of grey-dryness.

Another means of preventing incrustation on the edges of slip-body castings is by retarding their rapid drying, which is the direct cause of the efflorescence. This may be accomplished by applying a thin coating of glycerin or olive oil to the parts in question.—C. S.

### *Silvering Mirrors.* F. Fafet. Rev. de Chim. Ind. 1898, **9**, 5—8.

Great developments have occurred in the manufacture of mirrors since the substitution of the silvering process for the old method of applying amalgamated tinfoil to the surface of glass. Drayton's process for silvering glass (1844) consisted in using an alcoholic ammoniacal solution of silver nitrate to which essence of cassia was added, with a very small proportion of an alcoholic solution of essence of cloves as the reducing agent. Many other processes have since been commercially applied, the one now most favoured being that involving the use of tartrates. Each worker seems to have his own recipe for silvering.

Tartrates used with silver salts give a more brilliant coating of the metal on glass than any other reducing agent, not excepting formaldehyde. The simplest method of preparing the silvering liquid consists in mixing an ammoniacal solution of silver nitrate with Rochelle salt, this liquid depositing slowly in the cold, but quickly when heated, a bright film of silver on the surfaces presented to it.

M. Namias (Industria, Nov. 1897) has, after much investigation, arrived at the following formula as expressing the most favourable reaction.—

$$4AgNO_3 + 4NH_3 + C_4H_4(KNa)O_6 + H_2O = 2Ag_2 + 4NH_4NO_3 + C_3H_2(KNa)O_5 + CO_2,$$

in which the proportions are, silver nitrate, 680 parts, and of the double tartrate, 282 parts; but it is preferred to use the latter in slight excess. It will be observed that in the reaction the double tartrate is converted into the corresponding double tartronate. If the proportions here given are not approximately taken, the silver is irregularly precipitated, and is not bright. The best results are obtained by dividing the silver nitrate solution between the solution of the Rochelle salt and that which is taken for addition of ammonia to it; the tartrate solution is formed by warming a mixture of the tartrate and silver nitrate, and filtering after the liquid blackens; the ammoniacal silver solution is formed by addition of ammonia to the reserved portion of the silver nitrate solution until the precipitate at first formed is redissolved. The mixture of the two solutions gives a liquid from which the silver is deposited easily and uniformly.

The facility of the deposition of silver on a glass surface, and the character of the deposit, depend much on the manner of preparing the glass, which, if washed with a solution of stannous chloride and then rinsed with water, receives a silver deposit very rapidly. A 0·001 per cent. tin solution, when thus used in the preparation of a glass surface, renders the precipitation of silver upon it from three to five times more rapid than with an unprepared surface. It is suggested that the contact of the tin solution with the glass occasions the formation of a tin silicate, which resists the subsequent washing, and initiates the reduction of the silver nitrate, minute as is the quantity.

Such thin layers of silver as are deposited on glass, not exceeding $\frac{1}{500}$ of a millimetre in thickness, are in a molecular state to be acted upon by many substances to which compact silver is indifferent, resembling in this respect the silver deposited in photographic processes. Tartaric and citric acids, for instance, react energetically on such films. The nature of the varnish used to protect the silver deposit becomes therefore a matter of great importance. It is stated that there is no varnish which does not, in time, attack the silver film. Bitumen acts energetically; linseed and cotton-seed oils and colophony act less quickly; gum-lac and mastic have a feeble action; sandarach resin and wax act only after a long while; fatty matters and acids also act after a time. A mixture of resin with an insoluble base, such as zinc oxide or magnesia, is recommended as a varnish; and a very little ammonia added to an alcoholic solution of gum-lac has a favourable effect. Alcoholic varnishes are first used, followed by the application of zinc white mixed with benzine or water.—E. S.

### PATENT.

*Glass Tubes, Impts in the Manufacture of.* W. Butterworth, Newton Heath, Manchester. Eng. Pat. 22,511, Oct. 1, 1897.

Tubes for boiler gauges, thermometers, barometers, &c., are made capable of resisting sudden changes of temperature or pressure, by blowing them in three layers—an innermost layer of "lead casing" glass, and a middle and an outermost layer of "lime glass," the middle layer being preferably made so as to be intermediate in expansibility between the other two.—J. T. D.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### *Building Operations during Frost.* Dingler's Polyt. J. 1897, **306**, 264.

Experiments in Vienna are said to have shown that only such mortars as are prepared with Portland cement can resist the frost; but if quicklime be employed in the mortar mills and be thus mixed hot with the sand, building operations may be continued even with the thermometer several degrees below freezing-point. In Norway, this practice has been common during the winter for many years. At night, when the temperature falls many degrees below zero, the fresh masonry is protected by cardboard or straw.

—W. G. M.

### *Hydraulic Cements, Testing of.* F. M. Meyer. Thonind. Zeit. **21**, [103], 1091—1092.

Referring to the labours of the French Commission on Uniform Methods for Testing Portland Cement, the author considers that for technical purposes the tests should be divided into two groups—one relating to the properties of the cement in general, and the other to the special purpose to which the material is to be applied. This second class of tests should approximate to the conditions prevailing in practice, and cannot be restricted by hard-and-fast rules. For the general tests he prescribes the following determinations as sufficient :—(1.) Density and loss on calcination; (2.) Constancy of volume; (3.) Fineness of grinding; (4.) Alkalinity of the aqueous solution; (5.) Time of setting; (6.) Shearing strain of the concrete, since the purity of the cement, the intimacy of the mixture of clay and lime, the uniformity of texture and sufficiency of burning, as well as the strength and durability of the material, depend thereon.

The specific gravity of the fresh cement, heated to 800° C., should be at least 3·08 per cent., and the loss on calcination ought not to exceed 4 per cent. Chemical analysis is regarded as an insufficient determination of quality in the present state of our knowledge of the optimum ratio of the cement constituents. In testing fineness, the absence of residue when a 360-mesh sieve is used, or the presence of not more than 4—5 per cent. of residue on a 900-mesh sieve, is considered sufficient proof, the indications given by finer sieves (5,000-mesh) being characterised as illusory.

In testing the setting properties, the estimation of the alteration of temperature during setting is looked upon as important. Finally, the shearing strain of the concrete is regarded as preferable to tensile and compression tests.
—C. S.

## PATENTS.

*Cement, Impts. in the Manufacture of.* Société Métallurgique de Champigneulles et Neuves-Maisons. Eng. Pat. 1881, Jan. 23, 1897.

Slags containing more than 1·5 per cent. of oxide of iron, granulated as they leave the furnace, are mixed with lime to make a mortar. This, after setting, is broken up, and the pieces baked at a temperature between a dark-red and a bright-red heat; the product, finely ground, affords a cement which sets rapidly, and has a high resistance. (See also Eng. Pat. 1795, Jan. 22, 1897; this Journal, 1897, 1017.)—J. T. D.

*Building Material, Light Fireproof, possessed of the Quality not to be Eaten by Insects, and therefore specially adapted to Hot Climates; A new Way of Constructing.* C. W. Luther, Reval, Russia. Eng. Pat. 21,921, Sept. 24, 1897.

Three thicknesses of veneer are glued together crosswise with a cement composed of 60 to 85 per cent. of casein or similar substance, 40 to 15 per cent. of lime, and a suitable quantity of water. The boards so produced may be coated on one or both sides with a paint made of the dry materials of the above cement with 5 to 30 per cent. of coal tar; they are then fastened to wooden or iron posts or rafters so as to construct a building.—J. T. D.

*Gypsum and similar Hydrous Rock, Improved Method of Hardening or Preserving.* A. A. Dickson, Toronto. Eng. Pat. 22,741, Oct. 4, 1897.

The shaped material (held in clamps, if necessary) is dehydrated by dry air in a hot chamber, cooled gradually, immersed in a bath of ammonia alum till the pores are completely filled, and then air-dried. The clamps (if any) are removed, and the articles polished. The material is greatly hardened by the process, and little liable to crack or blemish during the polishing operation.—J. T. D.

## X.—METALLURGY.

*Steel Wire, Hardening of, in Molten Lead.* Dingler's Polyt. J. 1897, **306**, 264.

The wire is introduced into a lead bath at a full cherry-red heat, but not at a higher temperature, for at the boiling point of lead, the steel would be overheated. The wire should be thinly coated with chalk before heating, by dipping it into a paste of whiting and water, and then drying it. This protects the metal from any oxide of lead in the lead bath, and prevents the formation of hydrogen bubbles (and hence unequal hardening) when the hot wire is plunged into water. In water-hardening, the chalk detaches itself, and after oil-hardening it may be easily removed by rubbing. The process is recommended especially for the hardening of wire uniformly, and without oxidation during heating, and is especially suitable for the treatment of steel wire direct from the winding drum, as the wire may be mechanically and continuously unwound, passed through the hot lead bath, and quenched.—W. G. M.

*Nickel Production.* Dingler's Polyt. J. 1897, **306**, 264. (From the Mineral Industries.)

The firm Gustav Menne and Co., of Siegen, produce electrolytic nickel harder than the American product, and containing a trace of lead. The smelting process is held by German metallurgists to be in no danger from the competition of the electrolytic method for the treatment of ores free from copper, inasmuch as the smelted metal may contain 99·2—99·5 per cent. Ni, which result is as good as any attainable by the latter method. But the electrolytic process may be applied with advantage to Canadian ores. Electro-deposited plates, ⅜ in. in thickness, are produced by the Balbach Smelting and Refining Co. in Newark (N.J.). They are so tough and elastic that they are with difficulty cut with a chisel, whilst they do not split or crack under the hammer or punch. In producing market nickel, great loss is caused by the anodes breaking up and thus producing 50 per cent. of waste, which then has to be re-melted. The difficulties are, however, increased by the use of matte anodes. At the Balbach Works, the Orford anodes contain (per cent.) 95 Ni, 0·55 Cu, 0·75 Fe, 0·25 Si, 0·45 C, and 3 S, and from them are obtained daily about 1,000 lb. of nickel, containing less than 0·03 per cent. of As, and 0·02 per cent. of S.
—W. G. M.

*Aluminium and its Alloys, Impurities in.* Ed. Defacqz. Comptes Rend. 1897, **125**, [26], 1174—1177.

Silicon, iron, and copper are still found in commercial aluminium; the author has endeavoured to find in what forms they exist in it. He attacked aluminium obtained by electrolysis with dilute hydrochloric acid, and analysed the solution and the chocolate-brown insoluble residue. He attacked an alloy of 3 parts of aluminium, and 100 parts of copper with aqua regia, and another portion with dilute hydrochloric acid, and in both cases analysed the solution and the residue. The residues from the aluminium, and from the action of aqua regia on the alloy, had the properties of impure silicon. The residue from the action of dilute hydrochloric acid on the alloy was more complex, and contained copper, silicon, iron, and aluminium. The solutions all contained silica, probably resulting from the decomposition of silicides of iron, copper, and perhaps aluminium. The impurities were distributed indifferently in the residue and the solution, and therefore it would be useless to attack aluminium with dilute acids in order to effect their separation.—J. A. B.

*Glucinum, Preparation of Alloys of. Alloys of Glucinum and Copper.* P. Lebeau. Comptes Rend. 1897, **125**, [26], 1172—1174.

The high temperature necessary for the reduction of glucina, its volatility, and the ease with which the carbide is formed, have prevented the preparation of the metal. (Compare Comptes Rend. **121**, 496, and **123**, 818; this Journal, 1896, 140, and 1897, 72.) In attempting the reduction of the oxide, several alloys have been prepared. An intimate mixture of carbon with the oxides of glucinum and copper, best prepared by calcination of the nitrates, heated for five minutes in the electric furnace, gives a rosy red non-homogeneous ingot, from which a true alloy of a yellow colour is separated by fusion, and containing from 5 to 10 per cent. of glucinum. The 5 per cent. alloy is malleable, readily filed and polished, unaffected by air, but slightly tarnished by sulphuretted hydrogen. Fusion of this alloy with copper gives alloys less rich in glucinum, which are very sonorous. Alloys of glucinum with other metals have been similarly prepared, and are being studied further.—J. A. B.

### *Gold, The Action of Cyanogen on.* J. Park. Trans. Inst. Mining and Metallurgy, Jan. 19, 1898.

The experiments described in the paper were undertaken by the author to ascertain whether cyanogen in aqueous solution is a solvent for gold, in the commercial sense.

Discs of parted gold were exposed to the action of the solution, with the following results:—

*Series A.—Gold Disc $\frac{1}{8}$ inch diameter; weight, 0·340 grain.*

| No. | Time. | Hours. | Weight of Gold. | Gold dissolved. |
|---|---|---|---|---|
| | | | Grain. | Grain. |
| 0 | 4 p.m. | .. | 0·340 | .. |
| 1 | 10 a.m. | 18 | 0·338 | 0·002 |
| 2 | 4 p.m. | 24 | 0·337 | 0·001 |
| 3 | 10 a.m. | 42 | 0·335 | 0·002 |
| 4 | 4 p.m. | 48 | 0·334 | 0·001 |
| 5 | 10 a.m. | 66 | 0·332 | 0·002 |
| 6 | 4 p.m. | 72 | 0·330 | 0·002 |
| 7 | 10 a.m. | 90 | 0·328 | 0·002 |
| 8 | 4 p.m. | 96 | 0·328 | 0·000 |
| 9 | 10 a.m. | 114 | 0·327 | 0·001 |

Total gold dissolved, 0·013 grain = 3·8 per cent.

*Series B.—Gold Disc $\frac{3}{32}$ inch diameter; weight, 0·150 grain.*

| No. | Time. | Hours. | Weight of Gold. | Gold dissolved. |
|---|---|---|---|---|
| | | | Grain. | Grain. |
| 0 | 10 a.m. | .. | 0·150 | .. |
| 1 | 4 p.m. | 6 | 0·149 | 0·001 |
| 2 | 10 a.m. | 24 | 0·148 | 0·001 |
| 3 | 4 p.m. | 30 | 0·147 | 0·001 |
| 4 | 10 a.m. | 48 | 0·145 | 0·002 |
| 5 | 4 p.m. | 54 | 0·144 | 0·001 |
| 6 | 10 a.m. | 72 | 0·142 | 0·002 |

Total gold dissolved, 0·008 grain = 5·3 per cent.

The gold discs were placed in fresh portions of cyanogen solution after each weighing, except in the case of Nos. 7 and 8, Series A, which were returned to the same solution.

When the discs were returned to the same solution, no further solution of the gold could be detected.

The cyanogen was made by distilling a mixture of 2 parts of yellow prussiate of potash and 3 parts of mercuric chloride in a glass retort.

The salts were powdered, perfectly dried, and placed in a dry, warm retort. The gas was passed through a small flask of distilled water, and collected in a Winchester bottle containing distilled water, the first portion of the gas being rejected. The water became saturated after passing the gas for about half an hour.

It is essential that the salts used for the generation of the gas be perfectly dried, as in the presence of water the following reactions take place:—

$$2K_4FeCy_6 + 2H_2O + 5HgCl_2 =$$
$$8KCl + FeCl_2 + FeO + NH_4CN + 5Hg + 5Cy_2 + CO.$$

The presence of $NH_4CN$ will cause solution of the gold.

The solution of considerable quantities of gold by the action of an aqueous solution of cyanogen on that metal having been reported by some experimenters, the author considers that such results are probably due to the use of undried salts for the preparation of the gas: such, in fact, was proved to be the case in one instance.

The author then carried out experiments to ascertain the behaviour of cyanogen in aqueous solution towards gold as existing in ores. The ore selected was from the Martha lode at Waihi. It consisted of quartz, was free from sulphides, and contained the gold so finely divided that from 90 to 95 per cent. of its value was extracted by a 0·3 per cent. KCN solution at one operation. It was therefore a typical ore for treatment by the cyanide process. Its assay value was 1*l.* 5*s.* 6*d.* per ton, and the ratio of gold to silver 1 to 3.

From this ore an aqueous solution of free cyanogen extracted, after 72 hours' leaching, only 0·82 per cent. of the gold and 1·13 per cent. of the silver present in the ore—results which were subsequently confirmed by two different series of experiments on the same ore made by the author.

A sample of the Waihi ore, similar to that already described, was treated by the author with an aqueous solution of cyanogen prepared from the undried salts. The extraction obtained was, after 72 hours' leaching, 48·8 per cent. of the gold and 11·4 per cent. of the silver present in the ore.

The author has conducted a number of tests to ascertain, if possible, what action is responsible for the feeble solvent action of the aqueous solution of cyanogen upon gold; he states that his results tend to show that the cyanogen acts superficially on gold, forming a film of cyanide, insoluble in aqueous solution of cyanogen, but readily dissolved by a trace of ammonium cyanide, which may be formed by contact with the atmosphere, or during the preparation of the gas. He has found it almost impossible to make cyanogen perfectly free from $NH_3$, HCN, HCl, and even $NH_4CN$. He has noted that old solutions of cyanogen, showing signs of decomposition by the deposit of azulmic acid, have a greater solvent effect upon gold than freshly-made solutions.

The experiments described show that cyanogen in aqueous solution does not dissolve gold, or only so feebly as to be ineffective as a solvent for extraction purposes.

—R. A. P. D.

### *Gold Extraction by the Direct Filling Process [Cyanide], Treatment of Tailings on the Witwatersrand.* F. C. Pengilly. Inst. of Mining and Metallurgy. January, 1898.

The above process is now successfully worked at two mines in the district. The tailings from the battery plates are lifted by a tailings wheel into a launder, by which they are conveyed to the slime separators, of which there are two. The slimes from the first separator pass into the second, thus saving fine sand. Fifteen per cent. of the slime is thereby eliminated, and 10 per cent. more is washed out when running the remaining pulp into the treatment vats, thus producing tailings fairly free from slimes. These tailings are then drained, treated with an alkali solution to neutralise the free acid from pyritic ore, and subjected to 0·6 lb. of cyanide per ton of tailings in solutions of different strengths, this treatment lasting over a period of six days. The gold is precipitated by zinc, preferably as zinc-lead couples containing 1 to 2 per cent. of lead, to overcome the action of foreign metals in retarding the precipitation of the gold, which couples are made by dipping the shavings into weak acetate of lead solution. The precipitate, containing 4 per cent. of gold when dry, is agitated with a 10 per cent. solution of sulphuric acid, which dissolves the zinc and increases the gold contents to from 33 to 45 per cent. The bullion obtained yields an average fineness of 847 gold and 60 silver. The chief advantages of the direct filling process are the smaller plant necessary and the saving in working cost.—A. W.

### *Gold Extraction by Cyanide from Minerva Tailings, Black Reef, Witwatersrand Gold Fields.* W. F. Wilkinson. Inst. Mining and Metall., 10—13.

The ore contains much copper, and this necessitates the use of stronger cyanide solutions than those used on the Main Reef. With an 11 dwt. 2 gr. sample a 0·3 per cent. solution gave in six days an extraction of only 9 dwt. 8 gr., whilst a 0·5 per cent. solution extracted 10 dwt. 5 gr. It is true that the consumption of cyanide amounted to 2·75 and 3·8 lb. per ton respectively, but the increased yield of gold more than compensated for the greater loss of solvent. Fresh ore gives no trouble owing to acidity, but material that has stood for any length of time may contain so much free sulphuric acid and iron sulphate (from the pyrites present), that a special plant for washing and neutralisation prior to extraction would be required. In precipitating, the copper first deposits on the zinc, and then the gold on the copper, but the latter more slowly than it would upon fresh zinc.—W. G. M.

*Gold Fields, of the Hauraki District, New Zealand.* J. A. Wanchope. Trans. Mining. Inst. of Scotland, 1897, **19,** 19—45.

The Hauraki district consists of a long area (100 miles in length) of mountainous country in the North Island of New Zealand. The peninsula is situated between Cape Colville at the north, and the Waihi river at the south; to the east there is a steep descent to the Pacific Coast, and on the west lies the Waihou or Thames river, to within a few miles of Grahamstown. The highest point in the range is Te Aroha (3126 ft.), the whole area is more or less auriferous, but the main gold belt varies in width from 12 to 20 miles. Gold has long been worked, and sensational results have been obtained in the past. The whole region has suffered great geological disturbances; and for the most part a series of eruptive rocks is presented. The auriferous deposits in general, appear to resemble the gold-bearing veins of California, but they are rather ore-bodies, in which gold has been deposited by percolating solutions, than veins. The reefs for the most part contain gold, pyrites, galena, blende, ruby-silver, stibnite, manganese oxide, pearl spar and calcite, and have been proved to exist at a depth of 1,000 ft. below the surface. In the past it would seem that the miners abandoned workings whenever a rich shoot of gold ran out, or a fault was encountered, or when water was struck in a winze. In many cases the extraction efficiency has been of the worst; for example, in the Waihi mine, the battery and berdan process was used for 12 years before the introduction of cyaniding, and in this way out of an assay value of 3*l.* 12*s.* per ton, only 12*s.* of gold was extracted, gold to the value of 3*l.* being run into the Ohinemuri river. These tailings were treated in 1894, and bullion to the value of 25,000*l.* was obtained within a few months from 20,000 tons of the material at a total cost of 10,000*l.* Accounts are given of the different sub-districts in the Hauraki region, but it may suffice to say that the gold obtained in 1895 from the sections enumerated was as follows:—

| | £ | *s.* | *d.* |
|---|---|---|---|
| Upper Thames.................... | 198,925 | 4 | 7 |
| Lower Thames.................... | 31,663 | 6 | 1 |
| Coromandel...................... | 87,391 | 18 | 2 |
| Kuaotunu........................ | 14,685 | 17 | 4 |

The estimated yield for the past year is 500,000*l.* Besides the product of inland mining, there are millions of tons of black sands on the beach, which have so far resisted all efforts to recover the gold contained in them. The cyanide process is not available because the solvent is attacked by the iron compound of the sand: but the author proposes to roast and concentrate the material, and so to render it suitable for treatment. Dredging is successfully applied even at depths of 50 ft., and there are a few localities in which placer mining might be profitably applied. The cyanide process obtained a foothold in 1894, and since then has been adopted by 15 or 16 companies. Experimental tests at the Thames School of Mines have shown that 25 parcels of ore subjected to cyaniding gave an extraction of 84 per cent., and 20 to the Washoe process gave an extraction of 66·5 per cent., whilst 2 parcels were wet-crushed and passed over amalgamated copper-plates and gave an extraction of only 51 per cent.

In looking to the development of the mineral resources of New Zealand, there are 9 points which claim special attention and by which the contents of this paper may be summarised: (1.) The Hauraki district from its position and from the contiguity of salt water is pre-eminent as regards facilities for transport of machinery, &c. (2.) The district from its geological formation gives promise of such development in the near future that the golden harvest of the past will be comparatively insignificant. (3.) The country being mountainous, the mines can be worked level-free by adits, thus saving the cost of pumping machinery and winding-gear; so that there is no gold field in the world possessing greater facilities for economic test and development. (4.) There is abundant water for motive power and an unlimited supply of timber—sawn timber costing from 6*s.* to 8*s.* per 100 ft. (5.) Freights are low and labour cheap. (6.) No topographical or climatic difficulties present themselves. (7.) With the cyanide and other modern processes even the low grade rock may be worked at great profit. (8.) The reefs are numerous and frequently of great size and richness. (9.) The mines cannot profitably be worked by individual effort or small local companies, and their development will depend largely on the influx of outside capital.—W. G. M.

*Silver from Gold, Separation of, by Netto's Method.* Witter and Zuschlag. Berg- u. Hütteum. Zeit. 1897, **56,** 405.

The authors have extracted the metals from a number of gold- and silver-bearing ores with potassium cyanide, and treated the solutions by the Göpner-Diehl process. They found that the addition of cuprous salt was not necessary to cause the precipitation of the gold on acidifying, and that either sulphuric or hydrochloric acid might be used. With stronger cyanide solutions, acidified with sulphuric acid, 2 to 4 grms. of gold per c.c. remained in solution, according to temperature; acidifying with hydrochloric acid, at most 1·0 to 1·5 grm. so remained. This is caused by the formation of auricyanides, which, however, are broken up at 50° C., when the gold is completely precipitated. Netto's statement that on acidifying a gold-silver solution in potassium cyanide with hydrochloric acid, the silver alone falls and all the gold remains in solution, is not in accordance with the facts.—J. T. D.

*Tinfoil, Determination of Lead and Antimony in.* A. Seyda. Zeits. für öffentl. Chem. **3,** 364; Analyst, 1897, **22,** [261], 331.

*See under* XXIII., *page* 184.

*Phosphorus, Rapid Estimation of* [*in Iron*]. H. Wdowiszewski. Stahl u. Eisen, 1897, 814; Chem. Centr. 1897, **2,** [21], 1038.

*See under* XXIII., *page* 184.

*Lead, Fire Assay for.* J. F. Cannon. Eng. and Mining J. 1897, **64,** [21], 604.

*See under* XXIII., *page* 184.

*Sodium Thiosulphate, Action on Sulphates, Nitrates, and Metals.* J. Girard. Rev. Chim. Ind. 1887 **8,** 361.

*See under* XXIV., *page* 189.

## PATENTS.

*Ores, Refractory, containing Gold, Silver, Nickel, and the like, Improved Method and Apparatus for Treating.* J. E. Preston, London. Eng. Pat. 6051, March 8, 1897.

In the base of a low wide blast-furnace is a narrow central vertical flue with perforations. Within this flue is a pipe through which a part of the fan-blast used for the tuyères is diverted. The ore is fed into the blast-pipe from a hopper and is thus swept by the current of air into the furnace through the central tube, and then, after becoming heated to incandescence, it impinges upon an inverted cone. Metallised products are thus to be deflected back into the furnace, whilst the remaining dust is carried on and deposited beyond. A tap-hole is provided to run off the metallic portion after fusion.—W. G. M.

*Precious Metals from Minerals, containing them, Impts. in Extracting, and Apparatus therefor.* B. Becker, Eupen, Germany. Eng. Pat. 7218, Mar. 19, 1897.

The apparatus consists of a conical dissolving vat which may or may not be lined with amalgamating plates for the recovery of a portion of the precious metal, and a system of pipes and a rotary pump by means of which the solvent fluid is circulated through the vat. This apparatus may be used alone or in conjunction with an electrolytic vat through which the fluid containing the metal in solution is circulated by means of the same pump, and returned to the vat to dissolve a fresh portion of the ore.—G. H. R.

*Gold, Wet Process for Extracting, from Gold Ores or Gold Ore Waste.* J. Landin, Stockholm, Sweden. Eng. Pat. 18,640, Jan. 11, 1897. (Under Internat. Convent.)

The gold is extracted by the chloride or bromide of a metal which is capable of forming a higher chloride or bromide with chlorine or bromine. Thus the ore is leached with a chloride of lead solution containing also chloride of lime and free acid. The chlorine is said to act on the lead salt forming a super-chloride of lead which then attacks the gold, forming the normal chloride of lead again and chloride of gold solution.—A. W.

*Gold, Silver, or Copper; Impts. in the Treatment of Ores or other Matters containing, for the Obtainment of the Metal therefrom.* A. Etard, Paris. Eng. Pat. 880, Jan. 12, 1897.

The claims are for the extraction of gold, silver, or copper from ores by means of a weak and cold solution of hydrochloric acid, in conjunction with an alkali manganate, permanganate, or a chlorate, perchlorate, chromate or bichromate.—A. W.

*Gold, Impts. in Extracting, from Ores.* C. Wiehmann, London. Eng. Pat. 1778, Jan. 22, 1897.

The ore is crushed, dried, crushed again, and separated by sieves into three grades—coarse, middle, and dust. The first grade is roasted and extracted by amalgamation, the second is treated with cyanide solution in vats, and the third is extracted with a solvent in a vessel conveniently arranged for the agitation of the pulp.—A. W.

*Gold, Impts. in Apparatus for Extracting, from Ores.* R. Hodgson, Shirleywich. Eng. Pat. 1508, Jan. 19, 1897.

The ore is washed down inclined troughs, with longitudinal cups made of half tubes placed at short intervals right across the bed. The cups contain mercury and the ore is washed in and out of them on its way down the trough. It is discharged into an elevator which raises it to the upper end of another trough of similar design and so on in series. The cups are made to slide into position so that they can be removed with the amalgam when necessary.—A. W.

*Gold or other Valuable Metal, Improved Method and Machinery or Apparatus for Extracting, from Crushed Ore or Material containing it.* A. S. F. Robinson, Wantage. Eng. Pat. 516, Jan. 7, 1897.

An externally amalgamated revolving horizontal drum, with its lower surface just dipping into water, has its upper outward surface fed with ore. In the water trough underneath, is a series of amalgamated paddles and rollers supported on endless chains, dipping and travelling into the water and out again over chain wheels at each end. The ore falls from the revolving drum on to these paddles, and is carried forward by them along the curved amalgamated bottom of the trough to an inclined shute at the end, down which it falls and is carried into a second trough with paddles of similar construction, and so on to the end of the series. The drum and exposed amalgamated parts are subjected to air blasts to partially dry them, so that the ore and amalgamated surfaces at these stages are only partially damp.

—A. W.

*Furnaces for Ores, Heating and Roasting, Impts. in.* W. P. Thompson, Liverpool. From W. E. Roberts, J. E. Gaylord, and F. P. Davidson, all of Butte City, Montana, U.S.A. Eng. Pat. 24,122, Oct. 19, 1897.

In single or in superimposed multiple-stage hearths, a slot is provided along each side of the furnace just above the level of every hearth. Outside the slot is a track upon which rolls the end support of a rake, which is passed across the furnace. The mechanism of the rake is thus outside the furnace. The roof or various stages of the furnace are supported by brackets attached to uprights at frequent intervals. In single-stage hearths the rake travels backwards and forwards from end to end, but in those with multiple stages, the rake passes from one stage to the next.

—W. G. M.

*Steel, Impts. in the Manufacture of.* J. W. Spencer, Newcastle-on-Tyne. Eng. Pat. 28,836, Dec. 16, 1896.

This process is a combination of the basic Bessemer and open-hearth methods. The pig iron is blown in a basic-lined converter, into which is also introduced oxide of iron, as ore or scale, and lime. The oxide of iron, besides assisting the oxidation of the impurities, helps the slag to retain these impurities when oxidised, thus saving the necessity of an after-blow to remove phosphorus. The charge is then run from the converter into an open-hearth furnace containing scrap or pig iron, or both, when the necessary additions of carbon, manganese, &c. are made.

—A. W.

*Steel, Impts. in the Manufacture of.* C. de E. Llatas, Paris. Eng. Pat. 28,880, Dec. 16, 1896.

To produce steels of various grades, according to their requirements, the metal is first made to contain the necessary amount of carbon, and then the manganese and silicon are added in definite inverse proportions. A table of these proportions is given, starting with a mild steel containing 0·15 per cent. of carbon, 0·625 of manganese, and 0·025 of silicon. For every increase of 0·05 of carbon, the manganese decreases 0·025 and the silicon increases 0·025 per cent., until a hard steel is reached containing 1·00 of carbon, 0·20 of manganese, and 0·45 per cent. of silicon.

—A. W.

*Iron, Melting, Process of; Impts. in or relating to.* T. Doherty, Lambton, and P. D. Crear, Hamilton, both in Ont., Canada. Eng. Pat. 2226, Jan. 27, 1897.

The use of steam in cupolas was protected by Eng. Pat. 8368, of 1896, of which this is an extension. The steam must be introduced in exactly the right proportion; if in insufficient quantity, no benefit results; if in excess, harm is done. The steam is introduced through a narrow and horizontal concentric pipe in each tuyère, ¾ in. pipes communicating with a 1 in. main pipe sufficing for cupolas with a diameter of less than 3 ft. 6 ins. The quantity of steam used is controlled by a cock on each of the narrower pipes. An ordinary cupola may be used; it is started as usual, and steam is first let in when the water from the bed sand is evaporated (from 10 to 15 min. after the air-blast is started). Two certain indications of the correct proportion of steam exist. One is the formation of a flame or aureola within the cupola at the mouth of each tuyère; this may be seen through the usual sight hole to have a bluish centre and a yellow or reddish-yellow fringe. The other is the evolution of a multitude of bright, flashing, white sparks, which rise about 2 ins. from the surface of the metal as it flows from the tap-hole. The emission of coarse, reddish sparks indicates an insufficiency of steam, whilst the entire absence of sparks points to an excess of steam being employed.—W. G. M.

*Steel or Nickel Steel, and Articles made from Steel or Nickel Steel, Impts. in the Manufacture of, and in Apparatus employed therein.* B. K. Jamison, Philadelphia, U.S.A. Eng. Pat. 8578, April 3, 1897.

The steel or nickel steel is slowly heated to a red heat and then plunged, wholly if the entire piece is to be hardened, or partially if only the surface is to be so treated, into a bath containing to every gallon of water, 3 oz. each of spirits of nitre, aqua ammoniæ, sulphate of zinc, and sulphate of alumina and ammonia, with 4 oz. of ammonium chloride, and 6 oz. of glycerin. The steel is left in this bath at a temperature of about 110° to 125° F. for about 30 or 40 minutes, whereby it undergoes a molecular change and becomes fibrous and hardened. When low-grade steel is thus to be face-hardened, it may be partly immersed, and hot plates may then be applied to the back. Again the metal may be face-hardened to any desired depth, and then be superficially softened. A special arrangement of plant is protected. This consists of a tank with a crane for conveying the ingots and lowering them into it, an overflow pipe with a valve, leading to a reservoir, a pump to convey the liquid through a worm placed in a tank with a spraying-rose above for the purpose of cooling it, to an upper cistern from which it is again led into the hardening tank.

—W. G. M.

*Steel and Wrought Iron of Superior Quality, New Construction of Converter-Apparatus for Producing.* T. Levoz, Stenay, France. Eng. Pat. 27,546, Nov. 23, 1897.

The converter is roughly of dumb-bell shape supported in a horizontal position by trunnions placed on either side of the connecting channel. The chamber at one end is provided with side- or bottom-blowing tuyères, and with an aperture at the top for introducing the charge. This aperture is closed during the period of the blow, and the gases pass through the horizontal channel into the opposite chamber, and thence through an opening in the roof thereof. When this chamber is thoroughly heated, scrap is introduced into it through the escape-vent for the gases, and finally the usual additions are made through the same opening.—W. G. M.

*Zinc Blende and other Ores, Impts. in the Reduction of, and Recovery of the Lead with other Metals therefrom.* H. R. Angel, London. Eng. Pat. 1034, Jan. 14, 1897.

The ore is mixed with sodium sulphate and small coal, and fused in an open-hearth gas-furnace. The lead, containing most of the silver and gold, is tapped out, whilst the volatile metals (zinc, &c.) are to be collected in chambers. Any residual gold or silver is removed by adding galena to the molten slag. If uncalcined or imperfectly calcined ores are used, about as much zinc oxide as there is sulphur left in the ore is to be added to the charge. This oxide is to carry off the sulphur, and is to be recovered in the flues for use again. An alternative method of heating is given.—W. G. M.

*Zinc from Zinciferous Gases in the Distillation and Smelting of Zinc; Process of Recovering.* A. M. Clark, London. From R. H. T. Biewend, Clausthal, and Actien Gesellsch. f. Zink Industrie, vormals Wilhelm Grillo, Neumühl-Hamborn, Germany. Eng. Pat. 30,152, Dec. 31, 1896.

In the condensation of zinc, gases as highly concentrated as possible should be used; access of air, water vapour, or carbon dioxide to the gases should be prevented; the temperature of the latter should not fall below 450° C. before the completion of condensation; and the deposition of liquid drops of zinc should be facilitated. The almost complete condensation of the zinc in liquid form, without formation of zinc-dust, whether from retorts or blast-furnaces, is sought in this process. To this end the zinc-bearing gases are dry-filtered through condensers filled with fragments of charcoal or other porous or rough substance, the direction of the gaseous current being reversed from time to time. In retort practice, two groups of any convenient number of retorts communicate with a horizontal condenser (sloping slightly towards the middle from each end), each group being placed on the opposite side of a central depression and sump provided with a deflecting plate, which causes the gases to pass down one side of the depression and up the other side. At each end of the condenser is a vent, which may be opened at will. The groups of retorts and the vents are used alternately, so that the gases pass through the condenser from end to end before escaping, and in alternate directions. The condensed zinc collects in the sump, and is drawn off from time to time.—W. G. M.

*Lead Sulphide Ores, Method of Treating.* S. Ganelin, Philadelphia, U.S.A. Eng. Pat. 26,046, Nov. 9, 1897.

In the absence of water, lead sulphide reacts with fused zinc chloride to form lead chloride and zinc sulphide by simple exchange. Lead and silver sulphides are therefore projected into a melted charge of anhydrous zinc chloride. Subsequently the lead is to be separated either by introduction of zinc, or by leaching out the chlorides (the silver accompanying the lead). Lead-zinc sulphides may be similarly treated, the unaltered zinc sulphide being left behind on leaching. In place of zinc chloride, cryolite or aluminium fluoride may be used, in which case aluminium sulphide is formed.—W. G. M.

*Metallic Coating of Objects of Stone, Plaster, and Analogous Substances, Impts. in.* A. de Boischevalier, Paris. Eng. Pat. 5022, Feb. 24, 1897.

The objects are immersed in a bath of melted animal or vegetable wax till thoroughly impregnated, removed, and the superfluous wax brushed off. After cooling, they are coated with plumbago moistened with petroleum spirit, dried, well brushed, and then immersed in a coppering bath in the usual way. When the immersion of the object in melted wax is impracticable, the surface is well brushed over with a coating composed of wax, benzine, petroleum spirit, resin, linseed oil, and plumbago; thus prepared, it is immersed in the coppering bath.—J. T. D.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (*A.*)—ELECTRO-CHEMISTRY.

*Gases prepared by Electrolysis, the Reputed Active Condition of.* F. Winteler. Zeits. f. Elektrochem. **4**, [14], 342.

*See under* XXIV., *page* 189.

### PATENTS.

*Chlorates of Sodium and Potassium, Impts. in the Means employed in the Electrolytic Production of the.* [*Cathode Tank.*] T. Parker, Wolverhampton. Eng. Pat. 25,631, Nov. 14, 1896.

The electrolytic vessel is formed of iron or other suitable metal and acts as cathode, either alone, or in combination with metallic plates connected with it. The anode consists of pieces of carbon or platinum suspended in the electrolyte which is maintained at a temperature of 30° to 40° C., and covered with a layer of pumice or other non-conducting material, such as cork, diaphragms being dispensed with. —G. H. R.

*Ozone, Impts. in and applicable to Means for Electrically Obtaining and Employing the same for Curative and other Purposes.* [*Wire Gauze Electrode.*] E. Böhm, London. Eng. Pat. 615, Jan. 9, 1897.

The ozoniser consists of an inner glass vacuum tube containing a wire gauze cylinder or spiral of interlaced wire, connection with which is made by means of a loop of wire fused into the glass. Over this inner cylinder is drawn a sleeve of wire gauze connected to the opposite pole of the electrical circuit. This ozoniser is enclosed in an outer glass cylinder provided with union pieces through which pass the leads, and with a mouth-piece or nozzle by means of which the ozone can be applied as desired. —G. H. R.

*Ozone, Impts. in Electrical Apparatus for the Production of, or for other purposes.* A. Verley, Paris, France. Eng. Pat. 1337, Jan. 18, 1897.

In order to increase the frequency of the alternate electric discharges passing through the ozoniser, this is not connected direct to the transformer, but a condenser is interposed, one sheet of the ozoniser being in metallic connection with one plate of the condenser, whilst the connection of the other sheet with the other plate of the condenser is interrupted by an air space so that the alternate discharge of the condenser produces Hertzian waves in the ozoniser.—G. H. R.

*Electrical Accumulators* [*Electrodes covered with Rubber*], *Impts. in.* E. Heyl, Manchester. Eng. Pat. 4916, Feb. 23, 1897.

Claim 1. "In accumulators, a positive electrode made from lead or other metal in the form of fine threads or strips pressed closely together to form a porous plate, the plate being afterwards dipped into rubber or like solution [containing suspended material, chromate of lead] forming a film around each thread, which film is afterwards treated so as to render it porous," &c. It is proposed to cover the grids of paste accumulators with spongy vulcanised rubber, covering the paste with a similar film, and covering the said threads or strips by thin strips of rubber wound around them and afterwards vulcanised.—J. C. R.

*Electric Batteries* [*Zinc-Carbon*], *Impts. relating to.* L. J. H. Rochatte, Paris. Eng. Pat. 24,019, Oct. 28, 1896. [Date claimed U. I. C., April 21, 1896.]

The "exciting liquid" is: Bichromate of soda, 250 grms.; sulphuric acid, 300 grms.; water, 450 grms., prepared "in a cold state." Claim 1. "An electric battery comprising an outer vessel containing water in which is placed an annular porous vessel or cell containing the exciting liquid and consisting of two concentric cylinders fixed to the bottom of the outer vessel, rods of carbon united at their upper part and forming the positive electrode which is immersed in the said exciting liquid, and zinc cylinders forming negative electrodes placed in the water outside the annular vessel and in the centre of the same," &c. Other claims relate to "emptying and feeding the elements," and making electrical connections by means of "pure nickel."—J. C. R.

*Electrolytic Apparatus, Porous Diaphragms for* [*Vitrified Magnesia, &c.*], *Impts. in.* J. D. Darling and C. L. Harrison, Philadelphia, U.S.A. Eng. Pat. 22,236, Sept. 28, 1897.

Claims: 1. . . . "in porous diaphragms for electrolytic apparatus, the combination, with a support, of a granular filling consisting principally of vitrified oxide or oxides [magnesia, &c., fused in an electric furnace, then crushed] substantially resistant to combination or fluxing by a fused hydroxide or other above specified compound ['alkaline hydroxides, or the haloid salts of alkali metals, or the alkaline earth metals, or the nitrates or sulphates of such metals'] under the conditions of electrolysis." . . . 2. . . "a granular filling consisting principally of vitrified magnesia." . . 3. . . . "the combination of a pair of cylindrical, perforated supports, arranged with an interspace between them; bases for said supports, an intermediate granular filling consisting principally of vitrified magnesia arranged in the interspace between said supports; and an annular top of cement above said filling [the 'filling' constitutes the primary feature of this invention]."—J. C. R.

*Chemical-Electric Generators, Impts in.* [*Zinc-Lead Peroxide Couple.*] H. K. Hess, Syracuse, U.S.A. Eng. Pat. 25,386, Nov. 2, 1897.

The battery consists of an outer casing which contains the electrolyte, preferably zinc sulphate solution, within which are placed alternately the positive and negative plates. The former consist of a perforated casing of metal, such as aluminium, and contain a fluid amalgam of zinc, the perforations being closed with porous non-conducting plugs; or the amalgam may be contained in casings of porous non conducting material. The negative electrodes consist of perforated carbon casings containing semi-fluid paste of peroxide of lead and sulphuric acid, and openings are provided in the bottoms of each set of plates for removing the spent material, while conduits are provided for supplying fresh active material to the casings, and non-conducting separators which are open at the bottom, keep the plates apart.—G. H. R.

*Electrical Batteries* [*"Storage" Electrodes, &c.*], *Impts. in.* W. W. Hanscom and A. Hough, San Francisco, U.S.A. Eng. Pat. 25,703, Nov. 5, 1897.

Claim 1. "The method of constructing elements for storage batteries consisting in electrolytically producing slabs of spongy lead from litharge and sulphate of ammonia, then compressing parts thereof to a denser mass to form supports for the said elements." . . . 2. . . . "mixing litharge and sugar, setting the same by adding sulphate of ammonia, dissolving out part of the sugar and electrolytically reducing to spongy lead, &c."—J. C. R.

*Accumulator Plates, Impts. in the Preparation of.* Dr. C. A. Bennert, Godesberg, Germany. Eng. Pat. 26,441, Nov. 12, 1897.

"The improvement consists essentially in the use of plumbic oxide and peroxide prepared from electrolytic white lead or electrolytic plumbic hydroxide." "By means of the process detailed in Eng. Pat. 14,801, 1895 (this Journal, 1895, 975), a white lead may be obtained, a given volume of which weighs about one half of an equal volume of the product previously on the market. In like manner the plumbic oxide and peroxide obtained from this product of the electrolytic process is about one half as heavy as the finest and lightest kind of the respective compounds hitherto produced" . . . "these lead oxides possess a degree of purity previously unattained." The claim is: "preparation of accumulator plates characterised by the use of plumbic oxide or peroxide prepared from electrolytic white lead or electrolytic lead hydroxide, for the purpose of diminishing the weight of the plates and of increasing their capacity in consequence of the extreme fineness of division and purity of these oxides."—J. C. R.

### (*B.*)—ELECTRO-METALLURGY.

*Electrolytic Copper-baths in America, Treatment of spent.* T. Ulke. Zeits. f. Elektrochem. **4**, (1898), 309—313.

In the electrolytic refining of copper, the baths, after several months' use, become more or less saturated with impurities which come from the anode-copper. Unless these impurities are from time to time removed, they are partially deposited on the cathodes and make these unsuitable for drawing into wire. The baths also become gradually richer in copper sulphate, owing to the solvent action of the acid electrolyte upon copper: this effect is increased by the evaporation of water from the warm solution. The old solution may either be freed from its chief impurities (arsenic, antimony, and iron) and then returned to the bath: or a portion of it (replaced by fresh electrolyte) may be removed and worked up for copper sulphate and other compounds.

Arsenic may be present in the anodes either as the metal or as arsenate. In the former case it goes into solution during the electrolysis as arsenious acid, but does not deposit on the cathodes until the bath is saturated with arsenious acid. In the latter case, if the bath be neutral, the arsenic will be found wholly in the form of a spongy deposit: if the solution be acid, the arsenic will be found in the bath as arsenic acid. Antimony in the anodes goes partly into solution (from which, after standing for some time, it is partially precipitated) and partly remains as basic sulphate on the anode. Iron in the anodes goes into solution.

The following methods of dealing with the impurities are employed in various large copper works in America:—In Baltimore, a part (about one-fifth) of the electrolyte is periodically removed and worked up for copper sulphate, for which there is a ready sale (to the Western Union Telegraph Co.). The mother-liquor is treated with scrap-iron to recover the remainder of the copper. The composition of the bath is kept as constant as possible by adding freshly prepared solution. A similar method is followed at the Balbach works in Newark. The direct working-up of the old baths for copper by cementation or precipitation with iron is not found to be a success, principally because the iron sulphate thus obtained is not readily soluble. It is understood that the purifying process used at Anaconda consists in passing the used solution repeatedly through a layer of oxidised copper, so as to partially precipitate antimony and bismuth, and in oxidising the solution (now nearly neutral and saturated with copper) by means of air, so as to partially precipitate iron, &c. One of the best methods consists in purifying the old solution in special electrolytic baths in such a way as to deposit the impurities (chiefly arsenic and antimony) upon copper cathodes, the anodes being of lead. A current is used which is not strong enough to deposit iron, although it will deposit copper, antimony, and arsenic. The solution, freed from arsenic and antimony, is returned to the ordinary bath, and this goes on until the bath contains so much iron that it is necessary to remove the iron or work up the solution for copper sulphate. In the Western States the latter process is resorted to as rarely as possible, because copper sulphate there does not find a ready sale.—D. E. J.

*Precipitated Metals, Occlusion of Gases by.* F. Winteler. Zeits. f. Elektrochem. **4**, [14], 338—341.

Most metals, when deposited electrolytically, behave in different ways, so that each requires to be separately investigated. One characteristic, common to a great many metals, is the formation of a scaly deposit as soon as the film reaches a certain thickness, whilst another objectionable feature is the production of a spongy or powdery mass when the current density exceeds a certain maximum limit. Nickel furnishes an example of the metals which form a brittle scaly deposit, whilst copper on the other hand, readily yields a spongy mass. Förster (this Journal, 1897, 808), has shown that the difficulty with respect to nickel, may be obviated by keeping the bath at a temperature of 50°—90° C. Since the absorption of hydrogen by metals exercises a great influence on their physical properties, the author recapitulates some of the previous work, and then describes the behaviour of electro-deposited iron and copper, with a view to throwing more light on the subject. Iron obtained from neutral or acid baths is very brittle, and its hardness is 5·5. On ignition, however, these characteristics diminish, and thin plates become so soft, that it is possible to bend them backwards and forwards many times without breaking. This is ascribed to the escape of gas on ignition.

According to the author, iron reduced by electrolysis contains as much as 164 volumes of gas per unit volume of iron, the bulk of the absorbed gas, which was determined by ignition *in vacuo*, being in each case hydrogen. Nitrogen, carbon monoxide, and carbon dioxide, also formed a considerable but variable, proportion of the gas pumped off.

It appears that the thinner the film of iron produced, the greater is the relative quantity of gas occluded, and the author attempts to trace a connection between the volume of occluded gases, and the thickness of the film.

Under similar circumstances, copper occluded 4·4 volumes of gas, of which 3·4 were hydrogen.

On electrolysing hot solutions of these metals, however, deposits were obtained, which were quite free from occluded gases.

Commercial nickel in the form of cubes, appears to occlude 165 volumes of hydrogen, when it is made the cathode, during the electrolysis of dilute sulphuric acid.
—J. S.

*Silver and Copper, Electrolytic Separation of.* N. Revay. Zeits. f. Elektrochem. **4**, 1898, 313.

*See under* XXIII., *page* 184.

## PATENTS.

*Copper and other Metals, Impts. in the Method of and Apparatus for the Electro-deposition of on Rotary Mandrels.* [*Aluminium Anode Supports.*] W. E. Heys, Manchester. From E. Dumoulin, Paris, France. Eng. Pat. 2710, Feb. 2, 1897.

According to this process, which is an improvement on that described in Eng. Pat. 16,360, 1895, the anode supports consist of an aluminium supported cage or basket lined with suitable perforated or pervious material, which contains the Rio Tinto precipitate, or other granular or pulverulent form of the metal to be deposited. By the addition of a suitable quantity of aluminium to the copper anode, or of sulphate of aluminium to the electrolyte, an alloy of these two metals can be obtained. To facilitate the removal of the deposit, the rotary copper mandrel forming the cathode is slightly tapered, and coated with india-rubber solution, which is covered with powdered plumbago.

When there is animal membrane in contact with the mandrel, the temperature of the electrolyte is kept at or below 60° F., by means of a circulatory system, and its oxidation is effected by the injection of air, and the solution is filtered by passing the electrolyte through special tanks.
—G. H. R.

*Metal Articles by Electro-deposition, Impts. in Apparatus for Coating.* G. H. Nash, Birmingham. Eng. Pat. 14,708, June 17, 1897.

The articles to be plated are placed in a perforated box to which a rocking motion can be imparted, and which contains fixed anodes with suitable electric connections. The box is preferably supported in the electric tank in a cradle provided with trunnions for convenience of handling.
—G. H. R.

*Ore, Process of and Apparatus for Electrically Treating* [*Superheated Air*]. A. J. Boult, London. From F. H. Soden, Chicago, U.S.A. Eng. Pat. 22,203, Sept. 23, 1897.

This method of electrically treating iron ores more especially, to purify them preparatory to smelting, though intended principally for use with such as have already been partially purified by the Cherry or some similar process, is also applicable to the cases of gold, silver, copper, and lead ores, and is carried out in an apparatus having a receptacle provided with doors having connected with them hinged sections of a perforated diaphragm normally forming an upper gas chamber, whilst a sectional screen diaphragm in the lower part forms a lower gas chamber. A covered inlet pipe is provided to the upper chamber, a pressure valve for the escape of gases from the heater, and communication by a valve-controlled pipe, with the lower gas chamber which is provided with a discharge pipe. The finely divided ore is placed in the receptacle, which is formed of some refractory material, and through which pass resistances for the purpose of heating the ore electrically, and whilst it is being so heated, superheated air and a purifying gas such as hydrogen, are passed through it, ordinary atmospheric air being excluded.—G. H. R.

*Metals from their Ores and Solutions, Improved Electrolytic Process and Apparatus* [*Electrolytic "Sluice," &c.*], *for the Separation of.* E. Motz, of Brewer Mine, and H. F. Welch, Charleston, South Carolina, U.S.A. Eng. Pat. 25,400, Nov. 2, 1897.

The inventor says: "The ore containing the metal will first be reduced by crushing or otherwise into finely-divided particles, and then mixed with a solvent [cyanide mentioned] in solution, and fed in the upper end of the sluice (the electrical circuit having been closed), and allowed to pass by gravity through the *undulatory* (horizontal) channel therein." This form of electrolytic "sluice" is described and shewn, and would appear to be the main subject of the invention. There are 16 claims; 1 and 2 embracing:—The process of treating ore for the separation and recovery of metal consisting in reducing or crushing the ore, then passing it with an electrolyte between anodes and cathodes included in circuit with an electric generator and simultaneously agitating the electrolyte, ore, and metal by gravity [" the frame or sluice will be disposed in an inclined position "]." . . . . . " passing the pulp thus made through an undulatory passage having its opposing walls composed respectively of anodes and cathodes, &c."—J. C. R.

## XII.—FATS, OILS, AND SOAP.

*Illipé-Nut Fat.* H. Becker. Zeits. für Öffent. Chem. **3**, 545. Analyst, 1897, **22**, [261], 327.

The author has examined a sample of the fat expressed from the illipé-nut, which has been imported from the Dutch East Indies in ever-increasing quantities for the past 18 or 20 years; the fat is principally used, in conjunction with palm-kernel oil, in the manufacture of candles. The fat was readily soluble in ether, chloroform, benzene, and petroleum spirit, partly soluble in cold alcohol, and readily in hot alcohol, though separating out again from the latter on cooling. The following results were obtained on analysis:—Specific gravity at 100° C., 0·8854; saponification number of the fat, 194·04; saponification number of the fatty acids, 206·0; iodine number of the fat, 29·93; iodine number of the fatty acids, 31·64; melting point of the fat, 35·5°—36·5° C.; melting point of the fatty acids, 54·5°—55·5° C.; solidification point of the fat, 24·6° C.; solidification point of the fatty acids, 52·5° C.; unsaponifiable matter, 0·397 per cent.; Reichert - Meissl number, 1·23; free acid, 17·245 per cent.; mineral matter, trace.—A. S.

*Olives, The Transformation of Sugars into Oil in.* C. Gerber. Comptes Rend. 1897, **125**, [18], 658—661.

From experiments on the amount of oxygen absorbed by olives from the atmosphere and the carbon dioxide given off, the author has established that at a very early stage, when the amount of mannite is largest and that of the oil smallest, the respiratory quotient $\frac{CO_2}{O}$ is below unity. As the olives become reddish-violet, the stage in which the mannite begins to diminish and the oil to increase, the quotient rises above unity, and then gradually decreases as the ripening proceeds, until it is again less than unity, and the mannite has altogether disappeared. Since olives contain no acids such as citric, tartaric, or malic acids, and do not produce alcohol, in both of which cases the respiratory quotient would also be above unity, the author concludes that there must be connection between the higher quotient and destruction of the mannite. If the latter were simply oxidised ($C_6H_{14}O_6 + 13O = 6CO_2 + 7H_2O$), the quotient would be below unity $\left(\frac{CO_2}{O} = 0{\cdot}92\right)$. But oxidation of mannite with the formation of fatty substances according to the equation—

$$11C_6H_{14}O_6 = C_{51}H_{94}O_6 + 30H_2O + 15CO_2$$

can only be produced with a quotient higher than unity. Hence this is regarded as a direct proof that the oil is derived from the sugars, and in particular the mannite, which are found in the olive in its earlier stages.—C. A. M.

*Lecithin Content of some Seeds and Oil Cakes.* E. Schulze. Landw. Vers. Stat. **49**, 203; Chem. Centr. 1897, **2**, [21], 1031.

*See under* XXIV., *page* 190.

*Mineral Lubricants, Valuation of.* R. Kissling. Chem. Zeit. **22**, [4], 22—23.

*See under* XXIII., *page* 187.

*Wool-Grease, Application of, in the Leather Industry.* F. Veselý. Časopis pro. prumysl. Chemický. 1897, **7**, 313.

*See under* XIV., *page* 166.

## PATENTS.

*Fats and Oils, Impts. in Apparatus for Refining.* J. Davidson and C. J. Hepburn, both of Sydney. Eng. Pat. 14,238, June 11, 1897.

The oils or fats, heated to a sufficiently liquid state, are forced by centrifugal action through a filtering bed of sawdust carried in a centrifugal basket, usually composed of woven wire, lined with a porous fabric to retain the sawdust, and having (if necessary) an open wire cloth between the fabric and the shell of the basket. The filtered oil is thrown in a fine spray against the corrugated inner wall of an annular water chamber surrounding the basket, the cooling being greatly promoted by the current of air produced in the intervening space, by the rotation of the basket.
—C. A. M.

*Oils and Fatty Substances, An Improved Process of Deodorising.* C. Culmann, Hamburg. Eng. Pat. 26,191, Nov. 10, 1897.

The oils or fats are subjected to the simultaneous action of a saturated solution of an acid substance (such as alum or aluminium sulphate) and of steam of 108—110 C. Part of the malodorous matter is separated as a glutinous mass, whilst the volatile portion passes off with the steam. The dilution of the saturated solution of the chemical, with the consequent formation of an emulsion, is prevented by covering the receiver with non-conducting material, and so regulating the steam supply that the water in the solution does not increase. The oil is maintained at about 80 until the sediment deposits, when the clear upper layer is drawn off. It is quite odourless, nor does it reassume its former odour on warming, even in the case of fish oils.
—C. A. M.

*Glycerin, Impts. connected with the Concentration or Distillation of.* E. G. Scott, Liverpool. Eng. Pat. 5889, March 5, 1897.

The object of this invention is to recover the glycerin which in the ordinary process of distillation is volatilised with the steam. This is effected by carrying the steam coming from the concentrating apparatus through a "fractional condenser," in which the cooling surface is relatively small, and is so proportioned that the less volatile glycerin is condensed, whilst the greater part of the steam passes on into an ordinary condenser.—C. A. M.

*Soap Powder, Manufacture of an Antiseptic.* A. F. Dunn and A. Waithman, London. Eng. Pat. 5820, March 4, 1897.

Hard soap material, preferably stoved and pulverised, is mixed in suitable proportions with sawdust, preferably sterilised, and antiseptic material, such as $\beta$-naphthol. The following are given as suitable proportions:—Pulverised soap base, 60 per cent.; sawdust, 35 per cent.; and antiseptic material, 5 per cent.; but these may be varied within limits.—C. A. M.

*Soap Tablets, Impts. in or relating to.* G. H. Evans, Oroville, California. Eng. Pat. 24,779, Oct. 26, 1897.

A section of sand-soap or pumice-stone backed with or dovetailed into a section of ordinary toilet soap.—C. A. M.

*Soap for Use in Dyeing and Finishing Textile Fabrics and Materials, and for other Purposes; Impts. in the Manufacture of.* J. Stockhausen, Crefeld, Germany. Eng. Pat. 21,868, Oct. 27, 1897.

Claim is made for a gelatinous soap ("Monopole Soap") prepared by heating sulphonated oils or fats, whether specially prepared or occurring in commerce, with a larger amount of alkali or base than is used for Turkey-red oil (*e.g.*, 100 parts of sulphonated oil with 60 parts of soda-lye of 36°—37 B.), and boiling the precipitated soap until it becomes gelatinous when cold. This soap differs from Turkey-red oil in being a perfectly saponified solid substance, containing more alkali or base, and in having different properties. Thus, in dyeing fabrics with benzidines, diamines, &c., alone, or in combination with salts or acids, the soap is not precipitated, nor is there a deposit on the fibre. "Since the lime salt of sebacic acid is soluble in water, there is no precipitation when water containing that base is used, and the soap may be used to dissolve the precipitates which form in ordinary soap baths. There is a more complete utilisation of the dye-bath, with a consequent saving of colouring matter; and in the case of semi-silk materials, the cotton fibre is dyed a darker shade than the silk, which is advantageous. Similarly the use of this soap with semi-wool fibres is said to render them bright and soft, whilst Turkey-red oil leaves them dull and sticky. Alkali Blue is stated to give a better extract of colouring matter when used with "monopole soap." When mordants are used, the metallic oxide is fixed better, and the soap may therefore be used with advantage for printing purposes. For finishing and sizing it has the advantage that it is not precipitated by salts (Epsom salts, Glauber's salt, &c.) like ordinary soap, does not separate on cooling, like ordinary fats, or render the goods sticky and form emulsions, like Turkey-red oil.
—C. A. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

### PATENTS.

*Anti-Fouling Compositions or Paints, Impts. in the Manufacture of.* J. C. Mewburn, London. From J. C. Taylor, Las Palmas, Canary Islands. Eng. Pat. 3576, Feb. 10, 1897.

This is an ordinary paint containing mercuric chloride, the special feature of the invention being that the anti-fouling agent is dissolved in alcohol before it is added to the rest of the material, in order to ensure its thorough dissemination throughout the mass. Suitable proportions are 1 gallon of a 1 : 5 alcoholic solution of corrosive sublimate to 20 or 40 gallons of paint.—F. H. L.

*White Lead, Process of Manufacturing, by Electrolysis.* H. C. Woltereck, New York, U.S.A. Eng. Pat. 19,179, Aug. 19, 1897.

The process consists in employing a lead anode and an alkaline electrolyte composed of a solution of a salt of an alkali, preferably ammonia, in combination with any acid which will produce a soluble lead salt, that is to say, a salt the solution of which will give a precipitate of hydrated carbonate of lead in the course of the reactions taking place in the process. The electrolyte should contain more than 5 per cent. of the alkaline salt and an alkaline bicarbonate. The cathode is formed of lead, carbon, or other suitable material, and on the passage of the current, a soluble lead compound is produced, which is converted into the hydrated carbonate of lead by the simultaneous generation of free carbonic acid at the anode and caustic alkali at the cathode. The spent alkaline bicarbonate is regenerated by passing into the electrolyte a current of carbonic acid gas, and the process is continued until the anodes are completely dissolved.—G. H. R.

*Printers' Inks or Paints, Impts. in the Manufacture of.* A. Stoop, Amsterdam. Eng. Pat. 24,504, Oct. 22, 1897.

According to this patent, the mineral oil obtained at Grisee, near Soerabaya, in Java, which thickens on exposure to air, partly by evaporation and partly by oxidation, makes a good vehicle for inks or paints. The oil should have its impurities removed, either by treatment with petroleum spirit, or by heating to 70° or 80° C. for some time. Drying may be retarded, if necessary, by the addition of vaselin; but fatty oils must not be introduced.—F. H. L.

## (*B.*)—RESINS, VARNISHES.

*Xanthorrhœa Resins* (*West Indian*). Imp. Inst. J., Dec. 1897, 365.

Two varieties of Xanthorrhœa resin occur in commerce—the "yellow" resin and the "red" resin. The yellow variety is obtained from *Xanthorrhœa hastilis*, but the exact botanical origin of the "red" resin is not accurately known. The commercial article varies considerably in quality and colour, some specimens containing as much as 40 per cent. of woody material; while others, although free from extraneous matter, are very dark coloured, probably due to the charring which occurs during the forest fires so common in Australia. The resin is used in the manufacture of sealing-wax, the preparation of lacquers, stains, and varnishes, to a small extent in medicine, and for other purposes; and some of the darker varieties, when dissolved in alcohol, make an excellent mahogany stain for wood. Xanthorrhœa resin has been examined by Stenhouse (Ann. Chem. Pharm. **57**, 84) and Hildebrand (this Journal, 1897, 340). The volatile oil obtained by the distillation of the resin has also been examined by Schimmel and Co., (See Schimmel's Report, Oct. 1897), who found that it distils between 145° and 240° C.; optical rotation,— 3°; "relative" density, 0·937. When shaken with dilute alkali, the oil yielded a quantity of cinnamic acid; on saponification of the oil, a considerable quantity of cinnamene was obtained.

The West Indian resin which has been examined, resembles in character and appearance the commercial "red" resin. It is light in colour, quite soluble in alcohol, and yields only about 3 per cent. of ash. On treatment with alkali, cinnamic and benzoic acids were obtained; and, by boiling the resin with nitric acid, a large amount of picric acid was formed. The volatile oil obtained from the resin, yielded a large proportion of cinnamene (b. pt. 145° C.) on fractional distillation. A resinotannol was also obtained from the resin. The West Indian resin thus appears clearly to be identical with the Australian variety, and may be used for the same purposes as the latter, except that its light colour would probably preclude its use as a staining material for wood.

It is stated that the resin may be separated from wood fragments and extraneous insoluble substances, by melting at a low temperature and pouring through a suitable sieve; if the temperature be kept low, the resin is not injured by this treatment, the slight loss of volatile matter which may occur being really of advantage for the uses to which the resin is at present put.—A. S.

*African Copals: Their Solubility and Behaviour towards Alkalis, &c.* M. Bottler. Dingler's Polyt. J. 1897, **306**, [9], 212.

In a previous article (this Journal, 1893, 775) the author has already dealt with the physical properties of 12 different varieties of copal resin; he now describes some of the special features exhibited by seven African species on treatment with aqueous alkali hydroxides and carbonates, alcoholic alkalis, ammonia, acids, and several organic solvents. The samples examined were (1) White and (2) Yellow Benguela, (3) White and (4) Red Angola, (5) Sierra Leone, (6) Congo, and (7) Zanzibar copal. The specific gravities and melting points were found to be practically the same as those previously quoted, except in the case of (1) and (2), which melted at 185° and 190° C. respectively. The solubilities were determined (*a*) after drying, and (*b*) after heating; the former being effected by exposing the material in small fragments to the air at 15° C. or 20° C. for 12 weeks, and the latter by heating to 120° C. for 48 hours; but before the actual application of the different liquids, the resins were all finely powdered.

In cold 10 or 15 per cent. aqueous caustic potash and in strong ammonia the West African copals softened, swelled up somewhat, and dissolved to a certain extent with the formation of a yellow liquid; while on heating the residues with a fresh quantity of the same reagent, a little more soluble matter was extracted, but it separated again on further boiling. Weaker alkalis, especially warm 5 or 10 per cent. soda, acted rather more powerfully; and all the solutions frothed more or less on agitation with water, showing that they contained some soap. In a cold 10 per per cent. solution of potash in 90 per cent. alcohol, the resins dissolved partially; and by heating on the water-bath till the spirit was driven off, repeating the process, if necessary, they all, excepting (1), were completely or almost completely dissolved to a brown syrupy liquid, which gave a cloudy yellow solution on dilution with water, and from which the resin acids could be thrown down with hydrochloric acid as flocculent precipitates, drying to a white, yellow, or orange powder according to the colour of the original copal. White Benguela copal was but partly dissolved even on repeated treatment with alcoholic alkali, while Zanzibar copal was practically insoluble both in aqueous and alcoholic potash.

The process recommended by Schrötter for dissolving dammar, which consists in softening the resin in old, rectified turpentine, then adding aqueous potash and boiling until the turpentine has evaporated, is not satisfactory in the case of copal. Congo copal can be almost wholly dissolved in this manner, but the other West African samples are only partially soluble; and they all yield turbid solutions that deposit on standing. A liquid containing 25 grms. of caustic soda and 83 grms. of crystallised sodium carbonate in 2·5 litres of water, which has been found serviceable in similar instances for obtaining the resin acids, is also without much effect on these copals. It is possible, however, that Tschirch's method of passing steam through a strongly alkaline liquid containing the resins might lead to complete saponification, but some decomposition would probably ensue from working in this way.

For commercial purposes the copals must therefore be dissolved in alcoholic alkali, and the solvent recovered by distillation. On adding metallic salts to the resulting soap solutions, coloured copal-resinates may be produced: magnesium chloride gives a yellowish-white, ferrous sulphate a grey-green, copper sulphate a blue-green, chrome alum a grey-green, lead acetate a white, and manganese sulphate a whitish precipitate; and the two latter compounds can be employed as driers for linseed oil like the corresponding colophony resinates. Other coloured copal resinates may be obtained by precipitating the soaps with magnesium chloride in presence of basic aniline dyes.

Some of these products have been prepared from the white and red Angola copals; they are soluble in absolute alcohol, and form coloured varnishes suitable for glass, porcelain, &c.

The solubility of the copals themselves in organic liquids is always enhanced by the heating (120° C.) process already mentioned. The first five samples are soluble in acetone; (1), (2), and (5) (after roasting) are soluble in ether. (2), (3), and (6) are dissolved by old ozonised turpentine on prolonged heating at the temperature of the water-bath; but to effect this solution satisfactorily, the resins should first be spread out in flat dishes and sprinkled periodically with the solvent. After soaking in ether, (1), (2), (4), and (6) are soluble in warm absolute alcohol; (5) is also dissolved to a large extent if previously heated to 120° C., as already described; but when simply dried, about one-half remains unattacked. If the alcoholic solution of (2) is poured into water, a yellowish-white precipitate of the pure resin is thrown down, which has the same solubility as the crude copal. (2) is soluble in amyl alcohol, (3) in ethyl-alcohol, ether-alcohol, and in a mixture of equal parts of carbon bisulphide, benzene, and rectified turpentine. The best solvents for (7) are benzene and ether-alcohol, but long digestion is requisite. All the copals dissolve in strong sulphuric acid, giving dark red liquids; (3) and (4) are also soluble in hydrochloric acid.—F. H. L.

*Driers and Coloured Varnishes, Composition of some.* C. Collignier. Rev. Chim. Ind. 1897, **8**, 359.

A solid drier sold in France under the name of "Siccative de la Vieille Montagne" is nominally composed of 98 per cent. of zinc oxide with 2 per cent. of manganese sulphate, manganese acetate, and zinc sulphate, all dried and in equal proportions. The diluent is added to control the activity of the material; but as this ingredient is comparatively expensive, it is usually replaced either wholly or in part by sulphate of calcium or barium; and the author has met with a commercial sample containing as much as 96 per cent. of barium sulphate. "Siccatif l'Astral" contains 76 per cent. of inert matter.

In the manufacture of liquid driers, linseed oil should be boiled with the desired substance (litharge, red lead, lead acetate, manganese dioxide, or umber) till it solidifies in a minute or so, when it is cooled and diluted with turpentine. A suitable formula is lead acetate, 2 parts; red lead, 2; linseed oil, 2; and turpentine, 14 parts. This yields a very dark product; but hitherto the problem of manufacturing a pale liquid drier has not been satisfactorily solved.

Coloured varnishes, called in French "siccatifs à parquets," are of two kinds, some having alcohol and some turpentine as their diluent. The latter are preferable because they give a tougher film, and there is less danger of fire during their application. Owing to the low price obtained for them, they are made principally from ordinary rosin; but a certain percentage of dammar, which does not necessarily render their cost prohibitive, should be added in order to reduce the brittleness of the coating. A colourless varnish is first prepared by dissolving 5 or 10 kilos. of dammar and 35 or 30 of colophony in 50 kilos. of turpentine, and, after the solution has been strained, it is mixed with 25 per cent. of a finely ground ochre, selected according to the colour it is wished to produce, the whole being finally rubbed through a fine sieve.

The author remarks that English varnishes are valued very highly in France.—F. H. L.

## (C.)—INDIA-RUBBER, &c.

*Textiloid: A Substitute for India-Rubber, &c.* Cadoret. [Fr. Pat. 256,854 of June 2, 1896.] Leipziger Färber- u. Zeugdr. Zeit. **46**, [10], 450.

Textiloid is a plastic non-inflammable substance, capable of serving as a substitute for india rubber, celluloid, leather, linoleum, ivory, &c., and which may be rolled, stretched, pressed, or spun, and formed into plates, tubes, cylinders, or into soft, silky threads. It may be made either white or coloured.

The common basis of the different compositions used for the manufacture of textiloid, is "resinolin"; this is mixed with various other substances, these latter differing according to the nature of the ultimate product required. The resinolin is prepared as follows:—One part of oil is mixed with 0·75 part of lead carbonate, and 1 part of nitric acid (36° B.) gradually added; the whole is mixed together, and, after standing a few minutes, saponified with 1 part of caustic potash or soda and 2·5 parts of water. The soap thus obtained is decomposed by acid in order to obtain the "resinolin." Several receipts are given for preparing the textiloid, of which the two following are examples:—(1.) Resinolin, 100 parts; zinc oxide, talc, kaolin, or ochre, 20—30 parts; 90 per cent. methyl alcohol, 60 parts. Colouring matter may be added as required. The mixture is allowed to stand for three or four hours, then gum dammar is added, and the whole heated to 30°—40° C. (2.) Resinolin, 1 part; nitrocellulose, 0·5 part; camphor, 0·25 part; ether, 6 vols.; alcohol, acetone, or acetic ether, &c., 1 vol. The mixture is allowed to stand for six hours. In other preparations, the resinolin is mixed with natural resin, cellulose, glycerin, &c.—A. S.

### PATENTS.

*India Rubber Compounds and in Gutta-Percha Compounds, An Impt. in.* E. Garnier and S. J. Prescott, London. Eng. Pat. 11,168, May 5, 1897.

Powdered pumice stone is used to load india-rubber and gutta-percha instead of chalk, &c., the proportions varying according to the application of the finished article. It is claimed that the product is very tenacious, non-inflammable, almost odourless, and not injurious to health.—F. H. L.

*India-Rubber and Gutta-Percha, Impts. in and in the Manufacture of.* H. Markus and G. B. Behrens, Manchester. Eng. Pat. 26,107, Nov. 10, 1897.

Ground cork, which may previously be dyed to any desired colour, is employed as the loading substance in india-rubber and gutta-percha. Its special advantages are lightness, and preservation of the original elasticity of the material.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Leather, Analyses of, at Tharand.* A. Bartel. Dingler's Polyt. J. **305**, Hefte 3, 4, 5, 6, 7, and 8; 65—192.

The composition of leather has been investigated by A. M. Villon (Traité Pratique de la Fabrication des Cuirs et du Travail des Peaux, Paris 1889); Ferd. Jean (Industrie des Cuirs et des Peaux, Paris); H. R. Procter (A Textbook of Tanning, London 1885); The Vienna Research Station for the Leather Industry (W. Eitner and F. Simand, Zeit. f. angew. Chem. and Gerber); The German Tanning School in Freiberg (Deutsche Gerber-Zeit. 1892, 43, &c.).

The author gives a list of 247 samples of leather examined at the Tharand laboratory under Dr. von Schroeder. These are classed under the following heads:—

(A) Sole leather. (B) Light hides (Vasheleder) and unfinished belting leather. (C) Finished belting leather. (D) Harness leather. (E) Upper leather. (F) Weighted and other adulterated leathers.

The estimations are:—(1) Specific gravity, found by displacing mercury in a graduated measuring cylinder, or by Simand's method of direct measurement (Fr. Böckmann, Chem. Tech. Untersuchungsmethoden, 1893, **2**, 555). The sp. gr. is calculated for leather with 18 per cent. of $H_2O$, this being the average found by v. Schroeder (Dingler's Polyt. J. 1894, **293**, 139). The mean sp. gr. of 94 samples of bark-tanned leather with 18 per cent. of $H_2O$, was 1·012.

(2) Moisture, by drying in the air bath at 105° C. (3) Total ash and ash of extract. (4) Fat, natural and added, and its properties. (5) Organic extractive matter (tannin and non-tannin). (6) Sugars. (7) Nitrogen by Kjeldahl's method. (8) CaO and $SO_3$.

From the nitrogen in the dry ash-free leather, the amount of pure hide substance contained in it may be calculated by the formula—

$$H = \frac{L \times Nt}{Nb}$$

in which H = dry ash-free hide substance, L = dry ash-free leather substance, NL = nitrogen in the dry ash-free leather substance, Nb = nitrogen in the dry ash-free hide as prepared for tanning, the per cent. of N being constant for any one kind of hide (J. v. Schroeder and J. Paessler, Untersuchungen verschiedener Blössen, Dingler's Polyt. J. 1893, **287**, 258).

The difference between this (H) and the pure leather substance gives the amount of combined tannin.

These numbers are of value to the practical tanner, as they give him exact information of the amount and extent of the tannage (Durchgerbung). According to v. Schroeder and Pässler (Dingler's Polyt. J. 1892, **284**, 256; this Journal, 1893, 279), a complete tannage is one in which the dry hide substance is combined with its own weight of tannin. This proportion, multiplied by 100, may be expressed numerically by means of the following formula, as the "tanning number" = D, (Durchgerbungszahl):—

$$D = \frac{G}{H} \times 100 = \frac{L-H}{H} \times 100 = \left[\frac{L}{H} - 1\right] 100 = \left[\frac{Nb}{NL} - 1\right] 100.$$

in which G = combined tannin, the other signs as already given. From the nitrogen estimation we can also calculate how much finished leather 1 part of pure hide substance has yielded; this quantity is expressed in the tables as the "product number" (Rendementszahl)—

$$Rz = \frac{100}{H} = \frac{Nb}{L \times NL}$$

The tables are so extensive that the original paper must be consulted for the complete numbers.

The following samples have been selected as being of most interest to English tanners:—

A. 28. Sole leather from Celle, tanned by modern system, in the first and second layer a mixture of $\frac{3}{4}$ oak and $\frac{1}{4}$ mimosa, in the third layer a mixture of $\frac{4}{5}$ oak and $\frac{1}{5}$ valonia.

41. Sole leather from Langenfelde, near Hamburg, tanned by modern methods with quebracho, myrobalans, &c., the so-called "North German sole leather."

46. Sole leather from Steiermark, sweated and swelled in a barley-meal drench. In the first layer, pine bark and knoppern; in the second and third, knoppern and valonia. Hammered, so-called "Pfund" leather.

49. Ditto, after five days' liming, eight pine colouring liquors and three layers: in the first, $\frac{2}{3}$ pine, $\frac{1}{3}$ knoppern; in the second, $\frac{1}{3}$ pine, $\frac{2}{3}$ knoppern; in the third, knoppern only. Rolled, and dried for six hours at 40°—50° C., "Terzen" leather.

78. Sole leather from Cincinnati, U.S.A. Tanned with American oak.

79. Sole leather from Milwaukee. Swelled with sulphuric acid, pure hemlock tannage.

B. 105. Pure canaigre tannage.

108. Strap butt. Bristol tannage. English hide, rounded before tanning, tanned with extract (Levinstein), valonia, and myrobalans.

110. Belly from ditto. Tanned with extract, valonia, and algarobilla.

143. Belting leather from the Rhineland. Oak tannage; in the last layer, oak, valonia, and myrobalans. Damped with glucose solution before rolling.

151. English belting leather.

154. American strap butt (Ohio). Union tannage, 9 parts of hemlock bark, 1 part of oak bark.

D. 159. Unfinished harness leather (cheek). Pure pine-bark tannage.

164. Harness leather, Milwaukee. Pure hemlock.

E. 168. Horse leather from Altona. Slightly grained with pine liquors, and then tanned out with quebracho wood.

174. Upper leather, from an East Indian arsenic kip. Tanned with pine and quebracho at the German tanning school in Freiberg i/S. Description of the tanning in Günther's Deutscher Gerber Zeit. 1892, 13.

192. Brown calf from Barr, Alsace. Oak tannage, old system.

200. Brown Russian calf, Warsaw. Oak.

202. Black Russian calf, Warsaw. Oak.

211. Coloured Russian leather, Sarapul. Pure willow bark tannage smeared with birch oil on the flesh side.

F. 226. English belting leather. Weighted with glucose and barium salts.

233. Strap butt. Weighted with barium chloride.

243. Sheep leather. Pine tannage, with a little quebracho. Unfinished.

| No. | Water (normal). | Total Ash. | Fat. | Organic Extraneous Matter. |  | Leather. |  | Ash of Extract. | Sugars. |
|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | Tannin. | Non-Tannin. | Combined Tannin. | Hide Substance. |  |  |
|  | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |  |
| 28 | 18·00 | 0·78 | 0·67 | 6·75 | 3·12 | 29·11 | 41·75 | 0·43 | 0·16 |
| 41 | 18·00 | 0·52 | 0·23 | 4·64 | 4·71 | 33·36 | 38·54 | 0·40 | 0·16 |
| 46 | 18·00 | 1·18 | 0·35 | 5·84 | 5·18 | 34·65 | 34·80 | 0·90 | 0·43 |
| 49 | 18·00 | 0·86 | 2·97 | 8·56 | 4·29 | 27·94 | 37·58 | 0·71 | 1·03 |
| 78 | 18·00 | 0·85 | 2·94 | 6·96 | 5·73 | 25·84 | 39·68 | 0·73 | 0·23 |
| 79 | 18·00 | 0·30 | 0·81 | 4·13 | 4·88 | 30·69 | 41·19 | 0·25 | 1·39 |
| 105 | 18·00 | 0·75 | 0·87 | 5·18 | 4·76 | 28·37 | 42·07 | 0·56 | 0·12 |
| 108 | 18·00 | 0·64 | 0·16 | 8·32 | 4·12 | 29·55 | 39·21 | 0·41 | 0·05 |
| 110 | 18·00 | 1·44 | 3·19 | 9·77 | 8·13 | 24·22 | 35·25 | 1·30 | 0·07 |
| 143 | 17·02 | 0·55 | 5·42 | 6·92 | 5·23 | 24·89 | 39·97 | 0·35 | 1·38 |
| 151 | 14·09 | 1·11 | 21·70 | 6·25 | 2·07 | 22·28 | 32·50 | 0·55 | 0·07 |
| 154 | 16·46 | 0·19 | 8·54 | 4·42 | 1·33 | 28·83 | 40·34 | 0·10 | 0·10 |
| 159 | 18·00 | 0·89 | 1·30 | 0·81 | 1·62 | 32·34 | 45·04 | 0·48 | 0·21 |
| 164 | 18·001 | 0·23 | 0·15 | 4·37 | 0·98 | 30·77 | 45·50 | 0·20 | 0·16 |
| 168 | 18·00 | 0·48 | 0·29 | 3·12 | 1·29 | 30·43 | 46·39 | 0·33 | 0·17 |
| 174 | 14·81 | 0·28 | 17·73 | 3·67 | 1·90 | 22·71 | 38·90 | 0·29 | 0·00 |
| *174 | 18·00 | 0·33 | 0·82 | 4·42 | 2·29 | 27·32 | 46·82 | 0·34 | 0·00 |
| 192 | 14·15 | 0·67 | 21·39 | 3·18 | 1·12 | 24·23 | 35·26 | 0·24 | 0·14 |
| *192 | 18·00 | 0·85 | 0·82 | 4·01 | 1·40 | 30·52 | 44·40 | 0·30 | 0·17 |
| 200 | 14·74 | 0·51 | 18·11 | 3·45 | 1·19 | 23·53 | 38·45 | 0·43 | 0·39 |
| *200 | 18·00 | 0·62 | 0·82 | 4·17 | 1·43 | 28·47 | 46·49 | 0·52 | 0·48 |
| 202 | 15·61 | 0·55 | 13·28 | 5·01 | 0·68 | 22·96 | 41·91 | 0·54 | 0·20 |
| *202 | 18·00 | 0·62 | 0·82 | 5·72 | 0·78 | 26·21 | 47·85 | 0·61 | 0·23 |
| 211 | 17·10 | 0·60 | 4·99 | 2·45 | 0·33 | 24·16 | 50·37 | 0·32 | Trace |
| *211 | 18·00 | 0·62 | 0·82 | 2·56 | 0·35 | 25·17 | 52·48 | 0·31 | Trace |
| 226 | 17·45 | 4·35 | 3·09 | 5·99 | 10·95 | 22·55 | 35·62 | 2·50 | 6·10 |
| 233 | 15·83 | 14·54 | 12·08 | 4·28 | 1·20 | 21·65 | 30·42 | 11·93 | 0·08 |
| 243 | 16·88 | 1·90 | 6·25 | 3·27 | 4·02 | 16·22 | 51·46 | 1·24 | 0·17 |

| No. | N. in Leather. | CaO. | $SO_3$ | Total Tannin. | N. in Dry Ash-Free Leather Substance. | Product Number. | Tanning Number. | Specific Gravity with Normal Percentage of $H_2O$. | Thickness. | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Minimum. | Maximum. |
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | | | | Mm. | Mm. |
| 28 | 7·40 | .. | .. | 35·88 | 10·47 | 2·41 | 70·0 | .. | 2·80 | 6·75 |
| 41 | 6·86 | 0·072 | 0·246 | 38·09 | 9·54 | 2·69 | 86·5 | .. | 3·20 | 5·63 |
| 46 | 6·19 | 0·068 | 0·098 | 49·49 | 8·92 | 2·87 | 99·6 | 1·065 | 5·20 | 5·56 |
| 49 | 6·69 | 0·089 | 0·126 | 36·30 | 10·21 | 2·65 | 71·3 | .. | 5·22 | 5·78 |
| 78 | 7·06 | 0·244 | 0·035 | 32·89 | 10·78 | 2·52 | 65·1 | .. | 5·30 | 6·40 |
| 79 | 7·33 | 0·184 | 0·123 | 34·82 | 10·20 | 2·43 | 74·5 | 1·053 | 4·20 | 5·40 |
| 105 | 7·49 | 0·152 | 0·188 | 33·55 | 10·63 | 2·38 | 67·5 | .. | 3·28 | 4·30 |
| 108 | 6·98 | 0·117 | 0·148 | 37·87 | 10·15 | 2·55 | 73·4 | .. | 4·19 | 5·64 |
| 119 | 6·27 | 0·259 | 0·289 | 33·99 | 10·55 | 2·84 | 68·7 | .. | 2·28 | 3·36 |
| 143 | 7·11 | .. | .. | 31·81 | 10·97 | 2·50 | 62·3 | 0·992 | 4·68 | 5·40 |
| 151 | 5·78 | 0·149 | 0·193 | 23·53 | 10·56 | 3·08 | 68·6 | 0·985 | 4·15 | 6·50 |
| 152 | 7·18 | 0·043 | 0·035 | 33·25 | 10·38 | 2·48 | 71·5 | 0·942 | 3·60 | 4·80 |
| 153 | 8·02 | .. | .. | 33·15 | 10·36 | 2·22 | 71·8 | .. | 2·70 | 4·16 |
| 163 | 8·10 | 0·088 | 0·123 | 35·14 | 10·62 | 2·20 | 67·6 | 0·714 | 3·50 | 4·70 |
| 168 | 8·26 | .. | .. | 33·55 | 10·75 | 2·16 | 65·6 | .. | .. | .. |
| 174 | 6·92 | .. | .. | 26·36 | 11·24 | 2·57 | 58·4 | .. | 1·10 | 2·20 |
| *174 | 8·33 | .. | .. | 31·74 | 11·24 | 2·14 | 58·4 | .. | .. | .. |
| 192 | 6·28 | .. | .. | 27·41 | 10·55 | 2·84 | 68·7 | .. | .. | .. |
| *192 | 7·90 | .. | .. | 34·53 | 10·55 | 2·25 | 68·7 | .. | .. | .. |
| 200 | 6·85 | .. | .. | 27·00 | 11·04 | 2·60 | 61·2 | .. | .. | .. |
| *200 | 8·28 | .. | .. | 32·64 | 11·04 | 2·15 | 61·2 | .. | .. | .. |
| 202 | 7·46 | .. | .. | 27·97 | 11·50 | 2·39 | 54·8 | .. | .. | .. |
| *202 | 8·52 | .. | .. | 31·93 | 11·50 | 2·09 | 54·8 | .. | .. | .. |
| 211 | 8·93 | .. | .. | 26·61 | 12·03 | 1·99 | 48·0 | .. | .. | .. |
| *211 | 9·34 | .. | .. | 27·73 | 12·03 | 1·91 | 48·0 | .. | .. | .. |
| 228 | 6·34 | 0·173 | 0·568 | 23·54 | 10·90 | 2·81 | 63·3 | .. | 4·50 | 5·33 |
| 233 | 5·42 | .. | .. | 25·93 | 10·40 | 3·29 | 71·2 | 1·027 | 3·80 | 5·20 |
| 243 | 8·80 | .. | .. | 19·49 | 13·00 | 1·94 | 31·5 | .. | 1·06 | 1·34 |

—J. T. W.

*Wool-grease, Application of, in the Leather Industry.* F. Veselý. Časopis pro prumysl chemický, 1897, **7**, 313.

Various samples of leather were stuffed in a drum with mixtures of wool-grease and tallow. The results show that the neutral wool-greases penetrate the leather better than other fats of the same consistence, that they leave no sticky touch or ill odour, and that in the case of chrome-tanned leathers especially, they give a very good colour.

—J. T. D.

*Leather, Currying of.* Der Gerber, 1898, **24**, [560] and [561].

*Table Stuffing.*—After treatment of the leather on the grain side, the goods are then covered on the flesh side, with the prepared grease brushed on with a stiff, long-haired brush. The grease is laid on thicker on the stouter parts of the leather than on the thinner portions, after which the piece is hung up in a room heated to between 25° and 30° C. As the moisture dries out of the leather, the grease rapidly penetrates. This, however, does not take place uniformly unless the grease has been properly prepared, as described in the earlier part of this article (this Journal, 1898, 55). The oily constituents only will penetrate, and are liable to go right through the leather, causing dark blotches of grease on the grain surface. Even an increase in the above temperature will not cause the harder fats to penetrate. When the leather is in proper condition and the grease correctly prepared, the leather does not require to hang in the drying room for more than from 24—48 hours, according to the substance of the skin. When sufficiently dry, it is laid away in pile, one piece above the other, laid flesh to flesh. The piles are turned at intervals to prevent the generation of too much heat. By this process the grease unites with the individual fibres of the leather, combining with and coating them, thus rendering them softer and more pliable, the resulting leather being milder. When the desired mildness has been obtained, the excess of grease is removed. They are then ready for the warehouse. If mineral oils are used in the currying, no advantage is gained by laying in pile. The heating which is caused when in pile is not due to the fermentation of the oil, as is sometimes supposed, but due to fermentation in the leather itself, and in part to oxidation of the animal fats used. The advantages claimed for "table stuffing" are, better appearance, milder feel, and firmer leather. Should the excess of grease, removed by skiving (table grease), come off in cakes, it indicates that too much stearin has been used in the mixture.

*Drum Stuffing.*—This method of currying leather is generally only used for the commoner article, and when it is desired to get large quantities of grease into the fibre. Drum stuffing originated in America, and has the advantage that large quantities may be stuffed at a time; it appears easier, requires less labour, and cheaper fats may be used. The principle of the process is that the fats are driven into the leather by mechanical force, and equally distributed. Good results, however, depend on the amount of moisture contained in the leather, which must be perfectly evenly distributed; the thick and thin portions of the hides must be equally moist, but must contain less moisture than by the "table stuffing" process. If the leather is too wet, the grease will not penetrate; if, on the other hand, too dry, the leather is permeated with grease which will not combine, and on drying will come on the surface. It requires a great deal of care and practice to get the leather into the exact state suitable for drumming.

In "drum stuffing" of leather, when the softer fats are used, the ordinary temperature of the curriers' workroom is sufficient for the process, as the friction caused by the rotation of the drum always increases the temperature inside it, bringing this to about 20° C. When the harder fats are employed, it is necessary to maintain a temperature higher than this—a temperature sufficient to keep the fat in thin liquid state. This temperature may be secured by warming the drum; by blowing steam into it; by putting in the fat hot; by passing a constant stream of hot air through the drum; or, lastly, by making the drum double-cased, and injecting steam or hot air into the casing. Blowing steam into the drum is perhaps the simplest of these methods, but it is somewhat dangerous, as direct contact of the steam with the leather sometimes causes blistering. Blistering may also occur in double-case drums, but is less likely. The best method is to cause a current of hot air to enter the drum (it is usually made to enter through the axle), and to pass out through a suitable valve at the side of the drum. This mode of warming is largely used in stuffing "crown leather," as there can be regulation to any temperature; and is specially advantageous, in that it draws moisture from the leather and allows the fat to penetrate more thoroughly. An objection to "drum stuffing" is that the hard fats, in cooling, crystallise out on the surface of the leather; on the other hand, drum-stuffed leather rarely 'spews." "Spewing" is rare on waxed goods, whilst it is common on iron-blacked grain-leathers.

*Currying with Hot Fats* ("Einbrennen").—The purpose of this process is to get hard fats, &c., into the leather, such as stearine or beef tallow, or also paraffin, either singly or mixed, in large quantities. The fats, &c., first melted, and often heated to 100°—150° C., are put into the drum; the leather, which must be *perfectly dry*, is then introduced into the melted substance, and drummed for varying periods according to thickness or requirement, the drum being kept hot either by hot air or otherwise if double cased. No real combination takes place here of the leather-fibre with the fat or paraffin, the cavities between the fibres merely becoming filled; and when cold, the fat crystallises out. This may in part be remedied by a steeping in water and redrumming. The crystals are thus broken up, and driven, in a finer state of division, further into the leather between the fibres. Still further improvement is effected if the leather be brushed on the grain with wool-fat and laid in pile—by which crystallising out is partly prevented.

Japan tallow has been recently used for currying leather, with excellent results. It is obtained by boiling the fruit or berries of the "Rhus Succedanea," which grows in Japan and California. The tallow (for it is not a wax) is of a light yellowish-green colour; melts between 51°—53° C.; sp. gr. 0·993; and emulsifies well with water, this last characteristic being very important. It has been found to give such good results that the Swiss Government stipulates that in the curried leather for army contracts, only this material may be used. It has, however, this disadvantage: it throws out minute dust-like crystals of palmitic acid on the surface of the leather. A remedy for this is to treat the grain, after drumming, with a coating of "moëllon."

The "Einbrennen" process is not only suited for all classes of heavy leather, but for light and thin sorts as well. It gives a feeling to the leather, of firmness and fulness, and a tougher grain; at the same time it increases weight, even up to 35 or 40 per cent.—J. G. P.

*Tanning Processes, Study of the Progress of.* F. Knapp. Chem. Centr. 1897, **2**, [24], 1124. From Festschrift der Tech. Hochschule zur Naturf. Vers. 1897, 155—66, Brunswick.

In the opinion of the author, leather is not a chemical combination of hide and tannin. The fixation of tannin depends only on the surface attraction which the fibre exercises; this coating of the fibres prevents them sticking together on drying, producing soft leather instead of a horny mass. Tanning is not a chemical but a physical process.

In chamois leather, the fibres are coated with an oxidation product of the oil used for dressing.

Tawing depends on the fixation of aluminium salts with gluten of wheat, and albumin and oil from egg yolks; potato flour and salt simply assist the process. Brittleness of leather is caused by the presence of crystalline substances (sulphate of lime, &c.), which coat the fibres and render them brittle. Scientific knowledge of the tanning process has not, however, up to the present enabled many new methods to be introduced, chrome tanning being the only one which has stood the practical test.—J. T. W.

*Suède Glove Leather, The Dyeing of.* Der Gerber, 1898, **24**, [560] and [561].

*See under* VI., *page* 150.

### PATENTS.

*Tanning and Pickling, Impts. in, and Machinery and Apparatus for same.* A. Stagg and L. Seager, Milton-next-Sittingbourne. Eng. Pat. 4700, Feb. 22, 1897.

The hides, skins, flesh, and the like are treated with "tanning or chemicals" in a cylinder *in vacuo*.—J. T. W.

*Leather Pulp, Impts. in or relating to the Manufacture of Boards, Plates, or Sheets from.* G. Brigalant, Barentin (Seine-Inférieure). By A. J. Boult, London. Eng. Pat. 24,083, Oct. 19, 1897.

An ordinary paper-making machine is not suitable for manufacturing thick leather sheets from pulp; a special machine is employed with an arrangement for regulating the distance between the two rollers as the layer or pulp increases in thickness. The mechanical arrangement for doing this consists in a weight placed on a pressure lever being automatically moved at the right time. The mode of doing this cannot well be explained without reference to the drawing attached.—J. T. W.

*Leather, Impts. in the Preparation of.* H. Streich, Bonn. Eng. Pat. 25,008, Oct. 28, 1897.

The hides, after being unhaired by sweating, are partially tanned for one or two days. They are then stretched in a frame and the flesh side rubbed with a mixture of 50 grms. of sugar in 15 grms. of vinegar and 15 grms. of ammonia. The hide is allowed to become half dry, and the prepared side is coated with turpentine or copal varnish, and then thoroughly dried.

Hides thus prepared are stronger and more capable of resisting outer influences than leather tanned in the usual way; they are also lighter in weight.—J. T. W.

*Depilating Hides or Skins, Impts. in the Process of.* H. Holmes, Philadelphia, U.S.A. Eng. Pat. 26,760, Nov. 16, 1897.

The hides are subjected to a bath of sulphide of sodium solution of 2°—6° B. in a paddle for a short time in movement, then allowed to stand for 10—12 hours. They are again paddled while the solution is gradually diluted with water until the skins are free of "caustic matter."

—J. T. W.

*Whalebone, Process for Regenerating Used, and for Dressing Fin Whalebone without rendering the same Brittle.* M. Weishaus, Berlin. Eng. Pat. 27,661, Nov. 24, 1897.

Bent strips of whalebone are coated with a suitable acetate, *e.g.*, acetate of aluminium, then ironed with an ordinary iron, and whilst being stretched are cooled in water.—J. T. W.

*Product ["Lactoform"] derived from Casein or other Albuminous Substances, A New or Improved, and Processes for Producing the same.* W. Krische, Hanover, and A. Spitteler, Munich. Eng. Pat. 24,742, Oct. 26, 1897.

This consists essentially of casein precipitated by suitable metallic salts, and subsequently hardened by treatment with formaldehyde. The inventors find, however, that the insoluble product yielded by the direct action of formaldehyde on a casein solution, after being dried, swells in water and becomes quite soft and incapable of resisting mechanical influences; whereas if a similar substance is prepared in the manner indicated, it is very hard, elastic, and tough, and may be kept immersed in water for days without being injured.

According as the "lactoform" is to be employed in place of horn, ivory, ebony, amber, &c., it is precipitated alone, in presence of pigments and (or) ground cork, or simultaneously with white lead, barium sulphate, alumina, &c. The coagulated mass is ground, mixed with sufficient water, moulded into shape, drained till it becomes slightly but uniformly transparent, placed all night in a 20 per cent. solution of formalin, and finally dried under gentle pressure to avoid warping.

Walls may be rendered damp-proof by "lactoform" if they are painted twice with a 15 per cent. solution of casein and a 10 per cent. solution of zinc chloride, followed by the application of strong formaldehyde.—F. H. L.

## XV.—MANURES, Etc.

*Basic Slag, A Remarkable Observation on.* M. Schmoeger. Landw. Versuchs. Stat. 1897, **47**, 413.

In accordance with observations made by Hoyermann, it was found that, after fusion with silica, the citrate solubility of the phosphoric acid in basic slag was considerably increased. The first sample examined, contained 20·19 per cent. of phosphoric acid, of which 48·1 per cent. dissolved in Wagner's citrate solution. After fusion with silica, 83·5 per cent. of the phosphoric acid was soluble in citrate solution. When fused alone, and afterwards finely ground, only 20·1 per cent. of the phosphoric acid was soluble. It has been suggested that the apparently diminished citrate solubility of basic slag, after ignition, was due to the production of calcium manganate, and the consequent partial destruction of the citric acid. In the author's experiment the slag did increase in weight during ignition, but no manganate was produced.

Two other samples of slag were fused, both alone and with silica. In both cases the citrate solubility was very greatly increased (from 41·4 and 58·2 to 97·2 and 96·6 per cent. respectively) under the influence of silica; but the solubility was not, as in the first experiment, diminished by fusion without silica (owing, perhaps, to the presence of some siliceous material); and there was a loss, instead of a gain, in weight.—N. H. J. M.

*Chili Nitrate of Soda, Injurious Effect of.* P. Wagner. Landw. Presse, 1897, Nos. 18 and 19. (See also this Journal, 1897, 342.)

The results of experiments made at Darmstadt showed that perchlorate occurs as an impurity in nitrate of soda to the extent of 0·14 to 1·65 per cent. The large amounts, 3 and 7 per cent., found by Sjollema (this Journal, 1897, 152 and 163) are probably quite exceptional.

Whilst Sjollema concluded that an application of 200 kilos. per hectare of nitrate of soda, containing 0·5 per cent. of perchlorate, would be injurious, Wagner has ascertained that, in ordinary practice, nitrate of soda, containing as much as 1·65 per cent. of perchlorate, was found to be without injurious effect.

With regard to the effect of nitrate of soda itself on vegetation, a number of experiments were made in which oats were grown in pots, and manured with amounts of nitrate corresponding with 20 cwt. to 120 cwt. per hectare. The greatest yield of straw was obtained with 80 cwt. per hectare, the yield being very slightly less with 100 and 120 cwt.; the greatest yield of grain was obtained with the greatest application of nitrate (120 cwt. per hectare). The results show that oats will bear a very heavy dressing of nitrate under the conditions of the experiments. Nitrate of soda should be applied as uniformly as possible, and only after being finely ground.—N. H. J. M.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar Cane, The Principal Amide of.* E. C. Shorey. J. Amer. Chem. Soc. 1897, **19**, 881—889.

By precipitating cane juice in the usual manner with mercuric nitrate, the author isolated a crystalline optically inactive substance, which did not reduce Fehling's solution, and liberated ammonia only in concentrated alkaline solution with formation of hydrogen cyanide. These properties are possessed by glycocol, and it is concluded that this compound is the principal amide present, Maxwell's statement (Louisiana Sugar Planters' Association, January 1894) that the amide is asparagin being incorrect.—J. L. B.

*Cane-Sugar Molasses, Analysis of.* H. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 6983.

*See under* XXIII., *page* 187.

*Beet Sugar, The Extraction of.* F. Breyer. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 633—635. (From Oest. ung. Zeits. 1897, [2].)

By his interesting microscopical researches on the colouring matter of the beetroot, the author has discovered in the cellular tissue of the beetroot, globules in the form of discs, with a depression in the middle, similar to red blood corpuscles, but of uniform size and of a very deep colour. They are so numerous that they are estimated at 10 per cent. of the total suspended matter of the juice. The longer axis measures 0·9 μ, and the smaller 0·6 μ. They are smaller than the micrococcus of acute pneumonia, than the typhoid bacillus, and much smaller than that of cholera. They are almost the same size as the bacillus of influenza, but larger than the micrococcus prodigiosus. The latter, as is known, develops in 48 hours on cooked potato a thick reddish-purple layer, the colour of which disappears to the eye under a magnifying power of 1,500 to 2,000; the ovoid corpuscles of the beet, however, preserve their deep black colour even under the highest powers of the microscope; the globules may therefore be considered as forming the colouring matter of the juice. Other reasons favour this idea.

The corpuscles grow between the walls of the cellular tissue of the beetroot similarly to the bacilli of cholera and of tuberculosis, and form chains of five or six individuals, which break up, giving isolated individuals to the number of 600 or 700 billions in a cubic centimetre. They are also found inside the cells. Like many micro-organisms, they have a movement of their own, not affected by light. It would be interesting to find if these corpuscles are composed of protoplasm, of albuminoids, or of chlorophyll. Meantime, Breyer has succeeded, by his asbestos filter, in separating the corpuscles from the juice, and in microscopical preparations he finds that, heated in presence of lime, they swell up and dissolve. This solution is likewise produced on heating the juice in the absence of lime, but not in the cold.

This statement is evidently of enormous importance, showing that the quantity of lime used in carbonation should be diminished as much as possible.

The colouring matter of the beetroot is thus contained in the form of a very concentrated solution in these micro-organisms, which form a great part of the non-sugar. They are motionless in the interior of the cells of the beet, but have a very active movement outside them. The colouring matter is easily soluble in hot water without deforming the micro organisms which contain it; in cold water it only dissolves after 24 hours and in very small quantity. If the micro-organisms with their colour are removed by filtration, the beetroot juice is found to have entirely lost its characteristic smell. The micro-organisms are more resistant to alkalis than the other organic compounds, a faint alkalinity causing them in a short time to agglomerate into colloidal clots with a slight deformation, these remaining suspended in the juice. If the action of the alkali is more intense, they become entirely deformed, and give a vegetable gum, which may constitute one of the principal constituents of molasses. These properties lead to the conclusion that the quantity of lime hitherto employed is too high and should be greatly diminished, and that the juice should be treated by a special cold process.—L. J. de W.

*Beet Sugar Juices and Syrups, Loss of Alkalinity in.* E. Urbain. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 626—630.

In a former note (this Journal, 1897, 690) it was pointed out that, as manufacture proceeds, and in consequence of the storage of the beetroots in silos, there was a diminution of the saccharine content and an increase of reducing sugars. Moreover, the amount of nitrogen was shown to remain sensibly constant, the proportion of the nitrogenous constituents undergoing a relative variation from a continual transformation of albuminoids and nitrates into amides and ammoniacal salts.

These experiments have been repeated during the last campaign, merely modifying the estimation of albuminoid nitrogen by precipitating it with cupric hydrate. The analyses confirm the first results:—

| —— | Oct. 15th. | Nov. 1st. | Nov. 15th. | Dec. 1st. |
|---|---|---|---|---|
| Sugar per 100 c.c. ........ | 17·85 | 18·12 | 17·12 | 16·78 |
| Reducing bodies ........ | 0·007 | 0·007 | 0·024 | 0·10 |
| Total nitrogen ......... | 0·198 | 0·202 | 0·196 | 0·199 |
| Albuminoid nitrogen per cent. of total N. | 35·50 | 37·12 | 32·61 | 30·05 |
| Nitrogen of nitrates per cent. of total N. | 22·10 | 19·18 | 18·56 | 14·57 |
| Nitrogen of amides and ammonium salts per cent. of total N. | 42·40 | 43·10 | 48·80 | 55·38 |

It is known, from the researches of Strohmer, that the loss of saccharine of the beetroot during the period of preservation is principally due to the respiration of the plant. Garreau, in 1851, and recently Pallarin, showed that plants respire more energetically in proportion as they are richer in proteids. The author is therefore induced to recognise a relation between respiration, the phenomena of the transformation of albuminoid matters and nitrates into amides and ammonium salts.

An indirect proof of the conversion of nitrogenous substances in the beetroot is furnished by the difficulties found in the manufacture, and is due to a loss of alkalinity during concentration, this loss becoming more important as manufacture proceeds.

On calculating the figures obtained from the first carbonation juice to the same density as the original diffusion juice, there is found for this first phase of purification, an elimination of nitrogen sensibly equal to the quantity of albuminoid nitrogen, and the loss of alkalinity is almost exclusively due to disengagement of ammonia. The quantity of nitrogen eliminated during evaporation, increases constantly, and very rapidly at the end of the manufacture in consequence of the increase in amides and ammoniacal salts.—L. J. de W.

*Sugar in Alcoholic Solutions, Table of the Solubility of.* H. and L. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 630–631.

*Temperature, 15°—16° C.*

| Alcohol per 100 c.c. | Sugar. | | Density. | Alcohol per 100 c.c. | Sugar. | | Density. | Alcohol per 100 c.c. | Sugar. | | Density. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Per Cent. | Per 100 c.c. | | | Per Cent. | Per 100 c.c. | | | Per Cent. | Per 100 c.c. | |
| 0 | 66·40 | 88·2 | 1·329 | 34 | 55·0 | 67·2 | .. | 68 | 24·0 | 23·8 | .. |
| 1 | 66·15 | 87·9 | .. | 35 | 54·5 | 66·3 | .. | 69 | 22·5 | 22·2 | .. |
| 2 | 65·90 | 87·4 | .. | 36 | 54·0 | 65·4 | .. | 70 | 21·0 | 20·6 | 0·982 |
| 3 | 65·70 | 86·9 | .. | 37 | 53·4 | 64·5 | .. | 71 | 19·5 | 19·0 | .. |
| 4 | 65·50 | 86·4 | .. | 38 | 52·8 | 63·6 | .. | 72 | 17·9 | 17·5 | .. |
| 5 | 65·25 | 85·9 | .. | 39 | 52·2 | 62·7 | .. | 73 | 16·4 | 16·1 | .. |
| 6 | 65·00 | 85·4 | .. | 49 | 51·6 | 61·8 | 1·198 | 74 | 15·0 | 14·7 | .. |
| 7 | 64·75 | 84·9 | .. | 41 | 51·0 | 60·8 | .. | 75 | 13·7 | 13·3 | .. |
| 8 | 64·50 | 84·4 | .. | 42 | 50·3 | 59·8 | .. | 76 | 12·5 | 11·9 | .. |
| 9 | 64·25 | 83·9 | .. | 43 | 49·7 | 58·7 | .. | 77 | 11·3 | 10·5 | .. |
| 10 | 64·00 | 83·4 | 1·303 | 44 | 49·0 | 57·6 | .. | 78 | 10·1 | 9·3 | .. |
| 11 | 63·75 | 82·9 | .. | 45 | 48·25 | 56·5 | .. | 79 | 9·0 | 8·2 | .. |
| 12 | 63·50 | 82·5 | .. | 46 | 47·5 | 55·3 | .. | 80 | 8·0 | 7·2 | 0·898 |
| 13 | 63·25 | 82·0 | .. | 47 | 46·7 | 54·1 | .. | 81 | 7·0 | 6·2 | .. |
| 14 | 63·00 | 81·5 | .. | 48 | 45·8 | 52·8 | .. | 82 | 6·0 | 5·3 | .. |
| 15 | 62·70 | 81·0 | .. | 49 | 44·9 | 51·5 | .. | 83 | 5·0 | 4·4 | .. |
| 16 | 62·40 | 80·5 | .. | 50 | 44·0 | 50·2 | 1·141 | 84 | 4·1 | 3·5 | .. |
| 17 | 62·10 | 80·0 | .. | 51 | 43·1 | 48·9 | .. | 85 | 3·3 | 2·8 | .. |
| 18 | 61·80 | 79·5 | .. | 52 | 42·2 | 47·6 | .. | 86 | 2·7 | 2·2 | .. |
| 19 | 61·50 | 79·0 | .. | 53 | 41·3 | 46·3 | .. | 87 | 2·1 | 1·7 | .. |
| 20 | 61·20 | 78·4 | 1·277 | 54 | 40·3 | 45·0 | .. | 88 | 1·6 | 1·3 | .. |
| 21 | 60·90 | 77·8 | .. | 55 | 39·3 | 43·6 | .. | 89 | 1·2 | 0·98 | .. |
| 22 | 60·60 | 77·2 | .. | 56 | 38·3 | 42·2 | .. | 90 | 0·95 | 0·77 | 0·810 |
| 23 | 60·25 | 76·5 | .. | 57 | 37·3 | 40·8 | .. | 91 | 0·7 | 0·56 | .. |
| 24 | 59·85 | 75·8 | .. | 58 | 36·3 | 39·4 | .. | 92 | 0·5 | 0·40 | .. |
| 25 | 59·45 | 75·1 | .. | 59 | 35·3 | 37·9 | .. | 93 | 0·35 | 0·30 | .. |
| 26 | 59·00 | 74·3 | .. | 60 | 34·3 | 36·4 | 1·062 | 94 | 0·2 | 0·22 | .. |
| 27 | 58·50 | 73·5 | .. | 61 | 33·2 | 34·9 | .. | 95 | 0·15 | 0·15 | .. |
| 28 | 58·00 | 72·6 | .. | 62 | 32·1 | 33·4 | .. | 96 | 0·1 | 0·10 | .. |
| 29 | 57·50 | 71·7 | .. | 63 | 30·9 | 31·8 | .. | 97 | 0·07 | 0·07 | .. |
| 30 | 57·00 | 70·8 | 1·243 | 64 | 29·7 | 30·2 | .. | 98 | 0·03 | 0·04 | .. |
| 31 | 56·50 | 69·9 | .. | 65 | 28·4 | 28·6 | .. | 89 | 0·01 | 0·02 | .. |
| 32 | 56·00 | 69·0 | .. | 66 | 27·0 | 27·0 | 1·000 | 100 | 0·00 | 0·00 | 0·794 |
| 33 | 55·50 | 68·1 | .. | 67 | 25·6 | 25·4 | .. | | | | |

As regards temperature, taking the tables of Flourens, Herzfeld, Schrefeld, and Scheibler as a basis, the following table may be drawn up:—

| Alcohol per 100 c.c. | Sugar per 100 c.c. | | | | |
|---|---|---|---|---|---|
| | 0° C. | 10° C. | 20° C. | 30° C. | 40° C. |
| 0·0 | 85·5 | 87·3 | 88·8 | 91·8 | 94·2 |
| 10·0 | 80·7 | 82·5 | 85·0 | 87·0 | 90·0 |
| 20·0 | 74·2 | 76·5 | 79·0 | 82·0 | 84·0 |
| 30·0 | 65·5 | 68·0 | 71·0 | 74·0 | 77·5 |
| 40·0 | 56·7 | 59·0 | 62·0 | 65·5 | 69·0 |
| 50·0 | 45·9 | 48·5 | 52·0 | 55·5 | 60·0 |
| 60·0 | 32·9 | 35·5 | 39·0 | 42·5 | 47·5 |
| 70·0 | 18·2 | 20·0 | 22·5 | 26·0 | 31·0 |
| 80·0 | 6·4 | 7·2 | 8·1 | 10·0 | 14·0 |
| 90·0 | 0·7 | 0·85 | 1·1 | 1·6 | 2·3 |
| 97·4 | 0·08 | 0·15 | 0·25 | 0·36 | 0·5 |

This table, being based on other different tables not in absolute agreement among themselves at all points, is only approximate, and is given merely to show the sense of the influence of temperature on the solubility of sugar in alcohol at different temperatures and of different strengths.
—L. J. de W.

*Invert Sugar of Beetroot Juice and of Corresponding Diffusion Juice.* W. Dombrowski. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 654. (From Zapiski.)

The reducing sugar was titrated by Violette's solution, and verified gravimetrically. The tests commenced from the second half of November 1896, and were made on beets from silos, containing a sensible amount of invert sugar, with the object of finding the most favourable temperature for obtaining diffusion juice with a minimum of invert sugar. The results show that the invert sugar is found, in part, in the last seven diffusers; neither the weak juice nor the exhausted pulp contained any reducing sugar.

The mean of 30 comparative determinations is as follows:—Normal juice, 0·38 per cent.; diffusion juice, 0·17 per cent.; juice obtained, 120·00 per cent.

The diffusion juice therefore contained 0·17 × 1·2 = 0·204 per cent. of invert sugar, while the beetroot juice from which it was derived contained 0·38 × 0·9 = 0·342 per cent. There is thus a loss of 40 per cent. of the original reducing sugars of the beetroot. The other portion probably suffered oxidation in the battery, although the true cause of this oxidation could not be determined. The diffusion juice showed no bacteria under the microscope.—L. J. de W.

*Sugars, Action of Alkalis on.* C. A. Lobry de Bruyn and W. Alberda van Ekenstein. Zeits. Rübenzucker Ind. 1897, **46**, 1023—1041.

The authors have extended their observations on the transformation of sugars by alkalis (this Journal, 1896, 663) to the formation of other new substances, by studying the action of alkalis on galactose, and of lead hydroxide on glucose, fructose, and galactose. The most general case is where, by the action of potash or soda on one of the above sugars, four new sugars, exclusive of acids, are formed.

The solution contains a mixture of five sugars. Two (or rather three) are ketoses, being easily destroyed by dilute hydrochloric acid, and giving an intense reddish-violet colour with hydrochloric acid and resorcinol (Seliwanoff's reaction). In this way, besides *talose*, galactose gives rise to two new crystallisable ketoses, one of which, *tagatose*, occupies the same position in relation to the above-named aldoses that fructose does with regard to glucose and mannose. Tagatose gives a galactosazone. Another ketose, *pseudo-tagatose* (*ψ-tagatose*), although of similar composition, gives an osazone differing from galactosazone. Finally, a fourth reducing sugar, *galtose*, is formed, which gives a distinct osazone. Hitherto it has only been prepared in the form of a syrup. The three new sugars are not fermentable with Delft beer yeast. Romijn has shown that both tagatoses, like fructose, are very slightly, or not at all, acted on by iodine.

From ordinary glucose there is formed, in addition to fructose and mannose, a new ketose, *pseudo-fructose* (*ψ*-fructose); this could not, however, be obtained in a pure state. Another reducing substance, unfermentable, having the properties of a ketose, for which the name of *glutose* is proposed, is also formed. This substance may be easily obtained from commercial fructose by lead hydroxide, or from invert sugar and lime.

Attention is drawn to the part played by glutose in the sugar manufacture. The authors have shown that it is present in colonial molasses, a circumstance to be explained, as is the presence of mannose, by the action of lime on the glucose of the cane juice during the boiling. Its presence may account for the indeterminate losses in distilleries using molasses (this Journal, 1898, 59). ψ fructose and glutose give osazones differing from one another and from glucosazone. Glutose bears the same relation to galtose that ψ-fructose does to ψ-tagatose.

The quantity of glutose contained in cane molasses may be approximately determined by allowing it to ferment completely, and then finding the reducing power towards Fehling solution before and after destruction of glutose by Sieben-Damüller's method. The difference between the two determinations represents almost exactly the glutose present, its reducing power being assumed at half that of glucose.

By this method the authors found in a cane molasses from Louisiana, about 1·8 per cent. of glutose.

Prinsen Geerligs, in an article on the formation and presence of levulose in the products of cane sugar factories (this Journal, 1897, 688), is in error in stating that an aqueous solution of sodium acetate is neutral. The authors have already shown that this salt has a transforming action, and this is not surprising, for in aqueous solution, in consequence of hydrolytic dissociation, it acts as a feeble base. They also did not employ bases in excess, as he states; very small quantities suffice to cause intramolecular transformation of the sugars. The action of 0·1 to 0·2 per cent. of lime, added to a boiling dilute solution of glucose, is about 25 times greater than that of a 10 to 20 per cent. sodium acetate solution, for the same concentration and time of heating. In opposition to Prinsen Geerligs, they found that sodium acetate forms organic acids. In one experiment the reducing power fell to 83·3 per cent., and $(\alpha)_D$ amounted to + 10 per cent. The action of potassium tartrate is still weaker than that of sodium acetate, as was to be expected. The observation of Prinsen Geerligs, that ordinary molasses contains mannose, confirms that of the authors.

It would be possible to go further into this question, if any particular sample of cane-juice were found to contain glutose, and if this juice, after being boiled with an excess of lime, contained fructose and mannose. Prinsen Geerligs has not yet shown that the salts contained in cane-juice can themselves convert the glucose contained in the juice into mannose and fructose.

From the results obtained on boiling fructose with water, the conclusion may be drawn that water alone does not cause a reciprocal conversion of sugars, or that if this really takes place, acids are formed.—L. J. de W.

### *Vinasse, Carbonised, from a Sugar Factory; Some Rare Ash Constituents in.* E. O. von Lippmann. Ber. 1897, **30**, 3037—3039.

In the course of attempts to refine the carbonised vinasse from a factory in which the recovery of sugar from beetroot molasses is carried out, the author obtained certain muddy residues which contained 0·03 per cent. of lithium, 0·12 per cent. of titanium, and 0·213 per cent. of manganese, calculated on the dry substance. Truchot observed that the ash of beetroots contains 0·01 per cent. of lithium, but this was contradicted by Focke and by Grandeau. Wait has found titanium in the ashes of various plants. Dubrunfaut observed the presence of manganese in the ash of beetroots, and recent observations point to that element as indispensable in vegetable enzymes (compare this Journal, 1897, 692).

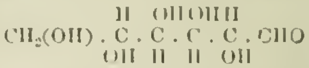
Statements have been made to the effect that the ashes of plants contain strontium, and that this element can partially replace calcium. Beetroot manured with lime containing strontia, contained in 1,000 parts, 0·206 part of strontia, whilst the average amount of lime which they contain is from 0·4 to 0·5 part. The hay from clover grown on soil containing strontia, showed 13·2 parts per 1,000 of strontia, whilst the normal lime content is 20 to 24 parts per 1,000. This hay can be consumed by animals without danger. The author cites a case of two persons who were attacked by eczema in strontia works; they recovered, however, when no longer working amongst strontium salts.

—J. L. B.

### *Galactose, Configuration of.* A. Wohl and E. List.

Ber. 1897, **30**, 3101—3108.

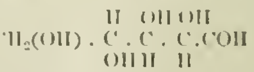
It appeared desirable to the authors to test by an independent method of investigation the correctness of the configuration—



$$\mathrm{CH_2(OH)}\,.\,\overset{\mathrm{H}}{\underset{\mathrm{OH}}{\mathrm{C}}}\,.\,\overset{\mathrm{OH}}{\underset{\mathrm{H}}{\mathrm{C}}}\,.\,\overset{\mathrm{OH}}{\underset{\mathrm{H}}{\mathrm{C}}}\,.\,\overset{\mathrm{H}}{\underset{\mathrm{OH}}{\mathrm{C}}}\,.\,\mathrm{CHO}$$

ascribed to *d*-galactose by Fischer (Ber. **27**, 382).

A sugar of the above structure should be obtained from a pentose—



$$\mathrm{CH_2(OH)}\,.\,\overset{\mathrm{H}}{\underset{\mathrm{OH}}{\mathrm{C}}}\,.\,\overset{\mathrm{OH}}{\underset{\mathrm{H}}{\mathrm{C}}}\,.\,\overset{\mathrm{OH}}{\underset{\mathrm{H}}{\mathrm{C}}}.\mathrm{COH}$$

by addition of HCN and reduction of the corresponding acid lactone. This pentose differs from xylose only in respect to the arrangement in space of the last OH.CH group. It has been obtained from xylose and described under the name "lyxose" by Fischer and Bromberg. The crowning evidence necessary to exhibit the connection between galactose and lyxose—*i.e.*, the conversion of one into the other—was, however, omitted, and is now supplied by the authors. They have employed for the purpose their general method (Ber. **26**, 730) for the conversion of sugars of a higher into those of a lower series. In its application to galactose it involves the following processes:—

1. The preparation of galactosoxime by the action of hydroxylamine on the sugar.
2. Conversion of the oxime into pentacetylgalactonic nitrile.
3. Preparation of the acetamido-derivative of the corresponding pentose by the action of an ammoniacal solution of silver oxide on pentacetyl galactonic nitrile.
4. Decomposition of the resulting acetamido-pentose compound by means of dilute sulphuric acid.

The pentose thus obtained is lævo-rotatory, and was clearly identified with lyxose by the optical and other properties of its derivatives.—H. T. P.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### *Fermentation without Yeast-Cells.* A. Stavenhagen.

Ber. 1897, **30**, 2963.

Referring to Buchner and Rapp's latest communication (this Journal, 1898, 59) on this subject, the author repudiates the suggestion that the entirely negative results obtained by himself were due to the use of deteriorated yeast, yielding an extract inactive before filtration. The extract used possessed fermentative power before, but not *after* filtration through a Kitasato filter.—H. T. P.

*Fermentation, the Influence of Carbonic Acid on.* H. Lange, Wochenschr. für Brau. 1897, **14**, [46], 593.

Four flasks were taken each containing 1 litre of wort with a strength of 21° B., and 3 grms. of yeast added to each. The flasks were connected together so that the carbon dioxide from the first passed through the fermenting wort in the second, and the carbon dioxide from both these through the wort in the third, and that from all three through the wort in the fourth flask. Between each flask was fitted a wash bottle containing sulphuric acid. The temperature was 77° F. In two days the amount of carbon dioxide lost was 58·4 grms. from flask 1; 59·6 grms. from flask 2; 60·7 grms. from flask 3; 62·3 grms. from flask 4. The weight of yeast in each flask was found to be practically the same.—A. L. S.

*Zymase, Remarks on Buchner's Communications on.* R. Neumeister. Ber. 1897, **30**, [19], 2963—2966.

The author considers zymase to be essentially different from an ordinary enzyme; on account of its complicated function, its striking unstability in air, whilst it can be kept for a long time *in vacuo* without losing its activity and its inability to withstand a temperature of 22° C. He considers zymase to be a mixture of several proteid matters, and the decomposition of the sugar to be produced by some complicated interaction.

Buchner ascribes the ready loss of activity of the yeast sap to the action of a proteolytic enzyme, as he found that the addition of a little trypsin or pancreatin quickly destroyed its activity. The author, however, points out that Hjort (Centrbl. für Physiol., 1896, 1892) failed to find any such proteolytic ferment in yeast plasma.—A. L. S.

*Sorbose, Transformation of Sorbitol into, by Mycoderma vini.* A. Matrot. Comptes Rend. 1897, **125**, 874—876.

A number of samples of sorb juice [sorbin from berries of Mountain Ash, or Service tree] were exposed for a few days, and the sugar determined. One sample, which had been kept at 25°—35°, was found to contain a considerable amount of sugar. The surface of this sample was covered with a ferment, apparently identical with *mycoderma vini*, together with bacteria. When sterilised juice was inoculated with the bacteria, the latter were found to be unable to produce sugar.

The sugar obtained from sorb juice was identified as sorbose. It is prepared as follows:—The juice is first subjected to alcoholic fermentation, and is then placed in thin layers in photographic dishes, inoculated with *mycoderma vini*, and kept at 30° C. It is tested from time to time, with Fehling's solution, and when the oxidation is found to be completed, treated with lead acetate. The excess of lead is precipitated as sulphate, and the filtered liquid evaporated on a water-bath until syrupy. On cooling, the sorbose crystallises out, and is purified by recrystallisation. The rate of oxidation depends on the surface exposed; with a surface of 3 square decimeters per litre, there is an increase of about 10 grms. per day.

In preparing sorbose from pure sorbitol, the latter is dissolved in wine or in an artificial nutritive solution. If, as sometimes happens, the ferment will not grow in the artificial solutions, it is first cultivated on wine. As soon as the surface of the wine is covered with the ferment, the wine is poured away from under the ferment, and replaced by the solution of sorbitol. (See also this Journal, 1898, 78.)—N. H. J. M.

*Cellulose, Fermentation of.* V. Omelianski. Comptes Rend. 1897, **125**, [25], 1131—1133. See this Journal, 1898, 60.

An account is given of an experiment on the fermentation of cellulose lasting 13 months by means of a reaction described *loc. cit.* above. 3·4743 grms. of dry cellulose were fermented in the presence of 5·7698 grms. of carbonate of lime which had been gently ignited. The gas (810 c.c.) collected, contained 154·3 c.c. of hydrogen and 659·3 c.c. of carbonic acid. After allowing for carbonic acid remaining dissolved in the liquid and for that evolved by the chalk, it was found that 0·9722 grm. had been obtained from the cellulose. The weight of the fatty acids (acetic and butyric, with a very little valerianic) was 2·2402 grms. The deposit of the fermented culture, composed of flocks of cellulose and spores of bacilli, washed with hydrochloric acid and dried, weighed 0·1272 grm. (3·66 per cent. of the cellulose employed).

The results are then summarised as follows:—

| Cellulose. | Grms. | Products of Fermentation. | Grms. |
|---|---|---|---|
| Cellulose added ....... | 3·4743 | Fatty acids .......... | 2·2402 |
| ,, (?) remaining | 0·1272 | Carbonic acid........ | 0·9722 |
| | | Hydrogen ........... | 0·0138 |
| Cellulose that has disappeared. | 3·3471 | Total ......... | 3·2262 |

The absence of marsh gas among the products of the fermentation is remarkable; the author believes that methane is produced by a particular ferment different from the one he employed in the above experiment, which he has not yet succeeded in isolating in the pure state.—A. C. W.

*Hops and their Storage.* By L. Briant and C. S. Meacham. J. Fed. Inst. of Brewing, 1897, **3**, 481—493.

Among the chief constituents of hops of value to the brewer are the tannin, the oil, and the resins. The resins are the most important. Hayduck has separated three resins, two of which are preservative, and the value of hops to brewers, apart from the aroma is directly measurable by these. The amount of the resins is dependent on the variety of hop, the method of culture, the ripeness, and the treatment during drying. The authors point out that, in general, foreign hops are richer in resins than the English; consequently they are of more value to the brewer. English hops are often picked when unripe and dried too hurriedly in thick layers—conditions which militate against the development of their full preservative value.

Another cause for the unsatisfactory character of English hops arises from the desire of the grower to produce quantity rather than quality. Since the continental system allows for perfect ripening and drying, and avoids any loss occasioned by "hovering" on the kiln, the superiority of such hops is indisputable. The authors are of opinion that if hop growers would remodel their systems of picking and drying they would be able to place hops on the market of nearly or equal value to the imported, whilst their far superior flavour would command higher prices.

It is well known that hops undergo considerable alteration during storage, and it is believed by the authors that the deterioration is due, not to oxidation, but rather to the presence of micro-organisms and enzymes in the hops themselves. From the result of experiments, the authors state that low temperatures will arrest the action of organised and unorganised ferments, and they have found that if hops are deprived of a portion of their normal moisture they may be preserved in a practically unaltered condition at a temperature of 40°—45° F., provided that the atmosphere surrounding them is also dry. This treatment is stated to be practically as effective as the American and German systems of storage at temperatures below 32° F., which is necessarily costly, and in most cases necessitates the defrosting of the hops when taken from the cold store. —J. L. B.

*Barley and Malt, The Phosphoric Acid in, with some Notes on the Presence of Organic Acids in Malt.* C. G. Matthews and G. H. Woolcott. J. Fed. Inst. Brewing, 1898, **4**, 6—20.

Following an account of Prior's work on the phosphoric acid of barley and malt (Chem. und Phys. des Malzes und des Biers) and Fernbach's investigations (this Journal, 1896, 621), on the importance of the soluble phosphates of barley and malt, the authors describe several attempts to determine phosphoric acid existing as soluble phosphates in malt extracts. They finally decided on the following method: 20 c.c. of a 10 per cent. cold-water malt extract were evaporated to dryness in a platinum vessel and the residue incinerated in the presence of pure lime in order to fix the phosphoric acid. The inorganic residue was dissolved in nitric acid, neutralised with ammonia, acetic acid and ammonium acetate added, and the phosphoric acid precipitated with molybdic acid. The phosphoric acid was thus determined in 15 samples of malt (see table), the acidity being estimated at the same time by means of $N\frac{1}{10}$ sodium hydroxide, using rosolic acid as an indicator.

| Samples of Malt marked...... | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. | 9. | 10. | 11. | 12. | 13. | 14. | 15. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Acidity in c.c. $\frac{1}{10}$N soda on 100 grms. malt. | 21 | 20 | 20 | 20 | 19 | 16 | 16 | 16 | 16 | 16 | 12 | 12 | 12 | 10 | 8 |
| $P_2O_5$ per cent. .......... | 0·55 | 0·62 | 0·52 | 0·51 | 0·59 | 0·47 | 0·63 | 0·61 | 0·56 | 0·54 | 0·58 | 0·58 | 0·47 | 0·45 | 0·43 |

These and Fernbach's (*loc. cit.*) figures support the authors' contention as to the relationship between high acidity and high soluble phosphates.

Attention is drawn to the varied results obtained in estimating the acidity of malt worts, &c., when different indicators are used. Thus a solution of disodium hydrogen phosphate reacts strongly alkaline to rosolic acid, faintly alkaline to phenolphthalein, strongly alkaline to methyl orange, and slightly alkaline to litmus. Phenolphthalein is recommended as an indicator for the determination of acidity, as it is practically neutral to secondary phosphates.

The phosphoric acid was next determined in the cold-water malt extract, the hot mash, and the whole malt. It is evident that there is no connection between the acidity and the phosphoric acid on the whole malt. The hot mash gives different results in respect of the amount of phosphoric acid to the cold-water extract. Fernbach's (*loc. cit.*) method does not show the soluble phosphates in their original state in the malt. At the same time, however, his results are perhaps nearer those which would be obtained in actual malt worts.

The authors also estimated the calcium and magnesium in cold and hot water mashes. Comparing the hot mash with the cold, the amount of calcium decreases, whilst the magnesium rises. As it is shown that the amount of phosphoric acid is variable in different malts, it cannot be said definitely how these metals are combined; it is certain, however, the magnesium phosphate tends to remain in solution if not actually increase in amount, whilst the general tendency is for some of the calcium phosphate to be rendered insoluble and to be left behind in the grains.

Inferior malting of fair and good barleys, or the malting of indifferent barleys, leads to an acid malt. A high acidity of the malt is an indication of an undue amount of easily extractable soluble phosphates, and insoluble phosphates rendered soluble in the mashing operation by the higher acidity of the malt wort. A wort is thus produced, not only inferior as regards the qualities of its carbohydrate and albuminoid constituents, but containing an excess of substances which, though useful and necessary to the yeast in normal quantity, may be productive of serious trouble when present in abnormal amount.

To determine whether organic acids were present, the authors extracted malt at the ordinary temperature with alcohol which had been distilled with caustic potash to free it from acid. The alcoholic extract was distilled under reduced pressure and the residue taken up with water, the albuminoids remaining undissolved. The fixed acidity calculated as lactic acid was 0·024 per cent., whilst the volatile acidity, estimated by titrating the spirit distillate, was 0·0054 per cent. as lactic acid. The possible presence of butyric, succinic, and lactic acids was indicated. These organic acids must be mainly present as salts in the malt.

—J. L. B.

### *Yeasts* [*Beer*], *Action of, on Milk.* E. Boulanger. Ann. Inst. Pasteur; La Bière, 5, [11], 161—163.

Eight different yeasts were sown in sterilised skimmed milk, and the cultures kept for 14 months. In appearance the samples varied considerably, some resembling a concentrated bouillon, whilst others were but slightly modified. The Frohberg and Meurant yeasts formed (in six months) a coagulum, which afterwards re-dissolved to a dark, almost limpid liquid, with a considerable deposit of yeast. The first named yeast was found to have become debilitated, but the other appeared well developed and healthy. The Riga X, No. 48, Brussels, and Weihenstephan yeasts, caused the liquid to turn yellow or brown, with a slight deposit of coagulum; the yeasts themselves were well developed. Finally the Loewenbrau and Neunkirchen yeasts produced little alteration beyond a yellow tinge, though themselves well developed. All the cultures were recognised as perfectly pure.

The liquid in each case was examined for its content of lactose, casein in solution and in suspension, mineral matters, and ammonia, the results being given below. The time required by the yeasts to effect the liquefaction of gelatin is appended to facilitate comparison:—

| — | Check Sample. | Frohberg. | Meurant. | Riga X. | 48 Copenhagen. | Brussels. | Weihenstephan. | Lowenbrau | Neunkirchen. |
|---|---|---|---|---|---|---|---|---|---|
| Time (Months) required to liquefy gelatin. | .. | 2 | 2 | 4 | 3 | 3½ | 4½ | over 6 | over 6 |
| | | | | | Grms. per Litre. | | | | |
| Lactose.................. | 50·2 | 47·2 | 48·1 | 48·8 | 49·1 | 49·2 | 50·2 | 49·5 | 49·3 |
| Casein, dissolved......... | 5·2 | 16·2 | 15·8 | 12·1 | 11·9 | 11·5 | 11·1 | 8·8 | 8·2 |
| " in suspension..... | 29·4 | 14·8 | 12·6 | 18·3 | 16·9 | 19·1 | 17·8 | 20·4 | 22·4 |
| Total casein ............. | 34·6 | 31·0 | 28·4 | 30·4 | 28·8 | 30·6 | 28·9 | 29·2 | 30·6 |
| Mineral matter, in solution | 5·0 | 4·1 | 4·2 | 4·7 | 4·6 | 4·3 | 4·6 | 4·8 | 4·9 |
| " " in suspension. | 2·3 | 3·3 | 3·4 | 2·8 | 3·1 | 3·2 | 2·8 | 2·4 | 2·6 |
| Ammonia................ | 7·6 | 24·8 | 52·0 | 33·8 | 16·4 | 29·3 | 17·5 | 38·8 | 29·7 |

From these particulars it is evident that the lactose in the milk is not attacked by the yeast to such an extent or so unequally as is the casein. With respect to the latter, whilst the yeasts most energetic in liquefying gelatin have the greatest power of dissolving casein, their subsequent action on this substance varies. Meurant yeast, for example, being very active in both cases, whereas Frohberg, though powerful as a solvent, exerts but little decomposing influence; Loewenbrau yeast dissolves casein to merely a small extent, but attacks it powerfully; and finally, Neunkirchen acts as the antithesis of Meurant yeast. Since, during the subsequent decomposition of the dissolved casein, the colouring matters, which it forms, are again broken up, depth of colour cannot be taken as a criterion of the activity of the yeast.

The decomposition of casein by yeasts thus resembles the effect produced by casease-forming microbes, though the action is much less rapid.

The figures for insoluble casein given above are somewhat higher than the truth, owing to the difficulty of removing the yeast cells present in the sediment. The same cause also accounts for the variation in the ratio of mineral matters in solution to those in suspension.—C. S.

### *Ales, Cold and Sparkling.* [*Carbonating Beer.*] H. van Laer. J. Fed. Inst. of Brewing, 1898, **4**, 24—29.

The author describes in detail a method of carbonating beers, produced by the vacuum fermentation system, as carried out in an American brewery. The carbonating of Belgian top-fermentation beers has been tried with satisfactory results. Experiments with English ales allow the author to state that if the carbonating process be properly carried out, it gives brilliant beers with a pure taste; when bottled, no deposit appears until many days after the time in which this would appear in the case of the same beers not carbonated.—J. L. B.

### *Brewing, On some Recent Progress in, in the United States.* H. T. Brown. J. Fed. Inst. of Brewing, 1897, **3**, 467—478.

American brewers have recently introduced a combined system of cold storage and carbonation, enabling ales to be cooled without the production of turbidity and flatness. The ale from the racking vessels is run through a refrigerating apparatus known as the "cooling tub," and passes out at a temperature of about 32° F. to the store vats. These are placed in a cellar kept at 32°—35° F., and are provided with apparatus enabling the contents to be forced through a filter. The ale remains in the store vessels at about 32° F. for two to three weeks, and during this time a settlement of the greater part of the remaining yeast takes place; also the resinous matters which would remain in solution at ordinary temperatures are deposited. By means of air pressure of 10 or 15 lb. to the square inch, the ale is next forced through the carbonator and filter. In some breweries filtration precedes carbonation; the author, however, favours the former treatment. The filter most generally used is the "Enzinger," the filter medium consisting of specially-prepared paper pulp, which can be used again after washing and sterilising. The ale leaves the filter at a temperature of 35°—36° F., and is racked into casks whilst under pressure by special racking machines.

Ales subjected to the above treatment will not possess any of the qualities produced by after-fermentation in the cask, and it is suggested by the author that such ales should be stored in vats at 55° F. with the addition of some dry hops until they are matured by a natural process of after-fermentation. They could then be submitted to the previously-described treatment.

Nearly all the lager beer in America is brewed by the infusion process, the boiling of the "thick mash" being practically unknown. Fermentation of lager beer differs but little from the Continental system. The author also describes the Pfaudler vacuum process of fermentation, which is employed at some of the American breweries.
—J. L. B.

### *Cider, "Hardening" of; Influence of Bismuth Subnitrate upon.* L. Dufour and Daniel. Comptes Rend. 1897, **125**, [25], 1125—1128.

Cider, when left exposed to the air, "hardens" or increases in acidity. This may be prevented, or at least considerably diminished, by the addition of bismuth subnitrate. The authors consider the use of 10 grms. per hectolitre to be sufficient for ordinary purposes, larger amounts exercise a greater retarding power on the acetification. The addition of the bismuth salt is followed by an increase in the slow alcoholic fermentation, which takes place when cider is kept.—A. C. W.

### *Wines, Russian; Examination of.* L. J. Wolpjan. Farmazeft, 1897, **5**, 730.

The following are the results of the author's analyses :—

| ——— | | Alcohol per Cent. by Vol. | Specific Gravity. | Total Acid as Tartaric Acid. | Volatile Acid as Acetic Acid. | Extractive in 100 c.c. |
|---|---|---|---|---|---|---|
| Fruit and berry wines | Max. | 16·00 | 0·916 | 1·4820 | 0·2520 | 32·68 |
| | Min. | 5·00 | 0·994 | 0·4125 | 0·0396 | 2·24 |
| Mead and honeyberry wine (Honigbeerenwein). | Max. | 10·60 | 1·575 | 1·1980 | .. | 37·70 |
| | Min. | 4·80 | 1·101 | 1·0405 | .. | 10·69 |
| Crimean grape wine, white | Max. | 12·77 | 0·995 | 0·7875 | 0·1572 | 3·84 |
| | Min. | 10·70 | 0·991 | 0·3825 | 0·0624 | 1·368 |
| " " red | Max. | 16·24 | 0·9952 | 0·7651 | 0·1500 | 2·87 |
| | Min. | 10·10 | 0·9915 | 0·3600 | 0·0600 | 1·63 |
| Caucasian " white | Max. | 16·12 | 0·9930 | 0·7050 | 0·1812 | 2·83 |
| | Min. | 11·60 | 0·9910 | 0·5025 | 0·0768 | 1·48 |
| " " red | Max. | 14·30 | 0·9945 | 0·8700 | 0·1760 | 2 75 |
| | Min. | 10·60 | 0·9930 | 0·5250 | 0·0468 | 1·62 |

The author gives besides the figures of other analysts, showing for the Caucasian wines higher acid and lower alcohol content. Southern Russia, including the Crimea and the Caucasus, as well as Southern Siberia and the Russian possessions in Central Asia, are well adapted for wine growing, and the industry ought to be developed. The fruit and berry wines are of high specific gravity, contain much extractive and much acid: their alcohol content is similar to that of grape wines, but this is often increased by the addition, either of sugar to the must or of alcohol to the finished wine. The honey berry wines are poorer, but improvement in them may be expected, as the bee-keepers are giving the matter attention.—J. T. D.

### *Distillery, Conditions for Obtaining Good Results in a Beetroot.* E. Durin. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 660—666. (From J. de la Dist. française.)

The cossettes (or slices) should be thick and of such a form as to be rigid and not become adherent, and it is important they should be cut and not torn in irregular pieces. Knives provided with ridges give suitable slices. On the perfection of the cossettes depends the regular circulation of the liquid in the macerators, and the complete exhaustion of the pulp.

The beetroots should be washed thoroughly, otherwise not only are the knives rapidly worn out, but the earth contains dangerous ferments, and calcareous matters neutralise a portion of the acid, rendering the degree of acidification uncertain.

The whole of the spirit is not always recovered in the column, but this is rarely due to imperfection in the apparatus. It is important that the hoods should dip exactly at all parts to an equal depth in the liquid on the trays, and deposits should not be allowed to accumulate on these.

An occasional test of the vinasse is not sufficient to show if the whole of the alcohol has been recovered; the operation should be *continuous and automatic*. For this purpose part of the vapour given off by the vinasse leaving the column is passed through a small condenser worm and the alcoholic strength of the condensed liquid is read off. This liquid runs continuously into a small cylinder containing a hydrometer with wide divisions—0° to 7°, for example—and should show 0° at 15° C. If the column is worked by open steam, and the test is drawn from the last segment of the column, a great part of the liquid obtained will be simply condensed steam from the boilers, and the results will be illusory. Besides, the violence of the agitation causes priming of the vinasse and further dilution. If the column is heated by coils, there is no admixture of steam, but priming is still existent. The vapour should be taken outside the column, on the vinasse outlet pipe. As there is a pressure in the column and the boiling point of the vinasse is over 100° C., it will give off vapour if passed through a small chest. It is this vapour, given off quietly, which should be collected to control exhaustion.

The most delicate point, and one least generally regulated, is the preparation and acidification of the juice before fermentation. Great misapprehension prevails as to the role of the sulphuric acid used. It is often supposed to be added to neutralise calcareous matters, to assist maceration, and its action is confused with that of the dangerous and injurious acids formed during fermentation; but its action may be briefly formulated as follows:—

A relatively small quantity of free sulphuric acid arrests fermentation, but a certain proportion is necessary to oppose the life and multiplication of dangerous ferments which transform sugar into organic acids instead of into alcohol. At the present time no definite relation is known between the sugar lost and the organic acids formed. From ½ to 1 part of sulphuric acid per thousand would not only prevent the formation of injurious organic acids, but probably also alcoholic fermentation, whilst 5 or 6 parts per thousand of organic acids may allow an injurious acid fermentation along with an incomplete alcoholic one. The sulphuric acid added, displaces the organic acids present, but a slight excess is necessary to prevent the action of the ferments producing organic acids without paralysing the life and multiplication of the alcoholic ferments. When an acid fermentation has been allowed to develop simultaneously with the alcoholic one, a general acidity is produced, which is considered excessive, and one is disposed to diminish the initial quantity of sulphuric acid. The very contrary should be done; the more acid there is formed during fermentation and the more must the initial dose of sulphuric acid be increased. At the commencement, macerating with water, a sufficient quantity of sulphuric acid should be added to the beetroot slices that the juice to be fermented may contain 2½ grms. of acid per litre. Thus if 1,000 kilos. of beetroot give 18 hectolitres of juice, 1,800 kilos. would require 5 litres of sulphuric acid, allowing a little to neutralise the earthy matters adhering to the roots.

This quantity of acid is altered in succeeding operations according to the quantity of vinasse returned. In order to obtain an exact test of the acidity of the juice, it is of advantage to have preparatory vats from which the juice may be run to the fermentation vats; otherwise samples should be taken at regular intervals while running off from the macerators, and an average of the samples tested. Accidents in fermentation and decreased yield are always due to defects in acidification. When an acid fermentation has occurred, a quantity of the vinasse should be rejected, and the dose of sulphuric acid increased until the working becomes normal.

Allowing a loss of sugar in the pulp of 0·5 per cent., and a loss of 1 per cent. of alcohol during distillation, juices of a density of 1·050, 1·055, and 1·060 should give 5·20, 6·0, and 6·6 per cent. of alcohol respectively in average work.—L. J. de W.

*Furfural in Alcoholic Liquids, Occurrence of.* H. van Laer. J. Fed. Inst. Brewing, 1898, **4**, 2—5.

Chapman (J. Fed. Inst. Brewing 1897, **3**, 247) has expressed the opinion that furfural is present in beer as an unfermented constituent of the original wort, and is not a fermentation product. To prove this statement, the author fermented a beer wort, under aseptic conditions, with Logos yeast. After fermentation, the beer, and, at the same time, a portion of the original wort, were distilled, and it was found that the distillate from the beer gave a less intense furfural reaction than that derived from the wort. Again, when dextrose, dissolved in yeast water, was fermented with pure cultures of various yeasts, no furfural could be detected in the distillates when the fermentation was over.

Fermentations were carried out with a pure English yeast in presence of arabinose and xylose. The pentoses, it is known, are not susceptible of alcoholic fermentation, but the yeast reproduces itself in the nutritive solutions of these sugars and assimilates a portion of them. (Compare Bokorny; this Journal, 1897, 345.) When the pentoses had disappeared, the liquids were distilled, and considerable quantities of furfural were found in the distillate. It is also stated that the distillate from a decoction beer contains more furfural than one from a beer brewed under the infusion system.

In view of these results, the author concludes that the furfural found in fermented liquids is not due to the vital action of the yeasts. It is probable that during the process of mashing, and especially during the development of acidity which accompanies the stand at a low temperature, a more or less considerable quantity of the pentosans present in the grains is hydrolysed, and passes into the wort as pentoses; these pentoses then give rise to furfural in the course of the distillation by the action of acids on them. To illustrate this statement, solutions of arabinose and xylose, when distilled with lactic acid, were found to yield furfural in proportion to the acidity of the solution.—J. L. B.

*Neutralisers to Beer: The Estimation of the Amount added.* E. Spaeth. Zeits. für angew. Chem. 1898, 4—5.

*See under* XXIII., *page* 188.

*Acidity, Determination of, in Beer and other Liquids containing Acid Phosphates.* A. Ott. Zeits. für d. ges. Brauwesen, **20**, [43], 549; [45], 579; [46], 593.

*See under* XXIII., *page* 188.

*Sugar in Alcoholic Solutions, Table of Solubility of.* H. and L. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 630.

*See under* XVI., *page* 169.

### PATENT.

*Worts, and Apparatus therefor; Impts. in or relating to the Clarifying or Fining of.* G. Putz, Hildesheim, Germany. Eng. Pat. 15,825, July 2, 1897.

In order to draw off worts from a fining or clarifying vat without disturbing the grounds at the bottom, the discharge pipe is connected with a float so as to automatically descend, as the level of the worts sinks.—A. L. S.

*Spirituous Liquors, Improvements in the Treatment of, and the Products obtained therefrom.* A. J. Boult, London. From K. E. N. Fryklind, Helsingborg, Sweden. Eng. Pat. 4441, Feb. 18, 1897.

This is a method by which a wine naturally too alcoholic may have a portion of its alcohol removed without spoiling its character. The wine is placed in a boiler connected with a still and receiver, and the pressure within reduced to one-twelfth of an atmosphere. The wine is then heated to 30°—60° C., and the required quantity of the spirit distilled off. The heating of the still is then stopped, and, when the whole is cool, the pressure is restored to the normal.

—A. L. S.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Casein in Milk, A New Process for the Determination of.* G. Denigès. J. Pharm. Chim. 1898, **7**, [1], 9.

*See under* XXIII., *page* 188.

*Milk: Indigo Carmine as a Test of Freshness.* L. Vaudin. Répert. Pharm. 1897, [3], **9**, 538.

*See under* XXIII., *page* 186.

*Yeasts* [*Beer*], *Action of, on Milk.* E. Boulanger. Ann. Inst. Pasteur. La Bière, **5**, [11], 161.

*See under* XVII., *page* 172.

## PATENTS.

*Food for Animals, A New and Improved.* B. Horský, Prague, Bohemia. Eng. Pat. 2744, Feb. 2, 1897.

A MIXTURE is formed of six parts of finely ground waste from mills or rice-shelling factories, three parts of finely ground oil cake or waste from rice starch factories, and one part of flavouring substance, as cornflour or ground oats. This is kneaded with a suitable quantity of water and moulded into strips or grains by rollers, and dried in an oven. This food may be given to horses in place of oats, or may be used for feeding other animals.—A. L. S.

*Tea, Impts. in or connected with the Preparation of, for the Purpose of Rendering the Infusion of it more Digestible.* J. A. Martin, Southwark, Surrey. Eng. Pat. 27,460, Nov. 23, 1897.

In order to prevent the tannic acid of tea being dissolved when tea is mashed in the usual manner, a small amount of gelatin or gum acacia or albumin, together with tartrate and carbonates of potash and soda are added to the tea in bulk. —A. L. S.

*Cattle Food, An Improved, and Process for Producing same.* O. Zürcher, Piccadilly, London. Eng. Pat. 27,588, Nov. 24, 1897.

A CAKE is prepared from a mixture of ripe and unripe bananas as follows:—

The unripe fruits are dried and ground to powder, and the ripe fruit pulped and the peelings removed by skimming or sieving. The two products are mixed to form a dough, which is moulded into cakes and dried; if required, the pulp may be first concentrated *in vacuo.*

The dried cakes will keep for a length of time and form a useful cattle food.—A. L. S.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Sewage, The Purification of, by Bacteria.* S. Rideal. Jour. Soc. Arts, 1897, **46**, 81—93.

After referring to the success which has attended the experiments at Exeter on the disposal of sewage by the septic tank process and the unsatisfactory results frequently obtained by chemical precipitation and subsequent land treatment of the effluent, the author points out that 30 years ago upward-filtration was suggested in place of chemical precipitation, and from the satisfactory results then obtained it is strange that the precipitation process was almost universally adopted. In slow upward-filtration the arrested suspended matter slowly disappears, doubtless as a result of changes such as are commonly observed in the decomposition into harmless gases, of vegetable or animal matter at the bottom of a stagnant pool.

In nearly all cases, the destructive oxidation of organic matter seems to be preceded by a preliminary breaking down of complex compounds into simpler by anaerobic processes, either by the action of enzymes (as in the digestion process,) or by hydrolysis, or by the agency of organisms. Thus albumen might conceivably be broken down by pure hydrolysis into nitrogen, methane, carbon dioxide and hydrogen, as represented by the following equation:—

$$4C_8H_{13}N_2O_3 + 14H_2O = 4N_2 + 19CH_4 + 13CO_2 + 2H_2.$$

Gelatin is resolved by anaerobic organisms into albumoses, ammonia, peptones &c., without absorption of oxygen or the formation of oxidised products, and Emmerlich has recently shown that fibrin is similarly liquefied into ammonia, methylamine and other soluble products (Ber. 1897, **30**, 1863). The fermentation of elastin into carbon dioxide, hydrogen, methane and nitrogen, whilst the sulphur remains in solution as mercaptan and is not evolved as sulphuretted hydrogen (Zoja, Zeit. Physiol. Chem. 1897, **23**, 236), is on all fours with the changes occurring in the septic tank at Exeter, where the gases consist of nitrogen 38·6 per cent. hydrogen 36·4 per cent., methane 24·4 per cent., and carbon dioxide 0·6 per cent.; no sulphuretted hydrogen is present, and the ammonia formed dissolves in the water. These and other examples are discussed to show that the purification of sewage instead of being simply a process of oxidation, as was formerly supposed, must be largely brought about by important preliminary changes in which oxygen does not take part. For the complete destruction of the organic matter, oxygen is essential, but the term "nitrification," by which this process is usually described, does not take into account the important oxidation of organic carbon which occurs at the same time.

A brief description then follows of the different bacteriological methods which are now being tried for treating raw sewage, in the order of their introduction.

1892. *The Scott-Moncrieff System.*—The raw unscreened sewage is conveyed to the under side of a filter bed (supported on a false bottom) composed of successive layers, 14 ins. thick, of flint, coke, and gravel. The arrested suspended matter gradually disappears or dissolves, and the clear liquid, in rising through the filter bed, undergoes fermentative changes which become more and more aerobic in character towards the surface of the bed. To further improve the quality of the effluent, so-called nitrifying channels were provided, along which it was made to flow, but only partial nitrification took place, owing to the denitrifying action of the remaining organic matter. By a recent modification, two or more filter beds are worked intermittently, so as to allow of their periodic aeration and resting.

1895. *The Exeter (Cameron's) System.*—In this process, the raw sewage, after passing through a grit chamber, is made to flow slowly through a long, closed, underground tank. The effluent flows out at the far end, through a transversely placed slotted pipe, over an aerated weir, on to coke-breeze filter beds arranged in series, and fitted with automatic gear, so that a cycle of aerating, filling, resting full, and emptying is ensured to each filter in rotation. In flowing through the tank, the suspended solids gradually disappear, leaving only a very small residue of black humus-like matter. Dr. Sims Woodhead's investigations (see next abstract) seem to show that though the changes taking place in the tank are mainly anaerobic, the aerobes are capable of surviving and even of doing a certain amount of oxidising work. Probably nitrates are formed, but are reduced again with evolution of free nitrogen. By this method, the sewage from about 2,000 inhabitants has been dealt with continuously for nearly 18 months, and provision is being made to extend the system to the whole of the city sewage.

1896. *The Sutton (Dibdin's) Process.*—In this process, the crude sewage is turned into an open tank filled with coarse burnt ballast. The liquid portion is then drawn off and distributed over fine coke-breeze filters. The idea is that the tank shall act as a sort of coarse strainer, the coarse suspended matters being drawn into the body of the material, which becomes thoroughly aerated when the liquid portion is drawn off. Since a breaking down without oxygen has been shown to be an essential preliminary, anaerobic. changes must take place in this tank in spite of the aerobic conditions, and the atmospheric oxygen can be only indirectly advantageous.

1897. *The Ducat System.*—In this system, a filter is used composed of coarse material at the top and fine at the bottom, which is contained within walls made of perforated tiles or drain pipes, so that air can blow into the body of the filter. It is designed to be continuous in its action, and is intended to be heated in winter. Thus, as in Dibdin's tank filter, the coarse sewage is aerated without allowing any period for anaerobic changes. It is possible, however, that in towns with old sewers the sewage to be treated may be already so far advanced in anaerobic decomposition that a filter of this description can at once deal with it.

A question of great importance in connection with all these sewage processes has reference to what constitutes a satisfactory effluent. The standards of 2 parts of organic carbon and 0·3 of organic nitrogen per 100,000, sought to be legalised by the Rivers Pollution Commission, have been modified since by various authorities; but no authority, so far as the author is aware, permits a variation in the quality of an effluent proportionally to its amount, or to the volume of the river into which it discharges; neither does any authority attach a value to the ratio which the remaining organic matter bears to the available or potential purification existing in the nitrites or nitrates.

Dupré and the author found, in 1895, that the discharge into the River Exe of the sewage of 40,000 people, the river being 40 times the volume of the sewage, was not indicated by any serious alteration in the quality of the stream.

Dupré found that the putrefaction of London sewage could be arrested by adding 35 times its volume of London tap water. The production of nitrites in the natural purification of sewage, and their importance as carriers of oxygen, has not been sufficiently recognised. Analyses are given showing the rapid increase of nitrites in the Exeter filtrates, and that their formation could proceed in some cases independently of the reduction of nitrate. Gayon and Dupetit, in 1886, isolated from sewage two organisms, which, in the presence of organic matter, decomposed nitrates with the production of nitrogen and nitrous oxide gases. They showed that the whole of the nitrogen of the nitrate was evolved as gas, and that the whole of the oxygen was combined with the carbon of the organic matter to form carbon dioxide. Organic matter is essential to the reaction. These denitrifying bacteria will not develop in liquids deprived of nitrate and out of contact with air, nor will they attack organic matter under these circumstances. The authors have proved denitrification to be a fermentation which consists in the direct burning up of the organic carbon at the expense of the oxygen in the nitrate. Therefore, after partial nitrification in the filter, these denitrifying bacteria which absolutely require a certain amount of organic carbon as food, convert the latter into carbonic acid, taking the oxygen required either from the air or from that dissolved in the water. The change is illustrated by a table of analyses of typical sewages and effluents in which the oxygen present as nitrate and nitrite is reckoned as "available oxygen," and the carbonaceous matter requiring destruction is measured by the ordinary figure of "oxygen consumed," as determined by permanganate. In certain effluents, the "available oxygen" is seen to be sufficient of itself to oxidise the organic matter; in others, the number of volumes of oxygen saturated water required to be added, is given. Mr Henry Law has suggested that the conditions for the discharge of any effluent into a stream may be expressed in the following form:—

X = flow of stream in 100 litres per minute. O = grms. of free oxygen in 100 litres of the stream. S = number of 100 litres of effluent discharged per minute. $n$ = grms. of available oxygen as nitrates and nitrites in 100 litres of effluent. $m$ = grms. of oxygen required to consume the organic matter in 100 litres of the effluent, as determined by the permanganate test. C = number of times that the free oxygen in the stream exceeds that required to oxidise the organic matter in the effluent.

Then ·

Quantity of free oxygen in stream = XO.

Oxygen required by the effluent = $(m-n)$ S.

So that XO = C $(m-n)$ S.

When there are no nitrates in the fluid, then—

XO = C $m$ S.

When XO $>(m-n)$ S, we have stability, and when XO $<(m-n)$ S, the effluent should be condemned.

The author has already expressed approval of Adeney's suggestion that "The limit of impurity allowed to a given water (or effluent) should be such that, when a given volume of it is mixed with a given body of fully aerated water, and the mixture kept in a bottle out of contact with air for a sufficient length of time, a decided oxidation of the ammonia originally present into nitrous or nitric acid shall be indicated."

Reference was made to the untrustworthiness of many of the published analyses of effluents made several days after collection, when considerable changes must have taken place; also to the difficulty of obtaining comparable samples of sewages and effluents, owing to variations in the strength of the sewage and also to the considerable time taken for a given sewage to appear at the effluent outlet, so that the collection of isolated samples, especially when the sewage and effluent are sampled almost at the same time, furnishes no index whatever to the extent of purification. The only satisfactory basis for a comparison between effluents and sewages would be to calculate them to a uniform standard of chlorine. Increase in the free ammonia is a proof that destructive processes are going on, and even albuminoid ammonia in an effluent may be a good sign, indicating that suspended organic matter has gone into solution, or that stable soluble matter has been partially broken up. As a practical test of the effect of dilution with river water in effecting the oxidation of sewage matter, five samples of crude sewage, free from suspended matter, but smelling very offensively, were mixed with sufficient river water to supply as dissolved oxygen the quantity required by the organic matter, as measured by the oxygen consumed. Whether kept in the dark or fully exposed to daylight, the odour rapidly diminished, and in three days all except one were inodorous, the exception becoming so in five days. Analyses made after seven days showed that the original amount of "oxygen consumed" had been reduced by from 48·3 to 84·5 per cent., and that this had been chiefly brought about through the agency of the dissolved oxygen of the river water, denitrification having occurred only to a slight extent. Nitrites in most cases indicate a further stage of improvement, as their production necessarily implies a disappearance of a greater amount of nitrogenous matter. Nitrates of course indicate a final change, and their presence, together with free nitrogen, in deep well waters show that denitrification had stopped when the organic matter was consumed. The author objects, therefore, to all arbitrary standards, and considers that an effluent which is shown to be in an active state of self-purification, if it be clear and nearly free from odour, may safely be discharged into a river of moderate volume. Its subsequent improvement will be very rapid, and will be further determined by the action of algæ and larger plants, in the presence of sunlight, and of the oxgenated water of the river.—L. A.

*Sewage, Bacterial Treatment of; or Biological Filtration.* Harben Lecture by G. Sims Woodhead. J. of State Medicine, 1897, **5**, 453.

That the organisms of putrefaction might be utilised for the decomposition of sewage and refuse matter of all kinds has been a growing conviction since Pasteur published his classical researches on fermentation and putrefaction. It is well known that fertile soil teems with micro-organisms. Those existing near the surface, most of which are aerobic, break up the organic matter, using some of the constituents for their own needs, whilst another and far greater portion is converted into inorganic plant food. Light spongy soil, alternately moistened and dried by evaporation, acts as a most admirable oxidising medium, in which the organisms flourish and rapidly bring about the oxidation of organic matter. Into the deeper layers of the soil the oxygen does not penetrate, but the organic matter is there attacked by another series of anaerobic organisms which obtain the oxygen they require from the organic matter itself, decomposing it and setting free large quantities of marsh gas, ammonia, sulphuretted hydrogen, and other products, some of which remain in combination with the soil; others rise to the surface and become oxidised. As we go deeper, even the anaerobic organisms become fewer, until at a depth of about 12 feet, or less, no micro-organisms can be found. The earth in this way acts as a true biological filter, splitting up into simple inorganic products the organic matter contained in fluids which filter through it. Similarly the solid matters contained in fæces undergo rapid decomposition when mixed with dry earth.

In ordinary town sewage, the organic matter is carried in a large volume of water, partly dissolved and partly in suspension, and it is essential in the first place to get the suspended matter into solution or to remove it by precipitation. In the experiments made by Scott-Moncrieff and Houston in 1891–92, the crude sewage was first passed through "cultivation" tanks, simply containing masses of flint, the surfaces of which soon became covered with organisms, by the agency of which the solid organic matter was broken down or peptonised, and was thereby brought into solution and made ready for further treatment. The effluent was passed down a nitrification channel, in which a continuous aëration of the liquid took place; or, as suggested by the inventor of the process, the peptonised sewage might be turned into a slow or well-aërated stream in which either bacterial change or oxidation and nitrification should be rapidly effected. This process gave very good results. In 1892–95, Dibdin proved that after removing the solid matter from sewage by chemical precipitation, he was able, by intermittent filtration through coke breeze and other similar materials, to obtain a purification of from 70 to 75 per cent.

The most recent process devised for the bacterial disintegration of sewage is that of Cameron, which is in operation at Exeter. This process resembles in principle Scott-Moncrieff's, in that the suspended organic matter, instead of being removed by chemical precipitation, is got into solution by the agency of organisms. The sewage is first received into a large underground tank, the top, bottom, and sides of which are formed of cemented brickwork. This tank, which has a capacity of 53,800 galls., receives the average sewage of about 1,500 inhabitants in 24 hours. Its length is nearly 65 ft., and if the flow of sewage were constant the rate would be rather more than 2½ ft. per hour. 7 ft. from the inlet is a submerged wall, 12 ins. below the surface of the liquid. In the chamber thus formed, heavy particles of gravel and earth are deposited, and are removed by dredging without disturbing the liquid in the remainder of the tank. From this chamber the sewage passes slowly over the submerged partition into the main chamber, where the movement of the liquid is so slow that the suspended organic matter for the most part gradually settles, though small masses of fæces, buoyed up by gas, rise to the surface and form a thick leathery scum. On the surface of this layer, numerous liquefying areas may be seen. Small particles from the under side are continually falling to the bottom of the tank, fresh particles rising to take their place. The thickness of the layer does not appear to increase after a time; it seldom exceeds 4 or 5 ins. in winter and 1 or 2 ins. in summer. The solid matter, which settles to the bottom of the tank, is gradually disintegrated and dissolved by the agency of organisms present in the liquid, the conditions for their development being so extremely favourable that even with the most careful dredging it is impossible to bring up more than a very small quantity of inorganic mud and "fibrillar" tissue, even the resistant cellulose becoming decomposed and digested by the organisms. As a result, the effluent from this tank, though somewhat darker in colour and distinctly more opalescent, contains less organic matter in suspension and more in solution than the original sewage. The effluent passes through a transversely placed slotted pipe into a trough, from which it overflows on to the surface of one of four filter beds worked in rotation. Each filter takes approximately 6 hours to fill, remains full for 6 hours, is then rapidly emptied in about ½ hour, and finally remains empty for about 11½ hours. The filtrate is usually bright, and under the worst conditions has seldom more than a slight opalescence and a faint cabbage odour.

In order to study the changes taking place in the septic tank, "counts" were made of the organisms in the various fluids. Of *anaerobic* organisms, the crude sewage contained the largest number. In the tank the total number of organisms was fewer, but the proportion of liquefying organisms had materially increased. In the tank effluent, as well as in the filter effluent, the number of liquefying organisms had fallen very considerably. Of *aerobic* organisms, again by far the largest number were found in the crude sewage and in the tank contents; non-liquefying organisms were also present in the samples in much greater numbers than in the filtrate from the biological filters. The various organisms separated in pure culture were found to multiply with extraordinary rapidity in sterile sewage, most of them causing more or less extensive nitrification, some reducing the nitrates to nitrites. In the tank, the process being purely anaerobic, nitrates are not formed. Dr. Rideal estimates that 400 lb. (about ⅕th of a cubic yard) of solids, principally fæces, are carried daily into this tank, and if none disappeared, the tank would become full of solid matter in about three years. As it has already been running about 11 months, it should, at this rate, be nearly ⅓rd full, instead of which there is practically little or no sediment to be found. The tank effluent, as compared with the original filtered sewage, shows an increase in the dissolved solids, a reduction of 33 per cent. in the oxygen consumed, an increase of about 33 per cent. in the free ammonia, and a reduction of about 54 per cent. in the albuminoid ammonia, or 50 per cent. in the organic nitrogen. There is also a slight production of oxidised nitrogen and a disappearance of a small proportion of the total nitrogen. Some gas taken from below the surface of the liquid in the tank was found to contain:—

| | |
|---|---|
| Carbon dioxide | 0·6 |
| Methane | 24·4 |
| Hydrogen | 36·4 |
| Nitrogen | 38·6 |
| | 100·0 |

The atmosphere of the tank contained from 0·54 to 0·81 per cent. of carbon dioxide, *i.e.*, fully 15 times as much as the outside air. There is also a considerably greater quantity of carbonic acid in the tank effluent than in the raw sewage, the quantity of carbon lost in this way being estimated to amount to about 12 lb. in 24 hours. Though the gases given off might be expected to give rise to offensive smells, such is found not to be the case, no smell being detected at a very short distance from the tank.

Biological examinations of the filtering material and of the filtered effluent showed that the organisms in the filter beds and those contained in the filtrate are mostly aerobic, though anaerobic organisms are also present. As most of the purification is brought about by aerobic organisms, it is absolutely necessary not only that the fluid passing on to the filter should be well oxygenated, but large quantities of oxygen should also be passed into the filter bed. This oxygen is very rapidly used up, and even while the filter is being filled, the amount of carbon dioxide is enormously increased in the upper layers, owing to the rapid action of the organisms in the filter. When the filter is full of liquid, no more oxygen can be taken up, and as soon as that present has been consumed, it is useless to keep the fluid longer in the filter. It is then drawn off, and in flowing away is replaced by a further supply of oxygen. If it were possible to maintain an excess of oxygen in the filter, there seems no reason to doubt that the whole of the organic nitrogen, some of which at present assumes the form of ammonia and perhaps free nitrogen, might be oxidised into nitrates and nitrites. The efficiency of the filters must be judged by the proportion of oxidised nitrogen contained in the filtrate. Rideal aspirated samples of gas from different levels in a filter two or three hours after the liquid had been discharged. At a depth of 18 inches the percentage of carbon dioxide by volume was 0·375, at 36 ins. 0·98, and at 54 ins. 0·75. From these proportions he calculates that the weight of organic carbon destroyed per day by each filter is about 2½ lb., but no experiments were made to determine the considerable amount of dissolved carbonic acid contained in the filtrate. From Rideal's experiments, the purification, as measured by the oxygen consumed, was 73 per cent., or, measured by the albuminoid ammonia, 60 per cent., whilst the purification due to both septic tank and filters amounted to 82 and 77 per cent. respectively. This 73 per cent. purification, measured by the oxygen test, may be compared with 75 per cent. obtained by Dibdin with coke-breeze filters and a chemically treated effluent.—L. A.

## PATENTS.

*Sewage and Polluted Waters, Impts. in the Purification of.* F. P. Candy, Chester. Eng. Pat. 27,513, Dec. 3, 1896.

The first improvement relates to screens for removing the larger suspended particles, which are formed of parallel bars, curved in shape, with a revolving rake, the teeth of which engage between the bars and carry away the solid matter deposited thereon.

The second part relates to precipitation tanks. Instead of taking away the effluent in a thin layer over a sill, a number of passages are provided in the end wall of the tank, at different levels, down to within about 2 ft. of the bottom of the tank, by which means a much more even flow of sewage is maintained throughout the tank.

The third part relates to filters. A bed of gravel, coke, or other material is placed in a tank, a space at either end being partitioned off by perforated walls. Filtration takes place in a lateral direction, and the filtering material diminishes in fineness from the inlet to the outlet end, and also diminishes in height, as a head of about 1 to 2 ins. per 10 feet is required to cause sufficiently rapid filtration, which may be from 500 to 1,000 galls. per 24 hours per sq. ft. of cross sectional area. Open spaces are sometimes arranged in the filtering material to facilitate the deposition and removal of solid matter, and an outlet is provided, connected with gutters in the bottom of the tank, by which the filter may be completely emptied for aeration and for flushing purposes.—L. A.

*Water Purification, Impts. in the Methods of and Apparatus for.* J. S. McDougall, Didsbury, Manchester. Eng. Pat. 29,223, Dec. 19, 1896.

For the purification of oily water, such as condensed water from a steam engine, it is passed into a suitable tank, raised to the boiling point, and mixed with a solution of calcium chloride, or a mixture of calcium chloride and lime, which causes the separation of the fatty matter in a condition easily removed by filtration.

If the water contain in solution calcium and magnesium salts, whether oily or not, it is treated at the boiling point with a mixture of caustic soda and sodium carbonate, and the precipitate formed is filtered off as before.—L. A.

*Filters [Water], and their Installation, Impts. in.* W. Defries, 1, Danes Inn, Strand, London. Eng. Pat. 30,147, Dec. 31, 1896.

In order to obtain an approximately constant output, the pump for producing the vacuum in the filtered water chamber, or the force pump of a pressure filter, is limited to the speed necessary for such output, and the pump, or a cock or orifice on the inlet, passing only such output, is employed in combination with a vacuum or pressure gauge indicating the variations in effective pressure. To facilitate the removal of the deposit from the filtering medium, the latter is coated, before commencing filtration, with a deposit of Kieselguhr, &c., which is floated by means of water on to the filtering material, a series of inclined baffle-plates being interposed in the direction of flow of the water, to facilitate the distribution of this deposit. In some cases the deposits may be removed from the filters by subjecting them to the action of a suitable solvent. The solvent may be introduced by means of a traversing sparge pipe provided with jets at various angles. Filters formed of hollow bodies with closed upper ends are cleaned by the withdrawal of a large part of the contained air from the filtering bodies, and the replacement of it by a suitable detergent liquid.
—R. A.

*Filtration of Water, Sewage, and other Liquids, Impts. in the.* A. J. and P. A. G. Bell, Manchester. Eng. Pat. 2384, Jan. 29, 1897.

The filtering material is contained in a closed pressure chamber containing a vertical, hollow shaft carrying perforated radial arms, and capable of revolution. The liquid to be filtered is led through a pipe and valve into the upper part of the chamber, and the clear effluent is drawn off from the bottom. When the medium becomes choked, the valve admitting the turbid liquid is closed, and the outlet at the bottom is also closed. Clear water is then forced into the chamber through the hollow shaft and radial arms, which are caused to revolve so as to stir up the filtering material, and the sludge which is washed out passes away from the top of the chamber through a special pipe.—L. A.

*Waste Matters [Garbage, &c.], Process of and Apparatus for the Treatment of.* G. Barker, Birmingham. From L. Rissmuller, Philadelphia, U.S.A. Eng. Pat. 24,085, Oct. 19, 1897.

The waste matter, consisting of city garbage, animal or vegetable matter, &c. is placed in a digestor lined with lead, and is there boiled with water and acid (a mixture of sulphuric and phosphoric acids is preferred) until it is converted into a uniform fluid mass. It is then transferred to a centrifugal separator in which the grease is completely separated. The acid solution and the suspended solids are then treated with phosphate of lime or other suitable substance which will neutralise or remove the free acid, and the dried product is used as a fertiliser.—L. A.

### (C.)—DISINFECTANTS.

### PATENTS.

*Methyleneditannic Acids, from Formoldehyde and Tannin; Impts. in the Manufacture of.* E. H. C. Dürkopf, Darmstadt, Germany. Eng. Pat. 6686, March 1, 1897.

To effect the condensation of tannins with formaldehyde, the patentee now claims the process which consists in heating the mixture under pressure, as an improvement on the process claimed in Eng. Pat. 816, 1896 (this Journal, 1896, 917).—A. C. W.

*Carbolic Acid, An Improved Process for the Preparation of, in a Non-hygroscopic Form.* F. Lutze, Berlin. Eng. Pat. 22,136, Sept. 27, 1897.

Crystallised or absolute phenol is stirred into melted anhydrous boric acid, the mass is allowed to solidify, and ground into powder. This powder does not become wet by absorption of moisture, because the water combines with the boron trioxide, forming solid boric acid. Instead of boron trioxide, other anhydrous oxides may be used, such as arsenic trioxide, alumina, and magnesia; also dehydrated salts, such as calcined borax, alum, pyro-phosphates, and meta-phosphates.—L. A.

*Superheated or Saturated Steam for Disinfecting, Cleaning, Sterilising, and similar Purposes; An Improved Process and Apparatus for the Production and Use of.* B. Greve, Berlin. Eng. Pat. 24,535, Oct. 22, 1897.

The steam generator is a vessel having a large inner chamber into which a highly heated metallic block is placed. A bell-shaped cover is then put on and firmly clamped down on to a washer. Steam is generated by water which is pumped into the vessel, coming into contact with the heated block, and the steam passes out through a receptacle contained in the upper part of the vessel, in which a disinfectant can be placed. If the water is pumped into the lower part of the vessel, the generated steam is superheated in rising past the glowing block; if saturated steam is required, the water is pumped into the upper part of the vessel. The steam charged with disinfectant is led into the apparatus which it is required to cleanse.

By pumping more water in, the apparatus can be made to deliver hot water, and if the receptacle is filled with soda instead of disinfectant, a hot alkaline liquid is produced, which is convenient for cleaning pipes, &c.—L. A.

*Creosote Compound [Phosphate], Improved.* J. Brissonnet, Paris, France. Eng. Pat. 25,945, Nov. 8, 1897.

*See under* XX., *page* 181.

## XIX.—PAPER, PASTEBOARD, Etc.

*Waste Liquors from "Sulphite" Wood Pulp Process.* H. Seidel. Mitt. d. k. k. Tech. Gew. Mus. N.F. 7, 1897, 119—225.

The liquor investigated by the author was obtained from a Silesian mill working the Ritter-Kellner process.

The following analytical results may be noted:—Sp. gr. 1·050; total solid residue (dry at 100°) 12·013 grms. per 100 c.c., *i.e.*, 11·44 per cent., containing 9·54 per cent. total sulphur. The statistics concerning sulphur are as follows, calculated in this case per cent. of the liquor:—

| | |
|---|---|
| Sulphur in dry residue, per cent. of liquor ..... | 1·0910 |
| Sulphur as free $SO_2$ .............................. | 0·0320 |
| Sulphur as $SO_3$ .................................. | 0·0074 |

of the total sulphur of the liquor, 93 per cent. exists in the form of "organic" compounds.

In reference to the claim for value of these liquors as fertilisers, by reason of their potassium-salt contents, a determination of potash was made, and found to amount to 1 grm. of $K_2O$ per 52 litres.

A study of the precipitates obtained by various processes of "salting out," *e.g.*, Ekman's series of so-called "Dextron" products [D.R.P. 81,543], confirms the author's previous conclusion (*ibid.* 1896) that this method of fractionation of the organic constituents of the liquor, fails to bring out any constitutional differences. A similar conclusion results from a study of the fractions obtained by precipitation with alcohol. The analysis of a carefully purified fraction gave the following numbers:—C, 56·27 per cent.; H, 5·87 per cent.; S, 5·52 per cent. in closest agreement with numbers obtained by Lindsey and Tollens, and corresponding with the empirical formula, $C_{26}H_{30}SO_{12}$. It is to be noted also that the results of these chemists were obtained with a liquor from a mill working under the different conditions of the Mitscherlich process.

Respecting the theory of the reactions taking place in the digester, the author advances no new hypotheses, but generally confirms those summarised in "*Cellulose*" (Cross and Bevan), pp. 177 to 201.—C. F. C.

### PATENTS.

*Waterproof Paper, Pasteboard, Millboard, and the like, Manufacture of; and the Production of Compounds therefor.* C. D. Abel, London. From "Farbwerk, Friedrichsfeld P. Remy," Mannheim. Eng. Pat. 29,829, Dec. 28, 1896.

The dark-coloured waste lyes from sulphite cellulose factories contain the lime salts of one or more organic acids, which, when converted into the corresponding compounds of lead, or preferably iron (ferrous or ferric), by any convenient method of double decomposition, possess the property of rendering paper, &c. waterproof. The material is saturated with one of these solutions of suitable concentration, dried at 80° to 130° from two to four hours, and washed in water, if so desired, in order to remove the soluble colouring matter. The same liquids are also applicable as size for inferior papers. Various methods of preparing the iron salts are given in detail in the specification.—F. H. L.

*Paper, Disintegrating and Washing Rags, or other Fibrous Material, for the Manufacture of; Improved Process of and Apparatus for.* J. H. Annandale, Polton, Midlothian. Eng. Pat. 30,103, Dec. 31, 1896.

Rags or other fibrous material, preferably after a preliminary cleansing to remove grosser impurities, are fed with water, by means of a hopper, into a disintegrating machine. This consists, essentially, of a revolving cylinder with a number of V-shaped cutting teeth on the circumference, surrounded by a stationary casing, which also has cutting teeth on the inside.

The teeth on the cylinder and casing are furthest apart where the rags are fed in, and gradually approach one another towards the end where the pulp leaves the machine. After leaving the disintegrator, the pulp is delivered on to a sand table (Eng. Pat. 23,743, 1895), to separate the heavy impurities, and thence through a series of cone washers, where it is repeatedly washed with water of increasing purity, the waste water from each washing being used for an earlier stage in the cleansing process.—C. M.

*Writing Paper, An Improved; and Ink therefor.* F. Cox, F. Kornacher, and M. Brumm, Frankfort a/Main. Eng. Pat. 24,560, Oct. 23, 1897.

Instead of employing a ready-made ink in the ordinary way, the inventors suggest that writing paper should be coated with a 1 or 2 per cent. solution of tannin or gallic acid, and the pen wetted with a 2 or 3 per cent. solution of ferrous sulphate, thickened if necessary, and perhaps containing some ferric salt, &c. On suitable paper, the ink yields good copies after a lapse of several days; yet it is equally available for "record" purposes simply. Among the advantages claimed for this process are that the fingers, &c. are not soiled, whilst the additional cost of the paper is more than compensated by the reduced price of the "ink."
—F. H. L.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Pure Iodine, The Preparation of.* B. Lean and W. H. Whatmough. Proc. Chem. Soc. 1898, [187], 5—6.

A few months ago the authors observed incidentally that no iodine is set free when cuprous iodide is heated, even at its fusion point, in a current of carbonic anhydride, although it is readily evolved when cuprous iodide is heated in air, oxygen, nitric oxide, or nitrogen peroxide. The action is represented by the equation $Cu_2I_2 + O_2 = 2CuO + I_2$. No iodine is evolved when cuprous iodide is heated in a vacuum.

The authors have examined the usual methods of preparing cuprous iodide. When a solution of cupric sulphate and a soluble chloride, bromide, or iodide is saturated with sulphurous acid, cuprous iodide, bromide, and chloride may all be precipitated; but there is so great a difference in their degree of solubility that, by securing a proper dilution, it is probable that cuprous iodide can be prepared free from cuprous bromide or chloride. A mixture of cupric sulphate and ferrous sulphate is still less liable to precipitate cuprous bromide and chloride along with cuprous iodide than cupric sulphate saturated with sulphurous acid. The authors have found that cuprous iodide can also be prepared by sprinkling iodoform in small quantities at a time upon a hot surface of copper. On account of the difference in the properties of chloroform, bromoform, and iodoform, it is probable that by this method also, cuprous iodide can be prepared entirely free from bromide or chloride. If, moreover, the cuprous iodide be fused in a current of carbonic anhydride or *in vacuo*, it can be freed completely from moisture.

Iodine is most conveniently prepared from cuprous iodide by heating it in a stream of dry air at 220°—240° and condensing the vapours upon a cold surface. Although the greater portion of the iodine in a given quantity of cuprous iodide is quickly expelled, it is not easy to expel the whole; after heating 1·7101 grms. at 400° for 18 hours, 0·15 per cent. of the iodide was still undecomposed—otherwise the relation $Cu_2I_2:2CuO$ could have been utilised for the exact measurement of the atomic weights of copper, iodine, and oxygen. The authors are making further experiments upon this point. The action of air upon cuprous iodide is not dependent upon the presence of moisture. This is established by sealing cuprous iodide in glass tubes in the presence of phosphoric anhydride.

Iodine liberated as described from cuprous iodide at 240° leaves absolutely no residue when volatilised at 75°. If examined spectroscopically, no evidence of the presence of copper can be found The melting point (uncorrected) is 112·5—114°.

*Glycerophosphates, Contribution to the Study of.* A. Astruc. J. Pharm. Chim. 1898, **7**, [1], 5—8. (See this Journal, 1898, 66, 73, 78.)

Calcium glycerophosphates in solution are alkaline to methyl orange, sometimes alkaline and sometimes acid to phenolphthaleïn. The alkalinity shown towards the latter indicator is always extremely small, but the preparations with an acid reaction require a considerable quantity of alkali for neutralisation. If the formula for calcium glycerophosphate be taken as—

$$CaPO_3.OCH_2.CHOH.CH_2.OH,$$

the amount of sulphuric acid required for neutralisation, using methyl orange as indicator, is given by the equation—

$$2(CaPO_3.OC_3H_7O_2) + H_2SO_4 = CaSO_4 + CaH_2(PO_4C_3H_7O_2)_2.$$

The amount of soda required to show alkalinity towards phenolphthaleïn in a solution neutral to Helianthin is expressed in the equation $CaH_2(PO_4C_3H_7O_2)_2 + 2NaOH = CaNa_2(PO_4C_3H_7O_2)_2 + 2H_2O$.

The author adopts the process of making the solution neutral towards methyl orange and then titrating with soda, using phenolphthaleïn as indicator. This procedure will not indicate the addition of disodium phosphate, but would show the presence of phosphoric acid or monocalcium phosphate. The conclusions drawn, are, that the glycerophosphates become decomposed during their preparation, as is shown by their acidity towards phenolphthaleïn, and that a glycerophosphate which has not decomposed ought to require for neutral reaction towards methyl orange a quantity of acid equivalent to the alkali necessary to render alkaline to phenolphthaleïn, a solution previously made neutral to Helianthin.—A. C. W.

*Absolute Alcohol, Use of Carbide of Calcium for the Preparation of.* P. Yvon. Comptes Rend. 1897, **125,** [26], 1181—1182.

Alcohol of 90, or better 95 per cent. strength, to which a quarter of its weight of calcium carbide in coarse powder has been added, is agitated at intervals for two hours, and allowed to rest for 12 hours, or longer, if gas be still disengaged. It is then distilled and a little anhydrous sulphate of copper is added to the distillate, which is re-distilled from the copper acetylide formed. The resulting distillate is absolute alcohol, and gives no precipitate with barium alcoholate.—J. A. B.

*Peppermint Eucalyptus, Essential Oil of the.* Imp. Inst. J., Dec. 1897, 366.

Baker and Smith have communicated to the Royal Society of New South Wales, the following facts with regard to the oil obtained by the steam distillation of the leaves of the Sydney peppermint, *Eucalyptus piperita.* The most important constituent found was a substance resembling camphor, named by the authors, "eudesmol," which crystallises in needles and melts at 74° C. It was obtained by fractionally redistilling the crude oil and collecting the fraction boiling between 265 and 270° C. The oil itself has a specific gravity of 0·9096 and contains, in addition to the eudesmol, a considerable quantity of eucalyptol. The amount of oil obtained from the leaves by distillation is comparatively large.—A. S.

*Vanilla, Treatment of [Drying Operation], by Calcium Chloride.* J. Soc. of Arts, 1897, **46,** [2349], 39—40.

The object aimed at in the treatment of vanilla is to endow it with keeping properties, and at the same time develop the aroma which has not yet come into being at the time of cropping. Pods of the best quality should be perfectly smooth and without excrescence or holes. The longer the pods, and the more perfumed they are without acidity, the more valuable is the vanilla. On a well-ventilated and properly exposed plantation, the pods are ripe when the lower part begins to turn yellow. The calcium chloride treatment is as follows:—On the day of cropping, or at the latest, the following day, the pods are placed in tin cases, which should not be too large, as the subsequent heating must be as uniform as possible. These cases are fitted with lids closing on the outside of the box; they are lined with wool carefully arranged along the bottom and up the sides, and a little over the tops of the sides. The vanilla pods are placed on end, close enough to secure pressure without damage by rubbing, a horizontal layer is placed on top of these, the woollen cover is folded over all, and the lid put on. The tins are then put into the halves of wine-barrels, and hot water emptied into the barrels up to the lids of the cases, care being taken that no water gets into the cases. The barrels are covered in order to prevent the sudden cooling of the hot water. After standing over-night, the cases are taken out and the pods withdrawn, dried by exposure to air, and then kept for two or three days under woollen coverings in full sunlight. If the drying by hot water has been properly carried out, the colouring of the pods, after this latter operation, should be uniform. The farther drying of the pods is carried out in boxes of galvanised iron with a hinged door which closes on an india-rubber edging to ensure air-tightness. Each box has 11 drawers or trays; the bottom and the sixth drawer are for the vessels containing calcium chloride, and the others for holding the vanilla. 18 kilos. of calcium chloride are used for 45 kilos. of vanilla. The trays should not be made of resinous or strong-smelling woods, as vanilla absorbs and retains odours it comes in contact with. The vessels for holding the calcium chloride are double bottomed, the inner one being perforated to allow of the escape of the liquid chloride, and fresh calcium chloride is added when necessary. After the receptacles have been charged with calcium chloride and vanilla, the boxes are hermetically closed. The drying operation requires from 25—30 days, but the vanilla is examined every two or three days, and any pods showing moisture are taken out and put aside to be placed in the sun and prepared by themselves in a special box where they are all collected. Vanilla insufficiently dried will not keep, and breeds small worms; vanilla over-dried, keeps well, but is not supple—it is called "broken" and has less commercial value. The drying is finished first on small frames, and then in tin boxes, traces of mildew being carefully wiped off the pods. The vanilla is then cleaned by agitation with water at 140° F., and is then lightly wiped and dried in the shade. Vanilla, after being packed, is always watched for at least a month before being sent away; the boxes are examined twice a week, and every pod showing the least sign of moisture is withdrawn. The mildewed pods are worked up by various processes, and sold as inferior vanilla.—A. S.

*Bismuth Subnitrate, Influence of, in "Hardening" of Ciders.* L. Dufour and Daniel. Comptes Rend. 1897, **125,** [25], 1125.

*See under* XVII., *page* 173.

*Digitonin Reaction.* C. C. Keller. Ber. Pharm. Ges. 1897, **7,** 470.

*See under* XXIII., *page* 186.

## PATENTS.

*Iodine and other Useful Products, Impts. in the Treatment of Seaweed and the like for the Production of.* A. James, l'Ilede, Bréhat, France. Eng. Pat. 422, Jan. 6, 1897.

Fresh seaweed is soaked for about 12 hours in limed seawater, followed by repetitions of the treatment for a shorter time, with smaller proportions of the same, the weaker solutions thus obtained being again used with more seaweed. The greater part of the iodine is stated to be thus abstracted, while constituents of manurial value are left in the weed. The solution is treated with ferrous sulphate or with caustic lime to precipitate mucilaginous matters, which are added to the following batch of seaweed. The cleared solution is treated with sulphuric or hydrochloric acid in stated proportion, in a wooden vat fitted with agitators, and the iodine is liberated by nitrous nitric acid, or by other suitable reagents. The liberated iodine is separated from the solution, preferably by petroleum oil, which is subsequently agitated with weak potash or soda lye, and from the solution of iodide and iodate thus obtained, the iodine is precipitated by potassium chlorate and hydrochloric acid. It is stated that from 90 to 95 per cent. of the iodine contained in seaweed may be thus extracted. Certain modifications in the processes are prescribed when the seaweed to be treated is dried.—E. S.

*Dichloroxypurine [Synthesis of Caffeine, &c.], Impts. in the Manufacture or Preparation of.* C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 3957, Feb. 13, 1897.

The patentees claim the preparation of dichloroxypurine, $C_5H_2N_4OCl_2$, by treating uric acid or a dry urate with phosphorus oxychloride (E. Fischer, Ber. **17,** 1787). The transformation is effected by heating to 160°—170° for some hours.—A. C. W.

*Piperidine, Manufacture of New Products [Salts] from* J. Turner and Co., Queensferry; and P. Schidrowitz and O. Rosenheim, London. Eng. Pat. 4657, Feb. 20, 1897.

The patentees claim the manufacture of piperidine compounds by treating piperidine, either alone or dissolved, with carbonic acid, by dissolving piperidine and tartaric acid together, and recrystallising, and by melting together piperidine and succinic acid. The products of these processes are claimed, *viz.*, piperidyl carbamate of piperidine, $C_5H_{10}N.CO.OH(NH.C_5H_{10})$, and piperidine tartrate and succinate, $C_5H_{11}N.C_4H_6O_6$, and—

$$(C_5H_{11}N)_2.C_4H_6O_4.$$

—A. C. W.

*Creosote Compound [Phosphated], Improved.* J. Brissonnet, Paris, France. Eng. Pat. 25,945, Nov. 8, 1897..

The processes for producing a compound of creosote and phosphoric acid are claimed, which consist in treating creosote in aqueous alkaline solution with phosphorus oxychloride, pentachloride, or pentoxide, or in adding sodium to a toluene solution of creosote, and then the phosphorus compound.—A. C. W.

## XXI.—PHOTOGRAPHY.

*Diamidohydroxydiphenyl as a Developer.* M. Andresen. Phot. Corr. 1897, **34**, 587.

A comparison between *p*-amidophenol and diamidohydroxydiphenyl. Concentrated solutions of the latter can only be made in caustic alkalis, so that developers cannot be prepared from it by using either sulphites or carbonates of the alkalis. In caustic alkali solution, *p*-amidophenol yields much more rapidly than the other a similar delicacy in the half tones to pyrogallol, and at the same time a similar clearness of shadow to ferrous oxalate. Precht has stated that diamidohydroxydiphenyl is the first instance of a body capable of acting as a developer without alkali, and attributes this property to the division of the active groups between two benzene nuclei; but para-amidophenol itself acts as a developer in neutral sulphite solution, so that the presence of the amido group in the second nucleus is not essential.—J. T. D.

*Photography in Natural Colours.* R. Neuhauss. Phot. Rundschau, 1897, **11**, 321 and 355.

The author has investigated the causes of the frequent failures in the Lippmann process. The mercury must be frequently filtered; best under pressure. On pouring the gelatin on the plate, just as much should be allowed to flow off again as does so easily, and any subsequent centrifugal draining should not be too severe. The author has used albumin instead of gelatin for the plates, and gives full directions for its application. The results with pure colours are very good, but with mixed colours the photographed tints always appear to be seen through a film of uniform red, yellow, or green. By carefully removing the upper layer of the film, however, by Farmer's solution of sodium thiosulphate and potassium ferricyanide, the colours come out as clearly as though photographed originally from pure colours.—J. T. D.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Gunpowder, High-Grade.* A. F. Hargreaves. Trans. Mining Inst. of Scotland, 1897, **19**, 2—13.

The author traverses the evidence adverse to the use of gunpowder, especially that of H. Hall, given in 1894 before the Royal Commission on Explosions from Coal-dust in Mines. Stuart has shown that not 2,000, as stated in his report for 1892, but only 664 deaths were caused by explosions in coal mines from 1873 to 1892; and that explosions brought about by candles, lamps, and high explosives are charged against gunpowder. Now, on analysis of statistics, it is found that at least 90 per cent. of the shots fired in the United Kingdom are those of gunpowder, and that the remaining 10 per cent. of high explosives have, during the last four years, caused 25·33 per cent. of the total deaths in handling, and 69·66 per cent. of those in shot-firing. Samples of gunpowder collected at random throughout the United Kingdom, fired in an 8-in. military mortar, projected a 68-lb. ball to distances ranging from 43 to 250 ft.—with an average of 131·4 ft.—the number obtained from each individual sample being the mean of three experiments. But the powder used in the Westphalian experiments, containing 65 per cent. of nitre, 20 per cent. of sulphur, and 15 per cent. of charcoal (a composition that should never be used in any coal mine), would have been equal only to the worst of these. It is not just to condemn all powders for the demerits of the worst specimens. The low figures obtained with some samples are caused by deficiency of nitre, imperfect incorporation, and inferior quality of ingredients. A deficiency of nitre leads to the production of a larger and hotter volume of flame than there should be, because some of the carbon is only partially oxidised, and this, with a residue of fine charcoal, is expelled into the air, where it mingles with oxygen and burns. The heat thus evolved should have been utilised in splitting the rock. Imperfect incorporation leads to slowness of combustion, with low shattering power, and the emission of unburned powder from the bore-hole. Hence the use of inferior, or low-grade powders, fired with insufficient or no stemming, adds greatly to the risks of the miner. High-grade powder may be defined as one containing 75 per cent. of nitre, 15 per cent. of charcoal, and 10 per cent. of sulphur, the ingredients being as pure as possible, and the charcoal made from either dogwood, willow, or alder. The ingredients should be so incorporated and finished that a 2-oz. charge gives a minimum projection to the ball of 140 to 160 ft. A low-grade powder would then be one containing different proportions from the above, and giving a mortar result of under (say) 140 ft. A high-grade powder may be used with a charge weighing $\frac{7}{10}$ that of low-grade powder for a given effect, so that for a given length of bore-hole more stemming may be used, and hence there should be less smoke and less risk of a blown-out shot, while the greater rapidity of the explosion causes a greater liability to shatter and less tendency to expel the stemming. Moreover, the slight increase in the cost of the high-grade powder would be neutralised by its greater efficiency.

The difference between the two classes of powder was demonstrated by sprinkling $\frac{1}{4}$ oz. of powder on a wire gauze, which could be supported at any desired height above a plate carrying $\frac{1}{2}$ oz. of the powder to be tested; by firing successive charges on the plate with the gauze at varying distances from it, the minimum distances required to produce ignition of the upper charge could be determined for any powder. It was thus found that, whilst low-grade powders caused ignition at 51-in., high-grade blasting- and the best sporting-powders might safely be brought up to within 24 in. Further experiments were made with sheets of paper plastered over with gunpowder paste and then well-dried, and placed singly in front of a cannon in which a 3-oz. charge of high-grade powder was fired. It was thus found that the gunpowder-paper was ignited on firing the cannon without stemming, when it was placed at any distance less than 15 ft.; but with 8 in. of stemming no ignition was obtained beyond 3 ft. from the muzzle, and with 10 in. of stemming no ignition of the paper could be obtained at all. If even gunpowder cannot be ignited by a properly stemmed blown-out shot, it is unlikely that coal-dust or fire damp should thus be fired. Experiments made at the rock-face under actual working conditions have all failed to produce explosion of coal-dust when stemming has been used.

While submitting that it is grossly unfair to test gunpowder without the stemming that is necessary in practice, the author suggests the application of the following tests to powder and all other explosives, before placing them on the list of "permitted explosives," for use in coal-mines. (1.) All explosives, before test, to be exposed for at least two weeks to an atmosphere like that of an ordinary magazine. (2.) Gunpowder to be tested in a mortar, and not to be passed unless it gives a distance of at least 140 to 160 ft. (3.) Gunpowder to be guaranteed to contain at least 75 per cent. of nitre, and the ingredients to be perfectly pure. (4.) In testing gunpowder, the charges to be well stemmed. (5.) The weight of charge of gunpowder not to be very different from that of ammonium nitrate explosives. (6.) The amount of gunpowder charge to be regulated to that of nitro-glycerin explosives in the proportion of 1 of gunpowder to 3 for the actual amount of nitro-glycerin present. (7.) In exploding gunpowder from a steel-mortar in a testing-gallery, the cannon is not to have a smooth bore, as this would lead to the blowing-out of the stemming. The side of a bore-hole is necessarily rough.—W. G. M.

*Gunpowder, Some Defects in, as a Blasting-Agent.* W. J. Orsman. Trans. Mining Inst. of Scotland, 1897, **19**, 57—61.

The author considers that the principal dangers in using gunpowder in shot-firing lie in the heated solid products of combustion and the inflammable character of the gaseous portion. The addition of nitre in quantity sufficient to produce complete oxidation may overcome the latter difficulty, and at the same time prevent the formation of poisonous carbon monoxide, but it increases both the temperature produced on explosion, and the proportion of solid product. In such a case solid matter amounting to more than half the weight of the charge would be projected into the air at a temperature of 2,000° F. Again in coal, 2½ parts, and in rock, 3 parts of compressed powder are required to produce the same effect as 1 part of roburite. The advantage of a smokeless explosive may be shown by substituting nitre for ammonium nitrate in roburite, whereby a detonating explosive is obtained of equal strength, but producing smoke, and invariably setting fire to coal-dust when fired from a cannon suspended vertically. Hence the writer is convinced that the substitution of high-grade for low-grade gunpowder would not guarantee the degree of safety necessary in the working of coal mines.—W. G. M.

## PATENTS.

*Safety Matches and Rubbing Surfaces therefor, Impts. relating to the Manufacture of.* J. Craveri. Buenos Aires. Eng. Pat. 27,520, Nov. 23, 1897.

The process claimed is "for substituting entirely the use of amorphous phosphorus in the friction surface used with these matches by means of the compounds of sulphocyanogen of its polymers and isomers."

The paste for the rubbing surface may consist of cyanogen persulphide, 2 parts; persulphocyanic acid, 1 part; potassium xanthate, 0·5 part: antimony sulphide, 4 parts; gelatin, 1 part.

The heads of the matches are prepared with the following substances:—Sulphur, 3 parts; nitronaphthalene, 2 parts; potassium bichromate, 5 parts; potassium chlorate, 50 parts; pulverised glass, 30 parts; gelatin, 10 parts.

In each case the gelatin is dissolved in water and the other ingredients stirred in.—W. M.

*Explosive Compounds, An Improved Manufacture of.* A. Nobel, Paris. Eng. Pat. 27,197, Nov. 30, 1896.

"This invention relates to the manufacture of a smokeless compound, consisting of layers of different chemical compositions, which are of such a nature, and are so arranged, that the combustion is slowest in the outer layer and increases inwards."

"Charges, in the form of sheets, which are specially suitable for large guns, are obtained by providing a thick rolled leaf or sheet, which may be called A, of a composition of quick combustion, and two thinner rolled leaves, called B, of a slower burning composition. These leaves B are then placed one on each side of the leaf A, whereupon they are all rolled or pressed together in their plastic state, or joined together by applying a suitable solvent or glue, as, for instance, a solution of collodion in acetate of amyl or acetone, upon the surfaces which are to be in contact with each other." The sheets thus prepared are coiled up into the form of rolls, of a diameter suitable to the cartridges to be filled.

Charges for rifles may be produced in the form of tubes, the nozzle of the apparatus being arranged for simultaneously squeezing out several concentric layers of different compositions, which, owing to their plasticity, will appear as only one coherent mass.

The following compositions have been found to give good results:—

I. For A (the inner layer):—
60 per cent. of nitroglycerin.
40 per cent. of nitrocellulose.
For B (outer layer):—
45 per cent. of nitrocellulose.
45 per cent. of nitroglycerin.
10 per cent. of succinate of amylic oxide or phthalate of methylic oxide or other "moderant."
II. For A.—Same as above.
For B.—90 per cent. of composition A.
10 per cent. of powdered sugar or starch.
III. For A:—
80 per cent. of ballistite of following composition:—
50 per cent. of nitroglycerin.
45 per cent. of nitrocellulose.
5 per cent. of succinate of amyl.
20 per cent. of highly nitrated wood meal or pulverised nitrocellulose.
For B.—Ballistite of same composition as for A, but without aforesaid addition of nitrocellulose.
—W. M.

*Nitroglycerin, Impts. in Manufacture of.* G. Kynoch and Co., Ltd., and A. T. Cocking, Witton. Eng. Pat. 28,890, Dec. 16, 1896.

A method of mechanically providing for the flow necessary to convey the nitroglycerin liquors by gravity from one tank to another, consisting in providing hydraulic gear for raising a containing tank within the nitrating house to a sufficiently high level to allow its contents to flow to the next house.

An arrangement of sets of nitrating and washing houses and tanks, in such a manner that they may be interchanged, is also described and claimed.—W. M.

*Explosives, Impts. in or relating to.* W. Greaves and E. M. Hann, Aberaman, Wales. Eng. Pat. 651, Jan. 9, 1897.

It is proposed to add ammonium oxalate to explosives of the cordite class for the purpose of neutralising the acid products of their explosion, and also lessening the speed of explosion. Ammonium or magnesium carbonate is added to neutralise the effect of any tendency of the oxalate to become acid.—W. M.

*Matches, Impts. in and relating to Manufacture of.* A. J. Fredrikson, Jönkoping, Sweden. Eng. Pat. 2785, Feb. 2, 1897.

Wood is cut into veneer, then passed between grooved rollers and cut into match lengths. This treatment softens or loosens the outside of the wood, so that it becomes more absorbent of the stearin into which the match stems are dipped. They are afterwards tipped with the igniting material.—W. M.

*Matches, Compositions suitable for the Manufacture of.* L. Braly, Lyons, France. Eng. Pat. 21,195, March 30, 1897.

This patent is for "a paste for the manufacture of inexplosive matches without phosphorus, which light upon all surfaces. The paste consists in the combination, in suitable proportions, of a liquid glue, potassium chlorate, and a powder formed of a suitable mixture of pounded glass, hyposulphite of lead, plumbates of calcium and strontium, sulphur, metallic aluminium, sodium chloride, and monosulphide of calcium."—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

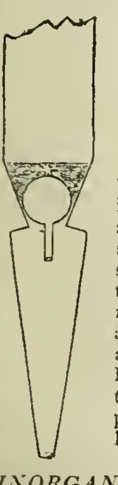
### *APPARATUS, ETC.*

*The Lamansky-Nobel Viscosimeter.* L. Singer. Chem. Rev. Fett- u. Harz-Ind. **4**, [18], 243—246.

This apparatus, which is used in Nobel's laboratory at Baku, consists of a water-bath, 285 mm. high and 115 mm. in diameter, fitted with an annular, perforated stirrer, a perforated steam coil, and a thermometer; also an oil cup, 300 mm. high, the diameter tapering from 50 mm. in the upper half to 12 mm. at the bottom, which is fitted with a screw piece containing the effluent aperture (1 mm. in diameter), closed by a metal valve. The lid of the oil cup fits air-tight, and is pierced by a copper tube 5 mm. in diameter, the lower end of which is exactly 200 mm. above the outflow. The oil cup is also provided with a thermometer.

The rate of outflow of the oil is compared with that of 100 c.c. of distilled water at 50° C. (*viz.*, 60 seconds) at the constant pressure of 200 mm., this pressure being secured by the aid of the aforesaid tube, by filling the oil cup (with water or oil) up to the height of the water-bath, then putting the lid on, and allowing the liquid to escape until the tube is empty. The results obtained in this manner are more accurate relatively than those from instruments wherein no such provision is made. It is necessary that the oil tested should be first freed from water by heating to 120° C., and from mechanical impurities by filtration. Oils incompletely refined by caustic soda cannot be tested until thoroughly purified.

As compared with the Engler instrument, the Lamansky-Nobel viscosimeter—according to Wischin—presents the advantage of constant pressure, uniformity of temperature, and the possibility of making several tests, with the same oil, in rapid succession. Of these, however, only the first-named is absolute, whilst, on the other hand, the new apparatus has several defects, such as the absence of a plumb-line or level, the length of the thermometer stem, and the weight ($4\frac{1}{2}$ kilos.), the difficulty of cleaning, and the steep pitch of the conical portion, all of which are, however, remediable. In general, the apparatus is easy to manipulate after a little practice, and, apart from the directions given for ensuring constancy of pressure, requires no special precaution in use. The author considers that it is well adapted to serve as the standard instrument for the viscosimetric examination of mineral oils, but recommends the acceleration of the rate of outflow to 30 seconds per 100 c.c. of water at 50° C.—C. S.



### *Asbestos Filter-Tube, An Improved.* A. Goske. Chem. Zeit. **22**, [4], 21.

The tube, which is made of combustion tubing, is drawn out at its lower end and constricted at the point where it begins to narrow, as is shown in the accompanying illustration. The small glass ball placed in the narrow part of the tube fits the tube closely except for two narrow lateral openings. Above this ball dry asbestos wool is lightly pressed down until a layer of about 5 mm. is formed. Another layer of asbestos is then lightly placed on this. Several quantities of water are now poured through, and the filter is finally dried. It is then ready for use.—W. P. S.

## *INORGANIC CHEMISTRY.—QUALITATIVE.*

### *Bromine, Detection of Traces of, in Mixtures by Means of Fluoresceïn.* H. Baubigny. Comptes Rend. 1897, **125**, [18], 654—657.

The test is based on the fact that fluoresceïn is transformed by bromine into its tetra-bromo derivative—eosin. Paper is steeped in a solution of pure fluoresceïn (40 to 50 per cent.) in acetic acid and allowed to dry. When moistened and brought into contact with bromine it shows a rose coloration. In using the test for bromides (in the absence of chlorides) the bromine is liberated by warming the salt or mixture in a solution containing copper sulphate 16 per cent., and potassium permanganate, 0·7 per cent., and passing a slow current of air through the flask. The formation of eosin after several minutes on the test paper applied to the delivery tube of the flask shows the presence of bromine. The test is not suitable for the detection of bromine in the presence of free chlorine, especially if the latter be present in large excess, since chlorine gives a white derivative with fluoresceïn, which masks the effect of the bromine. In such cases the free halogens may be converted into chloride and bromide, and then tested by treating the solution with copper sulphate and potassium permanganate, and passing air through the flask without warming. When chloride is present alone, the paper remains unchanged after 10 minutes, but if as little as 0·001 grm. of bromide be present with 5 to 10 grms. of chloride, rose-coloured streaks will appear within 4 or 5 minutes. The presence of an iodide does not interfere with the test if it is first transformed into iodate by warming the solution with a little permanganate, for iodine is not then liberated on subsequent addition of the copper sulphate. The author does not describe the method of proceeding in the case of insoluble bromides, as this is still the subject of study.—C. A. M.

## *INORGANIC CHEMISTRY.— QUANTITATIVE.*

### *Liquefied Ammonia, Methods of Analysis of.* A. Lange and W. Heffter. Chem. Ind. 1898, 2—7.

The authors criticise the methods in use, taking Bunte and Eitner's as representative of those in which the sample is drawn in a closed pipette (this Journal, 1897, 358), and Lange and Hertz's, in which it is brought into an open vessel (this Journal, 1897, 467).

The method consists in filling a glass pipette, which is provided at each end with a stop-cock, with ammonia from an iron cylinder and weighing. The pipette is then placed perpendicularly and connected at the upper end with a series of three weighed drying tubes containing caustic potash. The upper stop-cock is then opened so that the gaseous ammonia may escape in a slow stream through the drying tubes. As soon as all the ammonia has evaporated, the pipette is placed horizontally in an air bath, the temperature of which is gradually raised to 70°—80° C., and a slow current of air, freed from carbonic acid, is passed through.

They find that benzene, a mixture of acetonitrile and ethyl alcohol, and even pyridine, escape from the apparatus, very little of these being retained by the potash. The method therefore gives too low an estimate of the impurities.

Moreover, carbon dioxide and water cannot be determined by such means, since any alcohol and acetonitrile which is arrested by the potash, interferes.

Urban suggests leaving out the potash tubes and the current of air, and simply cooling to −30° the residue obtained by evaporation. This method is open to criticism, and difficulties arise in the final weighing of the residue.

Lange and Hertz's method was also examined, and found usually to give too high results, though the degree of accuracy varied according to the nature of the impurities.

The method of testing liquid ammonia in use in America and Australia consists in determining the boiling point of 200 grms. of a sample in an open vessel, a thermometer being fixed in the liquid. The boiling point must not be above − 28·6° F. or − 33·7° C. This method is not an accurate one, as it does not allow of a differentiation between products containing 1 and 0·1 per cent. impurity. Also the fact that during evaporation the thermometer indicates no perceptible rise in temperature, as it ought to do, seeing that the residual liquid becomes more and more impure, shows that the method leaves very much to be desired.

It thus appears that none of the methods which have been proposed is of value for accurate determinations. Lange said in his first publication that probably the only accurate method would be to fill a strongly cooled glass tube with the liquid, seal it hermetically, weigh it and determine the ammonia in the usual manner. The authors have tried such a determination with the help of the Bunte and Eitner pipette, but failed to obtain satisfactory results owing to the difficulty in manipulating the pipette, and partly perhaps owing to the disturbing influence of the pyridine present.

Liquefied ammonia is chiefly used for the production of cold in ice-machines, and though its efficiency varies directly with its state of purity, yet it is doubtful whether small variations in the amount of impurities would make any difference in the working of the machines. As Lange pointed out, the ammonia in course of circulation gradually becomes impurer and weaker, owing to the admixture of water from without, so that the possession of any very accurate method is not at present a matter of importance, yet in many cases it may be necessary to fix the limits of impurity and then, according to expediency, that method which was best suited to the case in question would be adopted. Lange and Hertz's method would give the "maximum," and Urban's method the "minimum" amount of impurities in any given sample. These two methods are the only feasible ones known at present. Though neither of them gives accurate results, yet, as the investigations of the authors have shown, when the mean of the numbers obtained by the two methods is taken, the result obtained is very nearly accurate. Many chemists would prefer Urban's method because by it the ammonia as it exists in the cylinder comes under investigation, but the great difficulties of manipulation connected with the use of the pipette would induce the majority to give the preference to the rougher method of Lange and Hertz.—E. H. T.

*Phosphoric Acid, New Method for the Determination of.* Woy. Zeits. für öffent. Chem. **3**, 321; Analyst, 1897, **22**, [261], 333.

On taking into consideration the variations induced by a large excess of molybdic acid, a large excess of nitric acid and the presence of ammonium nitrate, and also the statement of Hundeshagen that for the precipitation of phosphoric acid by ammonium molybdate, it is only needful for the molybdate to be present in the proportion 1 of $P_2O_5$ to 24 of $MoO_3$ and also that only a correspondingly small proportion of nitric acid and about 5 per cent. of ammonium nitrate are required, the author has come to the conclusion that the most efficient reagent is a 3 per cent. aqueous solution of ammonium molybdate, which always remains clear and may be relied upon at any time to contain the requisite amount of molybdenum. As certain chlorides are prejudicial to the precipitation, the raw phosphates are dissolved in sulphuric acid (taking care to prevent the gypsum formed from conglomerating), and the portion of the solution taken is neutralised with ammonia as far as is possible without causing a precipitate to form. (See also Analyst, 1897, [258], 250.)—A. S.

*Phosphorus, Rapid Estimation of [in Iron].* H. Wdowiszewski. Stahl u. Eisen, 1897, 814—815; Chem. Centr. 1897, **2**, [21], 1038.

It is stated that shaking is necessary in order to obtain complete precipitation of phosphorus as magnesium ammonium phosphate, and the following method, which only takes four hours, is recommended:—2 grms. of pig-iron are dissolved in 30 c.c. of nitric acid (sp. gr. 1·2), the solution is made up to 50 c.c. with water, and the silica and graphite filtered off. The filtrate is evaporated to its original volume, oxidised with permanganate, then hydrochloric acid is added, and after cooling it is neutralised with ammonia. The liquid is warmed to 60° C., molybdate solution added, and the whole shaken up for five minutes. The yellow precipitate is filtered off, washed with 10 per cent. ammonium nitrate solution, and dissolved in ammonia. The ammoniacal solution is neutralised with hydrochloric acid, a few c.c. of magnesia mixture, and ammonia in excess, added, and the closed flask, after standing ½ hour in ice or snow, is vigorously shaken for five minutes, and the solution immediately filtered. The precipitate is then washed and dried, &c. in the usual manner.—A. S.

*Lead, Fire Assay for.* J. F. Cannon. Eng. and Mining J. 1897, **64**, [21], 604.

About 20 grms. of a flux composed of 16 parts of sodium bicarbonate, 16 parts of potassium carbonate, 8 parts of borax glass and 5 parts of flour are placed in a "5 grm. crucible" and 5 grms. of the ore, weighed out carefully, are added and thoroughly mixed with the flux. The crucible is gently tapped to settle down its contents, and then 20 grms. more of the same flux added directly to the top. The crucible is again gently tapped and four or five iron nails added to the charge to take up the sulphur. The crucible is then transferred to the muffle furnace, which should be at a bright cherry red heat, and heated until effervescence ceases, after which the muffle door is closed, and a high degree of heat maintained until a perfectly fluid fusion is obtained. The charge is then withdrawn and the nails carefully taken out, tapping each one to free it from any adhering particles of lead. The crucible is tapped gently, and the contents poured out on to a level cast-iron plate, drawing the slag out to a thin point. The lead button should be found at the extreme end of the slag, and should leave it easily and cleanly.—A. S.

*Tinfoil, Determination of Lead and Antimony in.* A. Seyda. Zeits. für öffent. Chem. **3**, 364; Analyst, 1897, **22**, [261], 331.

*Lead Determination.*—10 grms. of the foil are dissolved in 25 per cent. hydrochloric acid, and 25 per cent. nitric acid is added to the hot solution to convert the stannous chloride into stannic chloride and to dissolve the antimony which remains undissolved by the hydrochloric acid. A slight excess of a 25 per cent. solution of soda is added gradually to the clear liquid, which is warmed and kept agitated until the precipitate at first formed, redissolves, leaving only a slight opalescence. A small quantity of precipitated sulphur is then added and sulphuretted hydrogen passed through until the liquid acquires a distinct yellow colour. After standing for 24 hours, the black precipitate is filtered off, and washed on the filter with sodium sulphide solution until a trial portion of the filtrate gives a pure white precipitate of sulphur on acidifying with hydrochloric acid; this washing takes a considerable time, but must be thoroughly carried out, or the tin will not be completely separated from the lead. The filter containing the moist precipitate is taken from the funnel, carefully heated with 25 per cent. nitric acid, then pressed together into a loose ball by the aid of a stirring rod, the ball replaced in the funnel and the nitric acid solution filtered through several times, until it becomes clear. The ball of paper is then washed, first with hot nitric acid and afterwards with hot water, until the washings no longer give a black precipitate with sulphuretted hydrogen. Any lead sulphate that may have formed, is thus completely removed from the filter. The filtrate is evaporated with about 20 c.c. of dilute sulphuric acid, and the lead sulphate is filtered off through a porcelain Gooch crucible, dried, and weighed.

*Antimony Determination.*—The following modification of Rose's method is used:—5 grms. of the foil cut into strips are dissolved, a portion at a time, in 100 c.c. of strong nitric acid and 25 c.c. of water. The metastannic acid formed, is washed into a porcelain basin with 25 per cent. nitric acid, the mixture evaporated to dryness and the residue dried in the water bath (the temperature of drying must not exceed 100° C., as the anhydrides of tin and antimony do not appear to combine with soda). The powder whilst still warm is added a little at a time to 60 grms. of fused caustic soda, and the whole thoroughly mixed. The fused mass is cooled to about 100° C., broken up, and dissolved by heating with 700 c.c. of water. The turbid liquid is cooled, mixed with one-third of its volume of 96 per cent. alcohol, let stand for 24 hours and the precipitate filtered off by decantation, the last portions being washed from the beaker by means of a mixture of two parts of alcohol to one of water. Instead of washing the precipitate with dilute alcohol, which causes a loss of some of the antimony, a second fusion with caustic soda is resorted to. The precipitate is dried at 100° C., detached from the paper, and the latter cut into strips and added to 10 grms. of fused caustic soda and a little sodium nitrate. When the paper is completely destroyed, the precipitate is added to the fused mass, and the fusion, &c. proceeded with as before. In order to ensure the insolubility of the sodium antimoniate, the liquid should contain about 10 per cent. of caustic soda and 25 per cent. of alcohol. The antimony is finally precipitated and weighed as $Sb_2S_3$.—A. S.

*Silver and Copper, Electrolytic Separation of.* N. Revay. Zeits. f. Elektrochem. **4**, (1898), 313—316.

These metals can be separated by electro-deposition of the silver from a solution containing salts of both of the metals; for silver salts require a smaller difference of potential for their deposition than the corresponding salts. The electrolysis may be carried out either in acid solution or in a solution of the double cyanide of potassium and silver.

A. *Deposition in Acid Solution.*—In the first experiments made, with an E.M.F. of 1·3 to 1·4 volts, the silver was deposited in a spongy form; but this difficulty was removed by warming the electrolyte. At a temperature of 70° the silver deposit, although not quite smooth, adheres well and can be easily washed and weighed. The E.M.F. should not exceed 1·4 volts. In order to avoid the formation of peroxide, the solution should be made strongly acid with nitric acid from the start. Estimations made with quantities of silver nitrate varying from 0·25 to 0·5 grm. (with approximately equal quantities of crystallised copper sulphate) gave as the percentage of silver present in the nitrate—(1), 63·43; (2), 63·47; (3), 63·45; and (4), 63·35.

B. *Deposition from Potassium Cyanide Solution.*—The separation by this method was first carried out by Smith and Fränkel, and has since been recommended by Heydenrich and by Freudenberg. Here it is more difficult to lay down a definite and constant E.M.F., for the difference of potential between the silver electrode and the solution varies greatly with the concentration of the silver salt and the potassium cyanide in the solution. Freudenberg states that the deposition of silver begins at 1·7 to 1·8 volts, whereas copper does not begin to be deposited until the E.M.F. exceeds 2·3 to 2·4 volts. The author considers the latter figures are somewhat too high. His experiments were made with E.M.F.'s of 1·6 to 1·8 volts, and at temperatures of 45° to 60°. The results were satisfactory, six determinations made with 0·23 to 1·63 grms. of silver nitrate giving percentages of silver varying between 63·37 and 63·45. The method is preferable to the first one, as it gives a more coherent deposit. It works best when the quantity of silver present is large compared with the copper; for then the respective E.M.F.'s required for deposition lie far apart. But it has the disadvantage that in the converse case they approach near to each other; and if much copper is present and very little silver, the values may reverse their order so that the copper comes down first. Hence the method must be used with care, for unless the proportion of copper present be small, the deposition of silver is never complete.—D. E. J.

*Antimony, Volumetric Process for the Determination of.* H. Causse. Comptes Rend. 1897, **125**, [25], 1100—1103.

ANTIMONY trioxide, free or combined, reduces iodic acid, $5Sb_2O_3 + 4HIO_3 = 5Sb_2O_5 + 2I_2 + 2H_2O$. The solutions required are as follows:—Iodic acid, 50 grms. in 250 c.c.; decinormal thiosulphate, 20 per cent. potassium iodide and starch paste. In the flask of a Fresenius' or Mohr's iodometric apparatus, 0·5—0·6 grm. of antimonious oxide is weighed, 20—25 c.c. of iodic acid added, and 10 c.c. of potassium iodide placed in the condensing apparatus. The contents of the flask are boiled until colourless, and the collected iodine determined with thiosulphate.

From antimony compounds, the sulphide is precipitated by sulphuretted hydrogen, then dissolved in hydrochloric acid, and the oxide obtained by the action of sodium carbonate. The halogen acids, sulphurous acid, and sulphuretted hydrogen must be completely removed, since these act on iodic acid. It is not necessary that the antimonious oxide should be dissolved, but for this purpose tartaric acid or potassium bitartrate may be employed; these substances are without action on iodic acid.

—A. C. W.

*Manganese, New Method of Determining.* F. Julian. Berg- u. Hüttenmänn. Zeit. 1897, **56**, 410.

A MANGANESE salt, heated with nitric acid and potassium chlorate, gives the whole of its manganese as dioxide; the amount of this is determined by dissolving it in excess of hydrogen peroxide of known strength, and titrating the excess of peroxide with standard potassium permanganate. The hydrogen peroxide is acidified with sulphuric acid, and made to correspond in strength with the permanganate solution. It must be standardised daily, which is done by heating 40 or 50 c.c. of nitric acid for a few minutes to expel nitrous acid, diluting with 200 c.c. of water, adding 15 to 25 c.c. of the hydrogen peroxide solution, and titrating with permanganate.

Samples of iron with differing carbon content require slightly different treatment. Of graphitic pig, 3 grms. are dissolved in 35 c.c. of strong hydrochloric acid and evaporated nearly to dryness, 50 c.c. of strong nitric acid are added, and the whole heated till brown vapours cease to come off; the cooled solution is made up to 100 c.c., 50 c.c. of this evaporated to a syrup, and then heated to boiling with 40 or 50 c.c. of strong nitric acid; 1 or 2 grms. of potassium chlorate are added, and boiling continued for 5 or 10 minutes; the solution is now diluted with water, and titrated with hydrogen peroxide and permanganate. Of manganiferous pig containing but little silicon or graphite, 0·5 grm. is dissolved in 15 c.c. of hydrochloric acid, evaporated, boiled with 50 to 60 c.c. of nitric acid, then potassium chlorate is added and the analysis finished as above. Of manganiferous steel or wrought iron, 1·5 to 5 grms. of borings are dissolved in 25 c.c. of hydrochloric acid, evaporated, boiled with 40 to 50 c.c. of nitric acid, and the operation finished as above.—J. T. D.

*Stannic Salts, Estimation of Tin in.* A. Fraenkel and J. Fasal. Mitt. d. k. k. tech. Gewerbe-Museums in Wien, 1897, **7**, [9, 10, and 11], 227—229.

THE authors have devised a method for estimating tin by precipitating the metal on to aluminium, dissolving both metals in hydrochloric acid and titrating the stannous chloride formed with iodine or potassium bichromate, but in the latter case the greater part of the hydrochloric acid must be neutralised first. The following method is recommended. The stannic salt solution containing 0·2—0·4 grm. of tin is treated in a flask with a few drops of concentrated hydrochloric acid and about 0·5—1·0 grm. of aluminium wire. On gentle heating, the tin is precipitated, this taking about half an hour, when 10 c.c. of concentrated hydrochloric acid are added and the whole is warmed up until the tin is dissolved and no further evolution of hydrogen takes place; this requires about 15 minutes. The titration is carried out with iodine, the solution being made alkaline with sodium bicarbonate in presence of Rochelle salt. The following results were obtained: I. containing 0·4317 grm., and II., 0·3988 grm. of tin estimated gravimetrically:—

| — | I. | II. |
|---|---|---|
| | Grm. Sn. | Grm. Sn. |
| 1 | 0·4105 | 0·3866 |
| 2 | 0·4103 | 0·3875 |
| 3 | 0·4098 | .. |

With a view of determining the error between the amount actually present and that found by this method, a weighed quantity of tin (2·8302 grm.) was dissolved in hydrochloric acid in a current of carbonic acid, made up to 250 c.c., and 25 c.c. titrated as above. An amount of a further 25 c.c. was oxidised, precipitated with sodium sulphate or ammonium nitrate, and weighed.

| | Grm. Sn. |
|---|---|
| Titration with iodine (1)................ | 0·2765 |
| „ „ „ (2)................ | 0·2767 |
| „ „ potassium bichromate .. | 0·2736 |
| Precipitation with sodium sulphate.... | 0·2870 |
| „ „ ammonium nitrate . | 0·2844 |

The amount actually present was 0·2830 grm. of Sn. The method is sufficiently accurate for technical purposes, more especially in making comparative analyses.—T. A. L.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

*Native Asphalt, Detection of Coal-Tar Distillation Products in.* J. Bengough. Mitt. k. k. tech. Gewerbe-Museums in Wien, 1897, **7**, [9, 10, and 11], 217—218.

THE method already given by Böckmann consists in pouring 100 c.c. of alcohol (36°) on to 10 grms. of the asphalt and allowing it to stand 24 hours. Native bitumen does not colour the alcohol, or at any rate only very slightly, whilst if coal-tar distillation products be present a yellowish colour is produced. It has been found that this method is only applicable when the lower boiling portions have been added and the addition of hard pitch cannot be detected by this process. According to Durand Claye, 1 grm. of the sample is shaken with 5 c.c. of benzene in a closed glass vessel until dissolved. A few drops of the filtered solution are then diluted with 5 c.c. of benzene and mixed with an equal volume of alcohol (85° Gay-Lussac). The yellow or yellowish-brown coloration of the alcoholic layer gives an indication of the presence of tar distillation products. Even on dissolving in benzene some indication can be obtained, since native asphalt dissolves with a pure brown colour, whilst tar products give a strongly yellowish-green fluorescence. A second method depends upon the action of sulphuric acid on the bitumen. The sample is warmed and cooled several times so as to make it brittle, it is then powdered and 0·1 grm. of the powder is mixed with 5 c.c. of concentrated sulphuric acid and allowed to stand 24 hours. The solution is then diluted carefully by pouring into cold water and is filtered into a 200-c.c. flask, the wash water from the residue being also added. Pure bitumen gives scarcely any colour, whereas the presence of tar products produces a deep brown to black solution of the sulphuric acids. The residual tar from mineral oil works behaves like native bitumen. It is not dissolved by 36 alcohol, it gives a non-fluorescent benzene solution and an almost colourless filtrate is obtained in the sulphonation process. The amount of sulphur in asphalt is no criterion of its purity since it is frequently very small in quantity. A sample of residual tar from a mineral oil factory contained 2·88 per cent. of sulphur.—T. A. L.

### *Silk, Identification of Artificial.* P. Truchot. Ann. de Chim. Anal; through Leipziger Färber- und Zeugdr. Zeit. 1897, **46**, [12], 535—536.

Lustro-cellulose is distinguished from natural silk by the following reactions and properties:—

1. The density of lustro-cellulose is 1·490, that of silk varying from 1·357 to 1·37.

2. When ignited, lustro-cellulose burns without the emission of any acid gases or smell.

3. When treated with Schweitzer's reagent, lustro-cellulose and silk both swell considerably before dissolving. On the addition of water or hydrochloric acid, to the solution obtained from the lustro-cellulose, a white precipitate of cellulose is produced.

4. Lustro-cellulose dissolves in concentrated sulphuric acid with a deep yellow colour, which, on the addition of diphenylamine or of brucine, turns respectively dark blue or red. These colour changes are very characteristic, and are due to the presence in the fibre of nitro-cellulose which has escaped reduction by the compounds with which it has been treated. The test may be made without dissolving the fibre, the latter being simply immersed in a solution of diphenylamine sulphate, when it becomes coloured dark blue, natural silk, when similarly treated, remaining colourless.

5. A simple test for lustro-cellulose consists in separating the fibres, moistening them with water, and determining their tenacity. The fibres of collodion silk when moistened have their outer coating dissolved and become very weak, whilst silk, whether moist or dry, withstands great tensile strain.—E. B.

### *Schiff's Reaction applied to some Substituted Magentas.* Bull. Soc. Chim. 1897, **17**, 998—999.

In a previous paper (Bull. Soc. Chim. **17**, 136; this Journal, 1897, 360), it has been shown that if Acid Magenta be decolorised by sulphurous acid, the colour is not restored by aldehydes. On the other hand, certain homologues of Magenta, such as *o*-tolyl and xylyl Magenta, are very sensitive to aldehydes after having been decolorised by sulphurous acid, although the colour obtained is more bluish than with ordinary Magenta. The author has further studied the behaviour of the following derivatives: — Tetraethylmethylphenylrosaniline hydrochloride and its monosulphonic acid, and Diethyldimethyldiphenylpararosaniline hydrochloride and its disulphonic acid. Employing the same proportions as already given, it is found that these dyestuffs are decolorised by sulphurous acid, although slowly. The Diethyldimethyldiphenylpararosaniline, however, gives a slight blue insoluble deposit, whilst its monosulphonic acid remains tinted a light greenish shade. The addition of an appreciable amount of aldehyde produces no change, whilst a large excess, probably saturating the sulphurous acid, causes a gradual reappearance of the original blue colour, but just like Acid Magenta, these dyestuffs cannot be said to give Schiff's reaction, which is so sensitive in the case of ordinary Magenta and some of its homologues.—T. A. L.

### *Food Substances, A Method for the Microscopical Examination of.* A. Hebebrand. Forschungs Ber. 1897, IV. [11], 306—307.

The author takes exception to the usual reagents employed for preparing food substances for microscopical examination (*e.g.* aqua regia and soda lye), on the ground that their action is often too energetic, and causes the amount of foreign material to be different in the residue from what it was in the original substance. If, on the other hand, weaker reagents are used, the structure does not appear with sufficient clearness. In their place he proposes the use of chlorine in alkaline solution. The finely divided material is suspended in 10 to 15 c.c. of a solution of soda (7 grms. of dry carbonate in 100 c.c.), whilst a current of chlorine is passed in for from 2 to 10 minutes, care being taken that the liquid remains alkaline. The bleached substance is washed twice with water, and separated from the liquid with the help of centrifugal force. The method is said to give especially good results in the case of starches, and has occasionally enabled the author to make a quantitative determination of rye meal mixed with wheat meal, owing to the former being much more rapidly bleached by chlorine than the latter. But quantitative results cannot always be relied upon.—C. A. M.

### *Milk, Indigocarmine as a Test of Freshness of.* L. Vaudin. Répert. Pharm. 1897 [3], **9**, 538.

A few drops of indigo carmine are added to the sample of milk till it is tinted pale-blue. This tint gradually disappears, the more rapidly as the number of developed bacteria (*i.e.*, as the age of the milk) is greater. Temperature also affects the rapidity of the disappearance of the colour. With fresh milk the colour should remain at least 12 hours at 15° C., 8 hours at 15°—20° C., 4 hours at temperatures above 20° C.—J. T. D.

### *Margarine and Butter, Detection of Sesamé Oil in.* P. Soltsien. Pharm. Zeit. 1897, **42**, 837.

In using furfurol and hydrochloric acid, or the German official alcoholic solution of furfurol and hydrochloric acid, a red colour is produced, slowly in the cold but more rapidly on warming, very similar to that caused by a trace of sesamé oil. It differs from the latter in that (*a*) it arises more slowly, (*b*) it soon passes through a permanganate-like tint to one more inclined to blue, which is fairly stable.

—J. T. D.

### *Digitonin Reaction.* C. C. Keller. Ber. Pharm. Ges. 1897, **7**, 470.

The following reaction is given by digitonin alone, not by digitalin, digitoxin, digitaleïn, nor digitin:—0·005 to 0·01 grm. is dissolved in a large test tube in 5 c.c. of hydrochloric acid of sp. gr. 1·19, and is then heated in a water-bath for 5 minutes. The solution becomes yellow, bright red, deep garnet red, lastly purplish. When cooled and diluted with 20 c.c. of water, the liquid becomes blue with a red fluorescence; this soon disappears and a floccular precipitate forms.—J. T. D.

### *Acetaldehyde, a Colour Reaction for.* L. Simon. Comptes Rend. 1897, **125**, [25], 1105—1107.

A large number of aldehydes and ketones give a red coloration with sodium nitroprusside and potash (Legal's reaction). The author finds that ordinary aldehyde in dilute aqueous solution, on the addition of a few drops of aqueous trimethylamine, followed by a few drops of very dilute nitroprusside, gives a fine blue coloration. This coloration is very clearly visible with aldehyde solutions of

0·01 per cent. strength; it is, however, fugitve, disappearing in about 15 minutes. The reaction is not given by other aldehydes or ketones. Pure acetone affords a red coloration, which is completely masked by the blue given by 0·1 per cent. of aldehyde.

Pyruvic acid and its derivatives give a blue coloration with ammonia and the amines in presence of sodium nitroprusside; ammonia does not give this coloration with aldehyde, and even causes it to disappear when already formed. On the addition of potash to the blue solution given by aldehyde, the red coloration of Legal's reaction is obtained.—A. C. W.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Nitrogen, Estimation of.* [*Kjeldahl-Gunning Method.*] K. Wedemeyer. Chem. Zeit. **22**, [4], 21.

The author has tried this method on a number of nitrogenous organic substances. 20 c.c. of sulphuric acid were used in each estimation, with the addition of a little phosphorus pentoxide and mercury, and 10 grms. of hydrogen potassium sulphate. (When substances are being treated which froth very much, the hydrogen potassium sulphate should be added after the frothing is over.) Very accurate results were obtained, and the time for complete oxidation in no case exceeded 30 minutes. The addition of copper sulphate was found to increase the time required for oxidation.—W. P. S.

### *Bromine, Chlorine, and Iodine in Organic Substances, Direct Estimation of. Separation of Bromine from Chlorine by Means of Permanganate.* P. Jannasch and E. Koelitz. Zeits. für anorg. Chem. 1897, **15**, 68 and 66; Analyst, 1897, **22**, [261], 330.

The halogens are separated from the organic compound in the form of silver salts by a suitable method, and the latter are collected on a filter, washed, and, together with the paper, placed in a silver crucible, and fused with 5 or 6 grms. of pure caustic soda. The melt is extracted with warm water, the silver filtered off, the solution acidified with sulphuric or nitric acid, and the iodine, bromine, and chlorine finally separated from one another by thallium sulphate (I from Cl), or by the processes of distillation with acetic acid and sodium nitrite (I from Br and Cl), and with acetic acid and potassium permanganate (Br from Cl), which have been already described by Jannasch and Aschoff (this Journal, 1892, 845).

It is stated that the separation of Br. from Cl by the action of acetic acid and permanganate cannot be effected in the presence of much sodium acetate, as the latter body almost entirely prevents the evolution of free bromine. The original solution, if alkaline, should therefore be neutralised with nitric or sulphuric acid before the acetic acid required in the distillation is introduced.—A. S.

### *Fehling's Solution, Spontaneous Reduction of, when Partially Neutralised.* M. Siegfried. Ber. 1897, **30**, 3133–3134.

Referring to two recent communications by Jovitschitsch and Gerock (this Journal, 1897, 1046, and 1898, 75), the author remarks that his observations concerning the action of mineral acids on Fehling's solution have been misinterpreted. The spontaneous precipitation of $Cu_2O$, which occurs when partially neutralised Fehling's solution is boiled, is due simply to the withdrawal of the alkali necessary for securing stability of the solution, and not to any direct reducing action of the added acid, or of the salt formed. Sugar solutions, prepared by the action of an acid on starchy materials, for example, should therefore be neutralised prior to further treatment.—H. T. P.

### *Lubricants, Valuation of Mineral.* R. Kissling. Chem. Zeit. **22**, [4], 22—23.

When dealing with lubricating compounds consisting of mixtures of mineral oil and lime soaps, the author, besides estimating the inorganic salts, taking the viscosity number, and determining the temperatures at which the mixture begins to flow and is completely fluid, finds it advisable to separate the various constituents of the mixture. For this purpose, 100 c.c. (accurately weighed) of the oil are heated in a water-bath to 90° with 10 c.c. of hydrochloric acid, and well shaken for at least five minutes. The acid solution containing the mineral salts is drawn off. The oils left behind are saponified, and the unsaponifiable matter separated in the usual way.—W. P. S.

### *Molasses from Cane-Sugar, Analysis of.* H. Pellet. Bull. de l'Assoc. des Chim. 1897, **15**, [6], 605—626.

The author reiterates the disadvantages resulting from the use of basic lead acetate in analysing molasses containing reducing sugars (this Journal, 1896, 831), and recommends for this purpose neutral lead acetate, as was done by C. Haughton Gill and others.

To prepare the solution of neutral lead acetate, 300 grms. of the salt are dissolved in a litre of water. There is always a slight turbidity, and often a little oxide of lead precipitates. To the distinctly alkaline solution acetic acid is added until it is neutral to litmus. The liquid is then filtered, and should mark 22° to 23° B.

As neutral lead acetate decolorises much less than basic lead acetate, an intense light is required, and that of acetylene is almost indispensable for the polarisation.

To estimate raffinose, he advises the destruction of levulose and dextrose by heating with potash for 15 minutes to 100° C. on a water-bath. The liquid is then neutralised by acetic acid, and basic lead acetate added until no further precipitate falls. The polariscope reading requires a slight correction according to the author, although Commerson and Laugier state that the acids formed have no action on polarised light, and are for the most part precipitated by basic lead acetate. The half-normal weight in 50 c.c. is heated with 5 grms. of potash, then neutralised with 7·5 grms. of glacial acetic acid, treated with basic lead acetate, made up to 200 c.c., and polarised.

From the various experiments, of which detailed results are given in the paper, and a great number of others made on cane-juice direct, the author feels justified in drawing the following conclusions as to the nature of the reducing sugars contained in cane molasses.

The reducing bodies contained in cane molasses are formed of levulose and dextrose in equal or unequal proportions. According to the cane juice worked, and the method used, the reducing bodies may contain more, as much as, or a less quantity of levulose than of dextrose.

The juice contained in the upper part of the canes appears to contain more levulose than that of the lower part, which contains apparently more dextrose.

In ripe cane, as has been remarked by Prinsen Geerligs, the quantity of levulose diminishes, but according to the author, does not completely disappear, since even in very ripe canes, the upper part contains reducing substances partly composed of levulose, if lead acetate be used in analysing the juice. If basic lead acetate be used with cane containing only a small quantity of reducing sugar, a direct polarisation is obtained almost equal, or often quite equal to that obtained by inversion.

Molasses contains in addition invert sugar formed during manufacture, and may reach 10 to 30 per cent. in amount.

The rotatory power of the reducing bodies should be calculated after taking account of raffinose, if it is present.

In analysing molasses when basic lead acetate is used, the real crystallisable sugar by inversion is obtained, but not the exact direct polarisation (raffinose being absent). The reason has been explained. A little levulose, and even of dextrose may be precipitated, although in small proportion, and the rotatory power is generally taken *too low*. This is because the levulose polarises more to the right owing to the influence of the basic lead acetate, and because some levulose is precipitated. Also, the calculation usually gives too much dextrose in proportion to the levulose. Following the conditions obtaining in practice, the author finds that in the presence of salts from 15 to 35 per cent. of the levulose present may be precipitated by basic lead acetate.

In contradiction of statements that have been made, we shall see that the reducing bodies may be fermented.

The possible presence of raffinose may explain why, in certain fermented cane molasses, more reducing sugars are found than in others, this product fermenting with difficulty under ordinary conditions, and leaving reducing substances.—L. J. de W.

*Beer and other Liquids containing Acid Phosphates, Determination of Acidity in.* A. Ott. Zeits. für d. ges. Brauwesen, **20**, [43], 549—552; [45], 579—583; [46], 593—596.

Of the various methods employed for freeing beer from carbon dioxide before titration, that of passing a current of air (previously freed from carbon dioxide) through it, is the most efficacious; but the ordinary procedure of warming the liquid up to 40°—50° C. leads to results which are in excess of the truth, on account of the incomplete removal of the carbon dioxide.

The error due to the influence of $CO_2$ on the test paper is greater in the case of violet-blue litmus than with violet-red litmus, and is greatest of all with perfectly blue litmus.

The results of the acidimetric examination of beer depend greatly, other conditions being equal, on the colour of the litmus paper employed, more normal alkali being required to neutralise the liquid when violet-blue paper is used, than when violet-red paper is used. Since both papers act with equal efficiency towards acidified water, the difference recorded is not due to unequal sensitiveness, but to some other cause. The total acidity of beer and other liquids containing acid phosphates cannot be determined with violet (so-called neutral) litmus paper, since the acid constituents of the liquid are saturated with alkali. Moreover, by reason of the lack of uniformity of shade in the "violet" test paper generally used, the results obtained therewith are not comparative.

Amphoteric reaction of beer during titration with normal alkali:—This is due to the presence of primary potassium phosphate and its gradual conversion by alkali into the alkaline secondary phosphate; it must obtain until that conversion is complete. The progress of this transformation can be estimated in solutions of primary phosphate by means of litmus indicators, standardised according to the number of c.c. of normal alkali required to produce a neutral reaction in each case in presence of a solution of 1·3583 grms. of mono-potassium phosphate. For example, if the addition of 6·3 c.c. of normal alkali to the solution gave a neutral reaction with blue-violet litmus paper, it would indicate the conversion of 63 per cent. of primary into secondary phosphate, and the "neutralisation value" of the test paper would be 63. Hence, by preparing a series of standard test papers it is possible to determine the amount of primary phosphate or free phosphoric acid in such a solution. The ratio of primary to secondary phosphate could then be determined either by a single paper to which the given liquid has a neutral reaction, or by two papers of different neutralisation values, the one to indicate the acidity and the other the alkalinity of the amphoteric liquid. Thus, if 6 c.c. of N-sulphuric acid give with one-half of the solution a neutral reaction with a No. 33 paper, and 2 c.c. of N-alkali be required for producing the same effect on a No. 57 paper with the remainder, then the ratio would be:—$33 + (57 - 33)\frac{3}{4} = 33 + 18 = 51$ per cent. of secondary phosphate, and 49 per cent. of primary phosphate.

Similarly the presence of free acid (*e.g.*, lactic acid) and primary phosphate can be determined by ascertaining the amount of N-alkali necessary to render the solution neutral to two papers of different neutralisation value, the difference in the two quantities being the measure of the primary phosphate, and the difference between this result and the number of the lowest standard paper used, giving the free acid. Example: For a No. 29 paper, 3·7 c.c. of N-alkali were required, whilst No. 63 paper required 7·1 c.c. Difference = 3·4 c.c. of N-alkali = 1·3583 grms. of primary potassium phosphate. Difference between 3·4 c.c. and 2·9 (= Paper, No. 29) = 0·8 c.c. N-alkali required to neutralise the free acid present. Where the amount of primary phosphate is known, the free acid can be determined with one test paper, by deducting from the N-alkali required to give a neutral reaction, the number of c.c. corresponding to the phosphate present; the remainder will give the acidity in terms of N-alkali. For this purpose a red test paper of low number (*e.g.*, 5) is preferable to violet-red, being more sensitive.—C. S.

*Beer, The Estimation of Neutralisers added to.* E. Spaeth. Zeits. für angew. Chem. 1898, 4—5.

500 c.c. of the beer to be examined are freed from carbonic acid, mixed with 100 c.c. of ammonia, and allowed to stand for five hours.

One tenth of the filtrate is evaporated and the residue ignited, and the phosphoric acid in this determined.

Five tenths of the filtrate are mixed with 25 c.c. of a solution of lead acetate, and well agitated for 5—6 hours. An aliquot portion of the filtrate from this is evaporated to a small bulk, diluted to its original volume, acidified with a few drops of acetic acid, and saturated with sulphuretted hydrogen. After removal of the excess of the gas by a current of air, the filtrate is evaporated and the residue incinerated. The amount of alkali in the ash is then determined. The considerations which led the author to devise this method are as follows:—The addition of the ammonia causes the precipitation of the lime and magnesia as phosphates. When lead is added to the filtrate, the remainder of the phosphoric acid is separated. The determination of the phosphates, after the separation of the lime and magnesium phosphates, indicates the amount of alkaline phosphate, which may be considered as primary potassium phosphate. This, in a normal beer, should correspond with the amount of alkali found in the ash after the separation of the phosphoric acid. If more alkali than this be found, neutralising agents have been added; $0·01\ P_2O_5 = 0·0191\ KH_2PO_4 = 1·4$ c.c. $\frac{1}{10}$N acid.

The author has tested this method and finds that the amount of phosphoric acid usually indicates rather more deci-normal acid than the ash requires, but the agreement is sufficiently close for practical purposes.—A. L. S.

*Casein in Milk, A New Process for the Determination of.* G. Denigès. J. Pharm. Chim. 1898, **7**, [1], 9—12.

In a former paper (this Journal, 1896, 832) the author described a volumetric process for the determination of casein in milk, he now gives full experimental details of a process amended to obviate uncertainties due to the lime salts present. Place 25 c.c. of the milk in a 200 c.c. flask, add 5 c.c. of cold saturated ammonium oxalate solution, then 20 c.c. of $\frac{1}{10}$N mercury potassium iodide and 2 c.c. of acetic acid, make up to the mark, filter, returning the first runnings to the filter. Take 100 c.c. of the filtrate, add 10 c.c. of potassium cyanide solution, equivalent to the $\frac{1}{10}$N silver nitrate to be employed, then 12—15 c.c. of ammonia and add drop by drop with agitation the silver nitrate solution until a permanent precipitate is formed. Suppose $q$ c.c. are used. Perform a similar experiment with 100 c.c. of water, 10 c.c. of the same potassium cyanide, 12—15 c.c. of ammonia, and 10 c.c. of mercury potassium iodide. Let $c$ c.c. of silver nitrate be required. The author gives a table from which the casein content of the milk may be obtained from the value of $(q-c)$. It may also be calculated with sufficient approximation as follows:—When $(q-c)$ lies between 9 and 24, the casein expressed in grammes per litre is given by $q-c-2$. When $(q-c)$ lies between 25 and 32, the expression to be used is $1·25(q-c)-8$. And when $(q-c)$ is equal to or greater than 33, $2(q-c)-33$ must be employed. If $(q-c)$ is greater than 37, it is better to employ half the quantity of milk in the estimation.—A. C. W.

*Hydroxylamine and Hydrazine, Volumetric and Gasometric Determination of.* K. A. Hofmann and F. Küspert. Ber. **31**, [1], 64—67.

The method is based on the oxidation of hydroxylamine or hydrazine solutions by vanadic sulphate, measurement of the nitrogen evolved, and titration with permanganate of the vanadious salt remaining. The equations—

$$2NH_3O + O = N_2 + 3H_2O$$
$$N_2H_4 + 2O = N_2 + 2H_2O$$

represent the reactions taking place. The back-titration with permanganate affords a check on the gas measurement when pure substances are being dealt with, whilst the oxygen equivalent of any other reducing substances is obtainable from the difference which then exists between the two results. The proportions of the two bodies in a mixture of hydroxylamine and hydrazine compounds can be determined by considering the ratio between the nitrogen evolved and the oxygen absorbed during the reaction.

Pure ammonium metavanadate is dissolved in strong sulphuric acid (5 grms. in 50 c.c.), cooling during the action, and the solution diluted. The authors have used solutions containing 12·27 grms. and 6·48 grms. of metavanadate per litre, and obtained concordant results. The substance to be analysed is dissolved in dilute sulphuric acid, introduced into a fractionating flask provided with a tap-funnel and a leading tube for carbon dioxide, and connected with a nitrometer; and vanadium solution is added till the green colour produced is permanent. In about 20 minutes the evolution of gas at the ordinary temperature ceases, and the liquid is then heated to 60° C.; should the green colour go, more vanadium solution is added. Lastly, the solution is rinsed into a porcelain basin, diluted with water till the colour is bright blue-green, and titrated with permanganate.—J. T. D.

*Leather, Analyses of, at Tharand.* A. Bartel. Dingler's Polyt. J. **305**, Hefte 3, 4, 5, 6, 7, and 8, 65—192.

*See under* XIV., *page* 164.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Gases prepared by Electrolysis, The Reputed Active Condition of.* F. Winteler. Zeits. f. Elektrochem. **4**, [14], 342.

The author points out a danger connected with the use of gas voltameters. When the oxygen and hydrogen are collected together in one tube it may happen, when the tube is nearly full of the explosive mixture, that the electrodes project into the gas. If the electrodes possess the power of occluding gases they may cause an error by inducing the slow recombination of the gases, or, the recombination may be so rapid as to produce an explosion. In any case this is due to the occlusion of the gases by the electrodes, and not to any special activity of the gases.

Reference is also made to another source of error in voltameters, *viz.*, the solubility of the oxygen in the electrolyte, and its diffusion to the cathode, where it is again reduced.—J. S.

*Strontium Sulphide, duration of the Phosphorescent Power of.* J. R. Monrelo. Comptes Rend. 1897, **125**, [25], 1098—1100. (See this Journal, 1897, 642 and 1050.)

The following table contains the results of observations on the phosphorescence of strontium sulphide, prepared according to different methods, the order is that of increasing phosphorescent power.

| — | A. | B. |
|---|---|---|
| Reduction of $SrSO_4$ by C .. | 3 hours | 2 minutes |
| „ $SrCO_3$ by S... | 3 „ | 1½ „ |
| Action of $H_2S$ on SrO ...... | 5 „ | 1 „ |
| Verneuil's method ......... | 6 „ | ½ „ |
| „ „ author's modification. | 12 „<br>(still feeble phosphorescence.) | 1 second |

In column A is given the duration of phosphorescence after exposure of the specimen enclosed in a sealed tube to diffused light for 15 minutes, column B gives the minimum period of exposure to diffused daylight required to excite phosphorescence.

Exposure to direct sunlight excites a somewhat feebler phosphorescence, but a much shorter exposure is required.
—A. C. W.

*Chromous Oxide and Soda, A Double Carbonate of.* G. Baugé. Comptes Rend. 1897, **125**, [26], 1177—1180.

A tepid solution of sodium carbonate in boiled water is introduced into a flask containing chromous acetate, which dissolves in it. The double salt settles out, and is washed in succession with cold boiled water, alcohol (98 per cent.), and similar alcohol saturated with carbonic acid. After removal of alcohol by aspiration, the salt is sealed in tubes filled with carbonic acid. All the operations are carried out in a stream of that gas. The double salt so prepared is a brownish-red powder, efflorescent, and at first readily soluble in water, but the solubility diminishes in course of time, probably through polymerization. It has the formula $CO_3CrCO_3Na_2.10H_2O$, but loses water in a vacuum, and at 100° is converted into the salt $CO_3CrCO_3Na_2.H_2O$, which is a yellow powder. Both salts oxidise in moist air, and dissolve in dilute hydrochloric and sulphuric acids to blue solutions. They act as powerful reducing agents, and decompose water at 100°, liberating hydrogen and furnishing an interesting compound, which will be described in a later communication.—J. A. B.

*Sodium Thiosulphate; Action on Sulphates, Nitrates, and Metals.* J. Girard. Rev. Chim. Ind. 1897, **8**, 361.

*Action on Sulphates, Nitrates, &c.*—Nearly all metallic sulphates are reduced by sodium thiosulphate at temperatures ranging between 70° and 85°, and the precipitates consist generally of sulphides. Aluminium yields a mixture of aluminium and free sulphur, with perhaps some traces of its little known sulphide. In the case of copper, several intermediate products are formed, which vary in composition according to the temperature of reaction. If two molecules of thiosulphate are added to one of copper sulphate in the cold, $Na_2S_2O_3.Cu_2S_2O_3.3Aq$ results; if the solutions are mixed together at 40°, the temperature rises to 50° or 52°, and yellow crystals, $2Na_2S_2O_3.3Cu_2S_2O_3.8Aq$ are produced, which split up on boiling into cuprous sulphide, sulphur, sodium sulphate, sulphurous anhydride, and water. By taking 8 mols. of the sodium and 6 of the copper salt, the solution of the latter is decolorised, and a yellow anhydrous $2Na_2S_2O_3.3Cu_2S_2O_3$, is formed. With hot liquids a brown precipitate, $Cu_2S_2O_3.Na_2S_2O_3.CuS.4Aq$ (Siewert's yellow salt), is obtained; but the cupric sulphide is not actually combined with the double thiosulphate. Heated to 100°, this substance yields cupric sulphide, sodium sulphate, and sulphurous anhydride. At 70° or 72°, whatever proportions are taken, the precipitate consists mainly of cuprous sulphide, but cupric sulphide and traces of oxide are also present; and the final reaction corresponds to the equation

$$4Na_2S_2O_3 + 2CuSO_4 = Cu_2S + 2SO_2 + 4Na_2SO_4 + 3S.$$

Sodium thiosulphate dissolves cuprous hydrate to a colourless liquid, from which cuprous sulphide is soon thrown down. The acetate is easily decomposed at 40° or 45°, and yields cupric sulphide with a little oxide. In presence of stannous chloride, the acetate is reduced to cuprous oxide. Thiosulphate that has partly decomposed into sulphite produces the colourless cuprous sulphite.

On adding a crystal of sodium thiosulphate to a solution of silver nitrate, the metal is reduced in a very fine state of subdivision; and an imperfect coating may be deposited on sheet copper in this way. If, however, the silver solution is previously treated with stannous chloride, and then the thiosulphate introduced (care being taken to avoid excess) a perfect film can be obtained on copper or nickel. The result of the two reactions may be combined as follows:—

$$Na_2S_2O_3 + 2AgNO_3 + SnCl_2 =$$
$$Ag_2 + 2NaCl + Sn(SO_4)_2 + N_2O.$$

Mercurous sulphate is quickly reduced, at 45° or 50°, to the metallic state; but mercuric sulphate is more stable, yielding a whitish precipitate, which rapidly turns black, unless the sulphate is in excess. Mercuric chloride is reduced to calomel.

*Action on Metals.*—According to Puvoznick, metallic copper is attacked by sodium thiosulphate with the formation of cuprous (and possibly some cupric) sulphide; other authorities think that the reaction only takes place when the sodium salt is melted in its own water of crystallisation. The present author finds that unless the surface of the copper has been thoroughly freed from grease, there is no action at low temperatures if the solution be moderately strong; even at 60° it is very slow. Brass is not attacked by hot thiosulphate; silvered copper is partially stripped. Metallic lead gives sulphide, and perhaps some thiosulphate as a white powder. Nickel resists a concentrated solution at 80°. Zinc yields traces of hyposulphite (hydrosulphite), or some complex mixture.—F. H. L.

### *Indigo, Artificial and Natural.* S. Lang. Chem. Zeit. 1897, 21, 961—962.

The most recent form in which synthetically prepared indigotin has been sent into the market is the "Pure Indigo" of the Badische Anilin und Soda Fabrik, a product consisting of 97 per cent. of indigotin with 3 per cent. of moisture and mineral substances. In the author's opinion this product cannot usefully replace vegetable indigo, since, although it chiefly consists of the most important constituent of the natural product, it does not contain in addition indigo-red (indirubin) nor indigogluten—substances which have a marked effect upon the dyeing properties of indigo. An indigo containing little indigo-red in proportion to the indigotin, however rich in the latter substance—*e.g.* Java indigo—does not penetrate the fibre so well as one in which a larger proportion of indirubin exists; and experienced buyers invariably prefer an indigo possessing a reddish tone, especially for the dyeing of thick or heavy yarn or cloth.

That the Badische Anilin und Soda Fabrik are conscious of this defect of their artificial indigo, is evident from the fact that they recommend an addition to the vat of indigogluten, Turkey-red oil, &c.; but these additions cannot replace the indigo-red, &c., present in natural indigo. Indigo-red is the most active of these minor constituents, indigogluten has a similar but less marked influence, whilst indigo-brown and the mineral substances present in indigo have little or no effect in dyeing.

The new product cannot at present compete successfully with indigo as regards price, the present price of indigo being from 12·5 — 16·5 marks per kilo., and that of "Pure Indigo" 17 marks. Besides this, the firm can only guarantee an output in 1898 of an amount equivalent to 1,200 chests of East Indian indigo, the quantity of indigo annually exported from Calcutta alone amounting on the average to 32,500 chests.—R. B. B.

### *Lecithin Content of Some Seeds and Oil-cakes.* E. Schulze. Landw. Vers. Stat. 49, 203—214; Chem. Centr. 1897, 2, [21], 1031.

The author defends the accuracy and certainty of the method for estimating lecithin, which he worked out in conjunction with Frankfurt and Steiger. In order that the lecithin should be satisfactorily extracted, the seeds should be very finely ground, those very rich in fat being first freed from the greater part of it, by coarsely pulverising them with ether. Anhydrous ether should be used, else those seeds which are relatively rich in hygroscopic moisture would cluster together, which would be prejudicial to the estimation. Methyl alcohol should not be used in place of ethyl alcohol, as the former is stated to possess, at boiling heat, a slight solvent action on the anhydrous phosphates of sodium and potassium. The author, in conjunction with M. Merlis, has recently estimated the lecithin content of the seeds of the blue lupin, yellow lupin, vetch, pea, lentil, wheat, barley, maize, buckwheat, flax, hemp, pine, fir, and silver fir. The leguminous seeds, which are rich in nitrogen, contain the largest amount of lecithin, the amount contained in the seeds of the grasses and oleaginous plants being considerably lower; the coniferous seeds also contain relatively little lecithin. The lecithin content of oil-cakes was very variable, but was much smaller than anticipated from the amount contained in the seeds. The author considers that this deficiency is due either to a considerable part of the lecithin being removed with the oil, when the latter is expressed from the seeds, or to a partial decomposition of the lecithin during the preservation of the oil-cakes.—A. S.

### *Tannin, The Optical Properties of.* P. Walden. Ber. 1897, 30, 3151—3156.

Günther (Ber. Pharm. Gesel. 1895, 5, 179 and 297) has shown that tannin—before regarded as an optically inactive substance—is in reality strongly dextro-rotatory. Schiff confirms this observation, and finds that commercial tannins, even when carefully purified, vary widely in rotatory power. These results, which are completely at variance with the accepted non-asymmetrical, inactive, constitution of tannin, are explained by the author's discovery that tannin is a mixed substance, which may be fractionated into portions possessing different gyratory powers.

The following methods for fractionation were used:—

A. Dialysis of a solution in strong alcohol, a porous earthenware partition being used.

B. The same method, using a parchment paper septum.

C. An aqueous solution of tannin was shaken with ether, and the lowest of the three layers formed, collected, and examined.

D. The tannin was dissolved in ethyl acetate, benzene added, and the oily precipitate and supernatant liquid examined separately.

E. The tannin was converted into the lead-salt, the latter decomposed by $SH_2$, and the filtrate from the PbS, concentrated and extracted with ethyl acetate.

F. The tannin was fractionally precipitated from its solution by common salt, and finally purified by ethyl acetate.

A sample of Schuchardt's purest tannin, examined as above, yielded the following results. All the polarimetric readings were taken in 1 per cent. solutions of the specimens:—

Original tannin, $[\alpha]_D = +67°·5$. Method A. Fraction from inner vessel, $[\alpha]_D = +52°$; outer vessel, + 42°. B. Outer vessel, + 21°; inner vessel, + 68°. C. + 75°. D. Precipitate, + 69°·5: solution, + 59°·5. E. + 75°. F. + 39°. A sample of Merck's tannin gave $[\alpha]^D = +15°$.

As to the true nature of "tannin," so-called, the author favours the view that it consists in great part of an inactive, distinctive substance (*i.e.*, true tannin) having a large molecular weight, together with a small percentage of bodies possessing either optical activity, or the power of combining with it to form active closed-chain, anhydride, or lactone compounds.—H. T. P

## New Books.

A Treatise on Chemistry. By H. E. Roscoe, F.R.S., and C. Schorlemmer, F.R.S. Volume II.—The Metals. New Edition, completely revised by Sir H. E. Roscoe, assisted by Drs. H. G. Colman and A. Harden. Macmillan and Co., Ltd., London. 1897. Price 31*s.* 6*d.* The Macmillan Company, New York.

This new edition of the Treatise of Roscoe and Schorlemmer, is actually the third edition. It is a volume of 1,163 pages, containing Table of Contents, Subject-Matter, illustrated with 188 engravings, and a copious Alphabetical Index. The matter is sub-divided and classified under the following heads:—The Metals. Determination of the Atomic Weights of the Metals. Molecular Weights of the Metals. Valency of the Elements. Classification of the Elements. Crystalline Form of the Metals. Alloys and Amalgams. Constitution of Salts, Acids and Bases, &c. Solubility, Fusibility, and Volatility of Salts. Spectrum Analysis. Systematic Description of the Metals and their Derivatives. Group I. The Alkali Metals. Group II. The Alkaline Earth Metals. The Magnesium Group. Group III. The Aluminium Group. Metals of the Rare Earths. Group IV. Titanium, &c. Group V. Vanadium, Bismuth, &c. Group VI. The Chromium Group. Group VII. Manganese. Group VIII. The Iron, Ruthenium and Platinum Groups.

A Handbook for Chemists of Beet-sugar Houses and Seed-culture Farms, containing Selected Methods of Analysis, Sugar House Controls, Reference Tables, &c., &c. By Guilford L. Spencer, D.Sc., of the U.S. Department of Agriculture, Author of "A Handbook for Sugar Manufacturers." First Edition. John Wiley and Sons, New York. Chapman and Hall, Limited, London. 1897. Price $3.

This work takes the form of a pocket-book. It contains 461 pages of subject-matter, inclusive of 127 pages of "Blank forms for practical use in Sugar-House work," each sheet of which is suitably subdivided and headed; also 31 pages of blank forms to be devoted to "Summaries of Yield and Losses." An alphabetical index of subject-matter follows, then some pages of advertisements, and finally 16 blank ruled pages for recording notes and memoranda. The text of the "Handbook" is illustrated with 74 wood engravings. What may be termed the "Apologia" for compiling it is stated in the preface as follows:—"In 1889, the sugar industry of the United States was confined almost exclusively to the cane sections of the South. Sorghum was attracting attention in the North with some prospect of success, the beet industry being represented by two factories in California, and dismantled factories in several other States. The conditions now are quite different. The beet sugar industry bids fair to attain enormous proportions, and Sorghum, for the present, has given up the struggle. Under these changed conditions there appears to be an opening for a book devoted exclusively to the sugar-beet."

The subject-matter is sub-divided as follows:—

I. Sugar-House Control. II. Weights and Measures. III. Estimation of Losses of Sucrose. IV. Sugar Analysis. (*a*) Optical Methods. (*b*) Chemical Methods. V. Sampling and Averaging. VI. Density Determinations, Apparatus, and Methods. VII. Analysis of the Beet. VIII. Analysis of the Juice. IX. Analysis of the Syrup. X. Analysis of the Massecuites and Molasses, of Sugars, of Filter Press-cake, of the Residues from the Mechanical Filters, of the Wash and Waste Waters, and of the Exhausted Cossettes. XI. Definitions of the Coefficients and Terms used in Sugar Analysis. XII. Determination of the Marc. XIII. Viscosity of Sugar-House Products. XIV. Control of the Osmosis Process. XV. Analysis of Saccharates. XVI. Examination of Bone-Black. XVII. Analysis of the Lime-Kiln and Chimney Gases; of Limestone; of Lime; of Sulphur and of Coke. XVIII. Lubricating Oils. XIX. Analysis and Purification of Water. XX. Seed-Selection. XXI. Seed Testing. XXII. Miscellaneous Notes. XXIII. Sugar-House Notes. XXIV. Special Reagents. XXV. From page 226 to 299, a Series of Useful Tables of Reference.

The Gas Engineer's Pocket-Book. Comprising Tables, Notes and Memoranda relating to the Manufacture, Distribution and Use of Coal-Gas and the Construction of Gas Works. By Henry O'Connor, Assoc. Member of the Instit. of Civil Engineers, Vice-President of the Society of Engineers. Crosby Lockwood and Son, 7, Stationers' Hall Court, Ludgate Hill. 1898. Price 10*s*. 6*d*.

This pocket-book for Gas Engineers contains Preface, Table of Contents and 411 pages of subject-matter, followed by the alphabetical index. This book is bound in morocco leather and gilt. The test is sub-divided as follows:—General constructing Memoranda. I. General Mathematical Tables. II. Unloading Materials and Storage (Construction). III. Retort House (Construction). IV. Condensers. V. Boilers, Engines, Pumps and Exhausters. VI. Scrubbers and Washers. VII. Purifiers. VIII. Gasholder Tanks. IX. Gas-Holders. X. Workshop Notes. Manufacturing:—XI. Storing Materials. XII. Retort House. XIII. Condensing Gas. XIV. Exhausters, &c. XV. Washing and Scrubbing. XVI. Purification. XVII. Gasholders. XVIII. Distributing Gas. XIX. Testing. XX. Enriching Processes. XXI. Products.

Glass Blowing and Working. For Amateurs, Experimentalists, and Technicians. Based upon a Course of Lecture-Demonstrations given under the Auspices of the Technical Education Committee of the Middlesex County Council. With Coloured Frontispiece and numerous Illustrations. By Thomas Bolas, F.C.S., F.I.C. Dawbarn and Ward, Ltd., 6, Farringdon Avenue, London, E.C. 1898. Price 2s. Truslove and Comba, 65, Fifth Avenue, New York, U.S.A.

Octavo volume, containing Frontispiece, Preface, 200 pages of subject-matter, Bibliography, and Alphabetical Index. The text is illustrated with 104 woodcuts, and is sub-divided as follows:—I. Glass Work in Miniature as a Decorative Pastime, &c. II. The Blowpipe and Bellows. III. Minor Tools and Appliances. IV. Glass. Rods and Tubing. Gauging, Storing, Preservation, and Cleaning. V. Various Methods of Working and Blowing Glass. VI. Examples of Special Articles for Laboratory and other Uses. VII. Colouring and Modifying Material, &c. VIII. Ageing, Disintegration, and Decay of Glass, &c. IX. Fancy and Decorative Articles made at the Blowpipe. X. Glassmaking at the Blowpipe and on a Laboratory Scale.

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, and MACHINERY.

### Applications.

1496. C. H. Cribb. Improvements in apparatus for the condensation of vapours for distilling and like purposes. Jan. 19.

1591. J. B. M. M. Guthrie and J. H. Hughes. Improvements in the apparatus for the manufacture of various gases. Jan. 20.

1708. F. D. Cummer. Improvements in apparatus for drying materials of various kinds. Jan. 21.

1811. W. Rushworth. Improved means for refrigerating. Jan. 24.

1893. P. G. de Schodt. Improvements in apparatus for compressing gas. Jan. 24.

2104. H. J. Warrington. An improved lining for puddling and other furnaces. Jan. 26.

2129. E. Jones and Kynoch, Ltd. Improvements in and relating to evaporative surface condensers. Jan. 26.

2193. L. Rousseau. Improvements in or relating to crucibles. Jan. 27.

2218. H. H. Lake.—From A. Mazzoni, Italy. Improvements in kilns. Jan. 27.

2274. W. Nelson. An improved apparatus to be used in refrigerating or freezing processes. Filed Jan. 28. Date applied for Nov. 26, 1897, being date of application in New Zealand.

2285. P. F. C. Willcox, H. W. Willcox, and R. J. N. Willcox. Improvements in and relating to apparatus for heating and evaporating liquids by means of steam. Jan. 28.

2452. T. Thorp. Improvements in apparatus for mixing gases. Jan. 31.

2464. V. E. J. Durafort. Improvements in apparatus for containing compressed carbonic acid, and charging water or other liquid therewith. Jan. 31.

2716. W. J. McClurg and The McClurg Gas Construction Company. Improvements in gas generators. Feb. 2.

3039. J. Q. Rayner. Improvements in reverberatory furnaces. Complete Specification. Feb. 7.

3106. F. R. Lipscombe. Improvements in filters. Feb. 7.

3108. I. P. B. Knudsen. Improvements in centrifugal separating apparatus. Feb. 7.

3116. W. Killeen. Improvements in refrigerating apparatus. Feb. 8.

3289. J. W. T. Cadett. Improved means for producing high vacua. Feb. 9.

### Complete Specifications Accepted.*

### 1897.

1843. A. J. Boult.—From H. H. Dikema. Method of mixing or separating solid, liquid, or gaseous bodies in solution with or from other bodies, and apparatus therefor. Jan. 26.

2931. R. G. Brooke. Apparatus for purifying fluids. Dec. 22.

3999. F. Eveleigh and P. H. Mellor. Fuel economiser or apparatus for heating the feed water of boilers by means of the waste products of combustion. Feb. 2.

4612. D. A. Blair. "Coffey's" distilling apparatus and the like. Feb. 2.

8247. J. Laidlaw and J. W. Macfarlane. Centrifugal machines. Feb. 2.

8775. N. S. Jenkins. Melting apparatus. Feb. 16.

22,816. J. V. Skoglund. Nitric acid condensers. Feb. 9.

24,084. G. Barker.—From L. Rissmuller, Germany. Centrifugal separators. Dec. 22.

24,201. J. Klein. Apparatus for the condensation of steam and vapours. Feb. 2.

29,576. R. H. J. G. Huet. Separator for use in connection with granulated substances, liquids, and the like. Jan. 26.

29,786. J. Klein. Apparatus for cooling water and other liquids. Feb. 9.

## II.—FUEL, GAS, AND LIGHT.

### Applications.

1234. J. St. C. Legge and A. S. Cooper. An improved gas generator used to generate acetylene gas and the like. Jan. 17.

1251. S. Chandler, jun., and J. Chandler. Improvements in apparatus used in the manufacture of gas from oil or other fluids. Jan. 17.

1310. G. F. Dinsmore. Process of transmitting heat for industrial purposes. Filed Jan. 17. Date applied for June 18, 1897, being date of application in United States. Complete Specification.

1312. W. R. Green. Improvements in the manufacture of illuminating gas, and in materials for use therein. Filed Jan. 17. Date applied for Oct. 18, 1897, being date of application in United States.

1343. W. H. Graham.—From H. Maxim, United States. Apparatus for producing acetylene. Jan. 18.

1526. A. Bezant. Improvements in acetylene lamps and generators combined. Jan. 19.

1584. H. de Thiersant and W. A. Coulson. Improvements in apparatus for the production of acetylene gas. Jan. 20.

1625. W. Owens. Improvements in or connected with the manufacture and storage of acetylene gas. Jan. 20.

1656. W. Rowbotham. Improvements in apparatus for generating acetylene gas. Jan. 21.

1665. R. Quatannens - Moens and E. Carrcer - Dilger. Machine for producing acetylene gas for private and public lighting. Complete Specification. Jan. 21.

1709. T. C. J. Thomas. Improvements in the manufacture of incandescent bodies for burners. Jan. 21.

1767. T. Macalpine, B. J. Atterbury, and The Alcohol Syndicate, Ltd. *See* Class VII.

1778. C. Davies, H. Arrowsmith, and S. W. Dod. A new or improved manufacture of fuel applicable also as firelighters. Jan. 22.

1920. R. J. Moss. Improvements in apparatus for generating acetylene gas. Jan. 24.

2015. S. H. Crocker. An improved incandescent mantle for gas lighting. Complete Specification. Jan. 25.

2131. P. G. de Schodt. Improvements in incandescent gas lighting apparatus. Filed Jan. 26. Date applied for Sept. 17, 1897, being date of application in Belgium.

2174. J. S. Goodwin. Improvements in acetylene generating apparatus. Jan. 27.

2175. H. P. D. Ohlhaver. Improvements in and relating to mantles and the like for incandescent gas burners. Jan. 27.

2309. G. Jacobi. Improved acetylene gas apparatus. Complete Specification. Jan. 28.

2444. A. E. N. Yeadon and S. N. Yeadon. An improved apparatus for the purification of gas or other analogous purposes. Jan. 31.

2452. T. Thorp. *See* Class I.

2453. T. Thorp. Improvements connected with gas conduits. Jan. 31.

2454. T. Thorp. Improvements in apparatus for the production of acetylene from calcium carbide and water. Jan. 31.

2592. E. Favaretto.—From V. Favaretto, Italy. Preparation to produce the acetylene gas which passes to the shaft through a special grill. Feb. 1.

2599. R. Boecking. Cooling device for the discharge mains or pipes of ovens or gas-producing apparatus through which the gases are led off for the condensation of tar, ammonia, benzol, &c. therefrom, and for use as illuminant fuel and the like. Feb. 1.

2601. A. J. Lucy. Improvements in apparatus for the production of acetylene gas, and lamps in connection therewith. Feb. 1.

2602. H. O. Marcks. Apparatus for generating acetylene. Complete Specification. Feb. 1.

2613. G. Himmel. Improvements in or connected with Bunsen burners for incandescent lighting. Complete Specification. Feb. 1.

2614. H. L. Robinson and H. S. Bishop. A new method of strengthening incandescent gas light mantles. Feb. 2.

2649. A. Handley and W. P. O'Neill. An improved method of preparing peat, charcoal, and using same in the manufacture of calcium carbide. Feb. 2.

2741. R. B. Adams. Mechanical means for (and mode of) burning off incandescent gas mantles. Feb. 3.

2780. H. S. Ryland and A. N. Townley. An improved automatic acetylene gas generator. Feb. 3.

2797. G. Winstanley. Improvements in and in connection with the regenerator settings of retorts for the manufacture of coal-gas and for other purposes. Feb. 3.

2802. W. P. Thompson.—From E. Charpentier, France. Improved lamp for generating and burning acetylene gas. Feb. 3.

2863. J. S. Ferguson. Acetylene gas generator. Filed Feb. 4. Date applied for July 19, 1897, being date of application in United States. Complete Specification.

2967. B. J. Atterbury, T. MacAlpine, and The Alcohol Syndicate, Ltd. New or improved production of acetylene compounds and of ethylene. Feb. 5.

3091. F. von Hof and H. Dobert. Improvements in apparatus for burning and shaping incandescent bodies or mantles. Feb. 7.

3172. G. B. Ellis.—From L. E. Lacroix, France. Improvements in the production of ozone, and apparatus therefor. Feb. 8.

3185. C. Mathieu. Process and apparatus for feeding gas engines with carburetted air for producing an illuminating and heating gas by recarburetting the burned products. Feb. 8.

3190. A. J. Morrison. Improvements in apparatus for generating and burning acetylene gas. Feb. 8.

3244. W. Rowbotham. Improvements in apparatus for generating acetylene gas. Feb. 9.

3292. W. W. Sarginson. An improved acetylene gas generator. Feb. 9.

3294. R. P. Ross. Improvements in acetylene gas generators. Feb. 9.

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* *See* Note (*) on previous page.

3493. W. MacKeap. A burner for burning off incandescence mantles. Feb. 11.

3531. T. Mitchell. Improvements in apparatus used in the production and storage of acetylene gas. Feb. 12.

### Complete Specifications Accepted.

1897.

2260. F. S. Thorn and C. Hoddle. Acetylene jet for optical lantern use. Jan. 26.

2261. F. S. Thorn and C. Hoddle. Receptacle for safely storing calcium carbide.

2554. E. G. Villejean and H. J. J. Frossard. Automatic generation of acetylene gas. Jan. 26.

2738. P. B. Watson. Producing atmospheric gas from liquid hydrocarbons. Feb. 9.

2894. H. Maxim. Apparatus for production of calcium carbide, applicable also to other purposes. Feb. 2.

3129. A. Freitag. Burning of pulverulent fuel, and apparatus therefor. Feb. 2.

3539. A. Rose. Burners for incandescent gas lights. Jan. 26.

4424. E. Gossart and H. Chevalier. Means for producing and burning acetylene. Feb. 9.

4428. K. Stattler and A. Strejz. Process for producing gas for burning and lighting purposes. Feb. 9.

6159. J. Moeller. Oil lamps for incandescence lighting. Feb. 2.

7013. L. L. Merrifield. Apparatus for the manufacture of carburetted water-gas. Jan. 26.

7043. T. Lyons, J. Lyons, and J. Pearson. Acetylene gas generators. Feb. 16.

7177. E. Bohm. Production of ozone. Feb. 2.

7330. R. Schoder. Stiffening incandescence mantles. Feb. 2.

8007. R. Dixon. Manufacture of coke. Feb. 9.

8270. J. G. A. Kitchen. Acetylene generators. Feb. 16.

19,889. F. J. Lothammer. Apparatus for carburising air. Feb. 9.

22,850. W. H. Wheatley.—From C. W. Beck, United States. Lamps for generating and burning acetylene gas. Feb. 9.

25,866. A. H. Pöppel. Apparatus for the manufacture of gas. Jan. 26.

27,240. A. Forcher. Acetylene gas generator for illuminating railway carriages. Feb. 9.

27,379. B. Talbot and W. B. Hughes. Construction of gas producers. Feb. 16.

27,536. O. Falbe and E. Borchardt. Burners for acetylene gas. Feb. 2.

27,886. J. Skar. Process for manufacture of fuel blocks. Feb. 9.

29,054. J. O. O'Brien.—From E. M. Boule, France. Apparatus for generating acetylene gas. Feb. 9.

29,234. W. H. Russell and G. E. Russell. Manufacture of gas. Feb. 2.

29,605. W. P. Thompson.—From The Compressed Coal Company, United States. Improvements in fuel. Feb. 2.

30,690. J. St. C. Legge and A. S. Cooper. Generator for acetylene gas. Feb. 9.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Application.

1411. V. T. Ragosin. A method of distilling, fractioning, and cracking mineral oils, and obtaining various products therefrom, and apparatus for that purpose. Complete Specification. Jan. 18.

### Complete Specification Accepted.

1897.

7233. G. W. Johnson.—From Kalle and Co. Manufacture of guaiacol. Feb. 2.

## IV.—COLOURING MATTERS and DYES.

### Applications.

1431. A. G. Green and A. Meyenberg. Method for the preparation of azo- and azoxo-aldehydes. Jan. 18.

2076. A. Ashworth and J. Bürger. Improvements in the production of colouring matters. Jan. 26.

2326. A. G. Green and R. Lévy. Improvements in the manufacture and production of colouring matter. Jan. 28.

2480. G. W. Johnson.—From Chemische Fabrik Griesheim, Germany. Improvements in the manufacture or production simultaneously of pure ortho-nitro-chlorobenzene and pure para-nitro chloro-benzene. Complete Specification. Jan. 31.

2501. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of new alizarine dyestuffs. Jan. 31.

2503. H. Weil. Manufacture of green colouring matters for dyeing wool. Jan. 31.

2892. J. Gardner. Improved liquid compound for dyeing purposes. Feb. 1.

3155. C. E. Gassmann. A new method of manufacturing stable diazoic salts. Feb. 8.

3393. A. G. Green and A. Meyenberg. Manufacture and production of intermediate products and of colouring matters therefrom. Feb. 10.

3409. J. Hauff. Method of manufacture of ortho-toluolsulpho-chloride. Feb. 10.

### Complete Specifications Accepted.

1897.

4369. W. H. Claus, A. Ree, and L. Marchlewski. New polyazo dyes. Jan. 26.

5535. H. R. Vidal. Carboxylated products derived from amido or phenolic compounds and amides and phenolics of triphenylmethane. Jan. 26.

5689. H. R. Vidal. Manufacture of colouring matters. Feb. 16.

5690. H. R. Vidal. Colouring matters. Feb. 16.

6694. H. R. Vidal. Colouring matters. Feb. 9.

7213. H. H. Lake.—From The Farbwerk Mühlheim vormals A. Leonhardt and Co. Manufacture of sulpho acids of the naphthalene series and of colouring matters derived therefrom. Jan. 26.

7329. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. New safranine derivatives. Feb. 9.

7337. S. Pitt.—From L. Cassella and Co. Production of azosafranine derivatives. Feb. 9.

7341. J. Y. Johnson.—From C. F. Boehringer and Soehne. Preparation of alkylated uric acids. Feb. 9.

8175. G. B. Ellis.—From La Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier. Colouring matters. Feb. 9.

8188. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of colouring matters. Feb. 9.

8383. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. New safranine derivatives. Feb. 9.

21,398. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. *See* Class XIII.

21,991. J. Möller. Ink. Jan. 12.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

1839. G. W. Oldham. Improvements in the method of and means for mercerising hanks of yarn. Jan. 24.

2313. H. H. Lake.—From C. W. Carter and J. S. Nickerson, United States. Improvements in the manufacture of waterproofed vulcanised fibre. Jan. 28.

2866. C. E. Wilkinson and E. Crewdson. An improved process of and apparatus for cleansing or purifying cotton, wool, silk, hemp, jute, and like substances or materials. Feb. 4.

3100. H. R. Romney and J. Thame. Improvements in treating waste cocoa-nut dust, fibre, or refuse. Feb. 7.

3553. F. Holtschmidt. A process for lining stuffs with paper paste. Complete Specification. Feb. 12.

3581. O. Imray.—From United States Flax Fibre Company of United States. Process of and apparatus for cleaning flat fibres. Complete Specification. Feb. 12

### Complete Specifications Accepted.

1896.

23,427. C. J. Dear. Machine relating to the decortication of ramie and other fibrous plants. Jan. 26.

1897.

2736. J. Shearer. Improving the colour of raw jute fibre. Jan. 26.

2960. C. O'Brien and J. Shearer. Improving the colour of raw jute fibre. Jan. 26.

18,728. A. L. C. Nodon and L. A. Bretonneau. Process or decorticating and degumming ramie and other like fibrous materials. Feb. 9.

27,651. K. Klietsch. Manufacture of linoleum, kamptulicon, and similar materials. Feb. 9.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

1634. F. Erban and L. Pick. Improved method of bleaching, dyeing, washing, or treating fabrics and other materials with liquid, and apparatus therefor. Complete Specification. Jan. 20.

2935. J. Longthorpe, W. Woodcock, and W. Dargue. Improvements in machinery for dyeing, sizing, and washing hanks of yarn and slubbing. Feb. 5.

3300. W. Mather and R. H. Haworth. A process and apparatus for continuous bleaching of fabrics or yarns in open condition. Feb. 9.

### Complete Specifications Accepted.

1897.

4853. A. Schmidt. Dyeing apparatus for carded wool. Jan. 26.

6498. J. Waugh.—From The Neue Augsburger Kattunfabrik. Method of washing and boiling off, and in the preparation of fabrics to be dyed or printed. Jan. 26.

13,591. J. Holden and S. Woods. Embossed or raised paper for covering walls and for other purposes. Jan. 26.

24,533. C. L. Horack. Apparatus for colouring yarns. Jan. 26.

25,117. E. Lehmann. Process for marbling, mottling, or colouring paper or cardboard on one side in the paper machine. Feb. 2.

30,038. O. P. Amend. Mordanting and dyeing animal fibre. Feb. 2.

30,039. O. P. Amend. Mordanting animal fibre. Feb. 2.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

1524. J. Sharp. Improvements in separating, purifying, and analysing mixed waste acids, and apparatus therefor. Jan. 19.

1767. T. Macalpine, B. J. Atterbury, and The Alcohol Syndicate, Ltd. An improved method for the production of copper acetylene. Jan. 22.

1819. J. W. Kelly and H. Roantree. Improvements in apparatus for the production of carbide of calcium. Jan. 22.

1983. G. Bower.—From J. E. Hewes, United States. Improvements in the process for the manufacture of carbide of calcium, and apparatus therefor. Jan. 25.

1984. G. Bower.—From J. E. Hewes, United States. Improvements in the process for the manufacture of carbide of calcium, and apparatus therefor. Jan. 25.

1985. G. Bower.—From J. E. Hewes, United States. Improvements in the process for the manufacture of carbide of calcium, and apparatus therefor. Jan. 25.

2815. J. Hawliczek and The United Alkali Company Ltd. Improvements in the manufacture of acetone and in apparatus therefor. Feb. 3.

2816. F. Hurter and The United Alkali Company, Ltd. Improvements in the manufacture of acetone and in apparatus therefor. Feb. 3.

2823. C. T. Owen. Improvements in the manufacture of ammonia and ammonia compounds. Feb. 3.

2997. E. Gearing. Improvements in the preparation of calcic and other gas-producing carbides. Feb. 5

3064. P. Marino. Improved manufacture of sulphuric acid. Complete Specification. Feb. 7.

### Complete Specifications Accepted.

1897.

2651. C. H. Mehner. Method of producing ammonia. Jan. 26.

## VIII.—GLASS, POTTERY, and ENAMELS.

### Applications.

1498. T. Pfister. Improvements in transparent effects of imitation stained glass by crystallisation. Jan. 19.

1875. E. Edwards.—From L. C. Janitz, Germany. Improvements in down-draught kilns for burning bricks, clay, pottery, and the like. Jan. 21.

1948. T. H. Stubbs. Improved methods of manufacturing tiles and other ceramic ware, with designs in colours therein. Jan. 25.

2142. N. Browne.—From The Glashüttenwerke Alderhütten Actien Gesellschaft, Germany. Improved hollow glass blocks for the construction of walls pervious to light. Complete Specification. Jan. 27.

2190. J. T. Szék. Improvements in plastic compositions for, and in the construction or manufacture of tiles, plates, and blocks for making, decorating, or covering walls, floors, ceilings, and the like. Jan. 31.

2539. L. H. Burgess and F. Sheppard. The process to unite any metal or metallic alloy to glass, china, earthenware, parian, stone, or like body. Feb. 1.

2727. E Moore. Improvements in or connected with spring punties employed in the manufacture of articles of glass. Feb. 2.

2758. J. H. Barratt. An improvement in the method of sieving or sifting dry or liquid materials for the use of potters and others. Feb. 3.

### Complete Specifications Accepted.

1897.

6347. A. H. Moore and J. W. Moore. Equable firing kilns. Feb. 9.

24,505. A. C. Tulloch. Method and means for the drying of china clay. Feb. 2.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

1319. P. Kleber. Process for the production of building stones and like articles. Complete Specification. Jan. 17.

1378. P. M. Justice.—From The Lithosite Manufacturing Company, United States. Improvements in the process of manufacturing cements. Complete Specification. Jan. 18.

1612. J. Clarke, J. J. Hewitt, and T. G. Stevens. Improvements in cement kilns. Jan. 20.

1758. H. K. Bromhead. Improvements in fireproof roofs and floors. Jan. 22.

1769. P. Kleber. A process for the manufacture of fireproof building stones, and for the production of cement from the same. Complete Specification. Jan. 22.

1875. E. Edwards.—From L. C. Janitz, Germany. *See* Class VIII.

2003. L. A. Garchey. Improvements in the manufacture of ceramic stone. Jan. 25.

2018. J. G. Lorrain.—From M. Marti, Hong Kong. A new or improved composition for building and decorative purposes and the like. Jan. 25.

2041. J. Boss. Improvements relating to paving and pavements. Jan. 26.

2142. N. Browne.—From Glashüttenwerke Alderhütten Actien Gesellschaft, Germany. *See* Class VIII.

2255. W. Wright. Improved method of seasoning timber. Jan. 28.

2328. T. H. Redwood and A. J. de Hailes. Improvements in fireproofing wood and other materials. Jan 28.

2822. H. H. Leigh.—From F. Cohn and H. G. B. Brunckhorst, Germany. Improvements in and relating to fireproof ceilings. Feb. 3.

2896. E. H. Hurry and H. J. Seaman. Improvements in process and apparatus for the manufacture of Portland cement, parts of which are applicable to other purposes. Complete Specification. Feb. 4.

2905. J. Hollick and G. Taylor. Improvements in kilns used in the manufacture of cement and like substances. Feb. 4.

3191. F. Bernstein. A process to produce a self-acting polish for natural and artificial stones, marbles, granite, &c., &c., and also to manufacture the same stones as mentioned by artificial means. Feb. 8.

3203. H. J. Harding. An improved process for preparing and treating lime, and apparatus therefor. Feb. 8.

3376. W. Wright. An improvement in the manufacture of building blocks or bricks and the like. Feb. 10

### Complete Specifications Accepted.

1896.

29,212. Petrifite, Ltd.—From J. Steiger. Manufacture of cements. Jan. 26.

1897.

2195. A. Suart and G. W. Robertson. Apparatus employed in the manufacture of paving bricks or tiles. Feb. 2.

6413. D. Burness and J. Burness. Concrete paving and the like. Feb. 2.

21,921. C. W. Luther. Way of constructing light fireproof building material possessed of the quality not to be eaten by insects, and therefore specially adapted for hot climates. Dec. 29.

30,344. F. H. Willis. Construction of fireproof floors. Feb. 16.

## X.—METALLURGY, MINING, Etc.

### Applications.

1427. L. Bonneville. Improvements in the manufacture of alloys or metal compounds. Jan. 18.

1432. O. S. Garretson. Improvements relating to the smelting and converting of metal ores, and to apparatus therefor. Complete Specification. Jan. 18.

1534. S. O. Cowper-Coles. Improvements in the treatment of zinc-bearing ores for the obtainment of zinc and other metals therefrom. Jan. 19.

2049. E. J. Ball. Improvements in the metallurgical treatment of auriferous and argentiferous ores and substances. Jan. 26.

2118. A. H. Pehrson and E. V. Pehrson. Improvements in moulded materials for use in the manufacture of steel castings, and in the method of preparing the same. Complete Specification. Jan. 26.

2293. J. T. Wainwright. Apparatus for exhausting molten metal. Complete Specification. Jan. 28.

2567. G. Hammond and T. Flint, jun. Solder and flux for aluminium. Complete Specification. Feb. 1.

2638. E. J. Ball. Improvements in the metallurgical treatment of metallic ores and other metalliferous substances. Feb. 1.

2799. W. A. P. Werner. Improvements in and relating to the metallisation of surfaces. Feb. 3.

2817. J. M. Gledhill. Improvements relating to apparatus employed for melting steel or other metal. Feb. 3.

2878. J. A. Mayes. Process of and apparatus for reducing and separating metals from ores or oxides of lead and zinc. Feb. 4.

3138. J. Swinburne. Improvements in the separation of metals. Feb. 8.

3296. C. V. Burton. A process for tinning and soldering aluminium. Complete Specification. Feb. 9.

3397. H. C. Bull, C. P. Shrewsbury, and F. L. Marshall. A new or improved process for extracting gold and other metals from sea-water. Feb. 10.

3406. D. Tschernoff. Improvements in the manufacture of iron and steel, and in furnaces for that purpose. Feb. 10.

3497. L. Liebmann. The production or obtainment of metallic beryllium from minerals containing it. Feb. 11.

### Complete Specifications Accepted.

1896.

23,275. E. B. Parnell, A. Smith, and C. E. Monkhouse. Treatment of ores, and apparatus therefor. Jan. 26.

1897.

2350. H. Wolff. Preparation of zinc amalgam containing aluminium or magnesium. Feb. 2.

5796. T. Korovine, N. Klatschko, and J. Berthenson. Treating iron and steel. Feb. 16.

7021. La Société des Cuivres de France. Obtaining pure copper in a single operation. Jan. 26.

7198. J. Cope. Nickel or nickel-alloy castings Feb. 16.

7532. E. A. Ashcroft. Combined electrolytic and leaching treatment of zinc bearing ores and zinc-bearing products. Feb. 9.

8626. F. Moss and S. J. Davis. Process for staining and colouring metals, and for other suitable purposes. Feb. 9.

27,717. W. Buckley. Improvements in ingots or similar moulds, and to the manufacture of ingots. Feb. 2.

28,568. C. M. White.—From King, United States. Process of annealing iron castings. Feb. 9.

28,896. J. Rudolphs and J. Landin. Method or process of extracting metals from pulverised or finely divided ores or compounds. Jan. 26.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### APPLICATIONS.

1628. W. A. Crowdus. Improvements in and relating to secondary batteries. Jan. 20.

1697. M. Haas. Apparatus for the electrolytic treatment of bleaching liquids. Complete Specification. Jan. 21.

1702. M. Reynard. Improvements in electric batteries. Jan. 21.

1735. A. G. Adamson, T. W. Allan, and Allan and Adamson, Ltd. Improvements in electrical accumulators or storage batteries. Jan. 22.

2024. C. Koenig. Improvements in and relating to primary batteries. Complete Specification. Jan. 25.

2030. E. Giglio. Improvements in primary batteries. Jan. 25.

2389. J. V. Sherrin. Improvements in secondary batteries. Jan. 29.

2415. H. Maxim and W. H. Graham. Improvements in methods or processes for electro-thermally treating materials, and particularly with reference to the manufacture of calcium carbide, and apparatus therefor. Jan. 29.

2681. F. Fisher, B. J. Round, B. J. Round, and A. Round. Improvements in apparatus for the electro-deposition of metals. Feb. 2.

2871. W. H. Graham. Improvements in electric furnaces. Feb. 1.

2967. H. L. Haas. Improvements in electro-depositing anodes. Complete Specification. Feb. 5.

2998. J. Greenwood. Improvements in and relating to the decomposition of alkaline salts by electrolysis, and in apparatus therefor. Feb. 5.

3136. J. Swinburne. Electrolytic treatment of sulphides. Feb. 8.

3137. J. Swinburne. Improvements in the treatment of complex sulphides. Feb. 8.

3192. E. de Pass.—From La Compagnie Electro-Sucrière, France. Improved method for the electrolytic treatment of sugar juice. Feb. 8.

3193. E. de Pass.—From La Compagnie Electro-Sucrière, France. Process for the electrolytic treatment of sugar juice. Feb. 8.

3198. Cecil Lord Saunders. Improvements in the manufacture of carbons for electrical purposes. Feb. 8.

3285. R. Kennedy. Improvements in and connected with plates for electrical storage batteries. Feb. 9.

3367. D. d'Arbel. Improvements in the production of acetylene light by electrolysis. Complete Specification. Feb. 10.

3368. D. d'Arbel. Improvements in secondary batteries. Complete Specification. Feb. 10.

3431. H. E. Beach. Improvements in anodes used in the electro-deposition of metals. Feb. 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1896.

25,036. J. L. Dobell. Electric batteries. Feb. 16.

1897.

1203. H. W. Hancock and A. H. Dykes. Secondary battery plate. Dec. 22.

2418. E. W. Bonson. Form of electrode for galvanic or secondary batteries. Jan. 26.

2790. L. Bresson and P. Pacotte. Electric furnaces for use in the manufacture of calcium carbide, applicable also for obtaining metals from their salts. Jan. 26.

3526. J. E. Hartley.—From F. W. Zingsen. Apparatus for the electro-deposition of metals. Feb. 2.

4142. J. L. Dobell. Electric batteries. Feb. 16.

5383. S. F. Walker. Primary galvanic batteries. Feb. 16.

12,028. R. W. Atkinson and S. F. Walker. Sulphuring carbon plates for galvanic batteries. Jan. 26.

25,400. E. Motz and H. F. Welch. Electrolytic process and apparatus for the separation of metals from their ores and solutions. Dec. 22.

26,141. C. A. Bennert. Preparation of accumulator plates. Dec. 22.

27,129. A. L. C. Nodon, L. A. Bretonneau, and P. d'Alton Shée. Electrolytic method of and apparatus for saponification. Feb. 2.

29,273. H. H. Lake.—From C. Francke, Germany. Plates or electrodes for electric accumulators. Jan. 26.

29,507. M. Engl. Method of and apparatus for producing perforated accumulator plates. Feb. 2.

## XII.—FATS, OILS, AND SOAP.

### APPLICATIONS.

1508. C. Pitcairn. Improvements in the manufacture of soap. Jan. 19.

1761. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in the oxidising of oils and in apparatus therefor. Jan. 22.

2801. C. Davies and H. Arrowsmith. Improvements in the manufacture of soap. Feb. 3.

2836. H. Young. Improved saponaceous and bleaching compounds. Feb. 4.

3399. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in or relating to the bleaching, purifying, refining, and hardening of animal and vegetable fats and waxes. Feb. 10.

3568. J. Frankenberg. A device for trapping or separating fatty or like matter from waste liquids. Feb. 12.

3585. G. Pommerhanz, C. Ristic, and V. Noback. Improvements in the production of fats suitable for lubricating purposes. Feb. 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

2662. W. E. Pearson. Production of prepared tar, mineral, and other oils for pharmaceutical, veterinary, technical, and other purposes. Feb. 9.

7228. P. Villain. Manufacture of scented antiseptic, medicinal, or multi-coloured or variegated soap, and machinery or apparatus therefor. Feb. 9.

9799. R. W. English. Apparatus for bleaching linseed oil. Feb. 2.

14,241. L. Grote. Bleaching soap. Jan. 25.

25,505. H. Harris and H. Harris, Sons, and Co., Ltd. Manufacture of soap tablets. Jan. 12.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### APPLICATIONS.

1384. F. S. D. Scott and T. Hawkins. Improvements in the manufacture of pigments for paints, dyes, stains, and the like. Jan. 18.

2114 M. J. A. F. Maurice. Improved process of extracting gutta-percha from trees and plants furnishing the same. Filed Jan. 26. Date applied for July 29, 1897, being date of application in France.

2483. E. F. Shorey, W. O. Collingwood, and E. Figg. Glazing enamel. Jan. 31.

2553. S. Warner and C. F. W. Perry. An improved anti-fouling composition for ships' bottoms or the like. Feb. 1.

2832. C. O. Weber. Improvements in the manufacture of lake pigments. Feb. 4.

2887. C. Ives. Plastic compositions. Feb. 4.

3031. P. L. Whitehouse. An improved pigment or paint material. Feb. 7.

3160. A. J. Boult.—From B. G. Work and J. F. Palmer, United States. Improvements in or relating to the treatment of rubber. Complete Specification. Feb. 8.

3394. P. C. D. Castle. Process for making vulcanite from a vegetable substance, with and without the addition of rubber. Feb. 10.

3582. G. Bischof. Apparatus for churning white lead and oil into pasty condition. Feb. 12.

### Complete Specifications Accepted.

1897.

6324. T. C. Sanderson. Manufacture of white lead. Feb. 2.

20,531. T. Thompson. Waterproofing compound or composition. Jan. 26.

21,398. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Manufacture of alizarine lumps. Feb. 16.

22,867. H. L. Bates. An improved paint. Feb. 16.

29,635. S. Hansel and E. Hornung. A waterproof coating and impregnating substance, and a process for the manufacture of the same. Jan 26.

30,242. A. H. Eyles, H. S. Rapelye, and A. Applegate. Manufacture of carbonate or sulphate of lead. Feb. 16.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

2330. J. Millwater and A. McMaster. Improved means and apparatus for staking and grounding leather. Jan. 28.

2400. R. Brauer. An improved manufacture of glue. Complete Specification. Jan. 29.

2872. E. E. M. Payne, J. Pullman, and E. E. Pullman. Improvements in the process of leather dressing. Feb. 4.

2873. E. E. M. Payne, J. Pullman, and E. E. Pullman. Improvements in the process of liming furs, hair, skin, skins, hides, pelts, or parts thereof. Feb. 4.

3177. H. H. Lake.—From C. Francis, Fiji Islands. Improvements in and relating to the production of tannin. Feb. 8.

### Complete Specifications Accepted.

1897.

1442. W. J. J. Hawkins. Printers' roller composition or the like. Jan. 26.

6769. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. *See* Class XXI.

## XV.—AGRICULTURE AND MANURES.

### Applications.

1435. G. O. J. Schüler. Improvements in the production of double superphosphates. Jan. 18.

1541. O. Imray.—From Bauer and Co., Germany. Albumen preparations soluble in water made from glycerinophosphates and albuminoids. Jan. 19.

### Complete Specifications Accepted.

1897.

5574. H. E. Newton.—From A. Caron. Increasing the growth of cereals and their yield by means of bacteria, and a method for developing and conserving the latter on a large scale. Jan. 26.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

1285. C. D. Abel.—From E. L. Erben, Germany. Improved process for the refining of raw sugar. Complete Specification. Jan. 17.

2287. M. Riegel and J. A. Rose. Process for the production of casein compounds and casein derivatives soluble in water. Complete Specification. Jan. 28.

2486. E. B. Le Beuf. Improved process and apparatus for the rapid refining of sugar. Jan. 31.

2890. J. F. Criddle. Improvements in vegetable syrups. Feb. 4.

3081. G. B. Ellis.—From Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. Improvements in the production of saccharin. Feb. 7.

3192. E. de Pass.—From La Compagnie Electro Sucrière, France. *See* Class XI.

3193. E. de Pass.—From La Compagnie Electro-Sucrière, France. *See* Class XI.

5242. E. D. Smythe. Apparatus for reclaiming crystallised syrups. Jan. 26.

### Complete Specifications Accepted.

1897.

7861. M. Sachs. Separating impure sugar at one operation into crystallised sugar and molasses. Feb. 9.

18,429. L. Sternberg. Extraction of sugar from molasses by the use of barium compounds. Feb. 2.

26,985. J. Peeters. Process for the production of new vegetable extracts and new vegetable peptones of commercial utility by the transformation of all kinds of yeasts and these new products of commercial utility themselves. Jan. 26.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

2031. H. Georgin. An improved process for the manufacture of beer by the sterilisation of the worts in the vat during the boiling. Jan. 25.

2782. M. J. F. Saladin. Apparatus for germinating barley and other cereals for use in breweries, distilleries, and the like. Feb. 3.

### Complete Specifications Accepted.

1897.

4089. L. Maiche. Process for the purification of spirit. Feb. 16.

4612. D. A. Blair. *See* Class I.

6343. E. G. Major-Lucas. Apparatus for separating beer from yeast. Jan. 26.

8080. B. J. B. Mills.—From A. Dalmote. Diastatic preparation to be employed in the manufacture of bread and other foods and beverages, and to be used as an aid to digestion. Feb 16.

19,858. A. Colette, jun. Extraction of alcohol by saccharification and fermentation by mucedineæ, and apparatus therefor. Feb. 16.

26,520. G. Delory. Manufacture of beer. Feb. 2.

26,985. J. Peeters. *See* Class XVI.

28,579. C. Schmitz. Process and apparatus for producing beer wort from malt flour. Feb. 9.

30,208. V. Lapp. Process for the manufacture of non-alcoholic beer. Feb. 9.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

### *B.—Sanitation.*

2679. J. P. van Gelder and P. van Gelder. Improvements in apparatus for collecting and sorting waste products, and generally for purifying air or other gases. Feb. 2.

2749. F. P. Candy. Improvements in and in connection with the purification of sewage and impure waters. Feb. 3.

3333. F. P. Candy. Improvements in the purification of sewage and polluted waters. Feb. 10.

3450. F. M. Spence and D. D. Spence. Improvements in the treatment of sewage. Feb. 11.

### *C.—Disinfectants.*

1723. E. Fournier. A new or improved disinfectant. Complete Specification. Jan. 21.

2407. R. Ehrhardt. Improvements in the manufacture of antiseptic soaps containing mercury albuminate. Complete Specification. Jan. 29.

### Complete Specifications Accepted.

### *A.—Foods.*

1897.

4730. B. L. Ryder. Apparatus for the preservative desiccation of vegetable and animal substances. Feb. 2.

8080. R. J. R. Mills.— From A. Dalmote. *See* Class XVII.

### *B.—Sanitation.*

1897.

5803. W. M. Ducat. Purification of water, sewage, and other foul liquids. Feb. 9.

6316. A. Apps. Sterilising, purifying, and deodorising receptacles for beverages and other materials. Jan. 26.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications

1703. C. H. Homan. Improvements in and in the manufacture of artificial ivory. Jan. 21.

1749. J. Hargreaves and M. Duxbury. Improvements in the method of damping paper during the process of manufacture, and in apparatus for effecting the same. Jan. 22.

2790. A. L. C. Nodon, L. A. Bretonneau, and P. d'Alton Shée. A new or improved method of manufacturing papier-maché and the like. Feb. 3.

### Complete Specification Accepted.

1897.

1390. D. Pearson and D. N. Bertram. Paper-pulp refining engines. Jan. 26.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

1897. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for the production of albumoses. Jan. 24.

1989. J. U. Lloyd. An improved method of obtaining the salts of nicotine or kindred alkaloids in a concentrated state from the vegetable substances which contain them. Complete Specification. Jan. 25.

2224. H. J. H. Fenton. A process for the production of salts of dihydroxytartaric acid. Jan. 27.

2603. H. H. Lake.—From the firm of Lepetit, Dollfus, and Gansser, Italy. Improvements in the preparation of extracts of tannin. Feb. 1.

2882. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of a pharmaceutical compound. Feb. 4.

3000. A. W. Shirley. Improvements in or connected with menthol cones. Feb. 5.

3020. W. H. Claus, A. Rée, and L. Marchlewski. Improvements in the manufacture of saccharine. Feb. 7.

3391. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for the production of albumoses. Feb. 10.

### Complete Specifications Accepted.

1897.

6655. H. Oppermann and R. Goehde. Formic aldehyde compounds, and processes of preparing the same. Jan. 26.

7341. J. Y. Johnson.—From C. F. Boehringer and Soehne. Feb. 9.

8308. F. Richard. Vaporising apparatus suitable for converting methyl alcohol into formic aldehyde. Feb. 16.

17,169. A. F. Llobet. Manufacture of milky juice of cereals. Feb. 2.

## XXI.—PHOTOGRAPHY.

### Applications.

1136. H. Rheinlander. Improvements in manufacturing films for animated photography. Jan. 19.

2570. J. B. J. Sekutowicz. A process for the direct transformation of a photographic, calcographic, or pellicular autographic plate into photoplastic reliefs. Feb. 1.

3257. C. Henry. Smokeless magnesium powder for photography, and apparatus for employing the same. Feb. 9.

### Complete Specifications Accepted.

1897.

6769. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Prepared sensitised leather for photographic purposes. Feb. 16.

28,596. J. Hauff. Photographic developers. Feb. 16.

29,832. A. Weiss. Method of and apparatus for igniting flash powder in taking photographs by artificial light. Feb. 16.

30,115. V. I. Feeny.—From F. von Bentivegni, United States. Process of applying colours to photographs. Feb. 16.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

1339. J. Plummer. An improvement in the manufacture of water-absorbing explosives. Jan. 18.

1629. H. H. Lake.—From Explosivstoff-werke Spiralit Gesellschaft mit beschränkter Haftung, Germany. Improvements in explosives. Jan. 20.

1631. L. Gathmann and E. Gathmann. Improvements in safety fuses for high explosive shells. Complete Specification. Jan. 20.

1650. The Hon. R. W. H. Rodney. Improvements in fog-signals. Jan. 21.

2264. A. de Stubenrauch. Improved process of manufacturing smokeless gunpowder. Jan. 28.

2405. S. J. von Romocki. Safety explosive. Jan. 29.

2412. E. Gurovits and M. Bals. Process for rendering more effective lead oxide-hyposulphite as constituent part for priming or firing compounds. Jan. 29.

2730. D. G. Fitz-Gerald. A non-inflammable pyroxylin compound. Feb. 2.

2809. J. G. Dixon. Improvements in detonators for fog signalling. Feb. 3.

3088. E. S. Clark. Improvements in or relating to explosives or explosive charges for use in artillery and for other purposes. Feb. 7.

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Monday, April 4th:—
Election of Officers and Five Members of Committee.
Mr. W. J. Dibdin. "The Bacterial Treatment of Sewage containing Manufacturing Refuse."

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April 1st:—
Election of Officers and Five Members of Committee.
Mr. J. Carter Bell's paper (this Journal, 1898, 11). Adjourned discussion.
Mr. R. J. Flintoff. "Metallic Tannate Lakes of Basic Colours on Cotton Cloth."
Mr. J. Carter Bell. "Comparative Effect of Lime and other Chemicals on Sewage."

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Tuesday, April 5th:—
Election of Officers and Committee.
Dr. C. M. Aikman. "Progress of Agricultural Chemistry during the last Sixty Years."
Mr. Jas. Hendrick, B.Sc. "Methods of Control Analysis in the Fertiliser Industry."

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## NOTICES.

### Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

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### Telegrams.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

### Annual General Meeting.

The Annual General Meeting will be held in Nottingham on the 13th, 14th, and 15th July next. A Programme is in preparation. Tickets of Membership will be issued in time for the meeting.

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Hurter, Dr. F., Holly Lodge, Cressington Park, Liverpool. March 5th.
Langbeck, H. W., The Park, Loughton, Essex. March 6th.
Lassing, Dr. H., 19, Park Place, New York, U.S.A.
Napier, Jas., 89, London Road, Ipswich. March 17.
Pritchard, W. S., Farnworth, Widnes. February 16th.

## Liverpool Section.

*Meeting held Wednesday, February 23rd, 1898.*

DR. C. A. KOHN IN THE CHAIR.

### THE COMPOSITION OF INDIAN SUGAR-CANE AND RAW SUGARS.

BY J. WALTER LEATHER, PH.D., F.I.C.

During the last three years I have had occasion to carry out for the Government of India a number of investigations relative to the growth of sugar cane in India and the manufacture of the raw sugar.

In introducing this subject of the Indian Sugar Cane Industry to your notice, I wish to refer very briefly to the areas involved and the methods of cultivation, &c.

*Areas.*—The average area under this crop in India is very considerable, and is distributed as follows:—

| | Acres. |
|---|---|
| Punjab | 317,000 |
| North-West Provinces and Oudh | 1,300,000 |
| Bengal | 1,040,000 |
| Central Provinces | 10,000 |
| Bombay | 76,000 |
| Madras | 116,000 |

Although there is such a large proportion of the total area situated in the North-West Provinces and Bengal, the crop is nevertheless very well distributed through the empire. Indeed in many parts every village has at least one or two small plots of cane.

*Exports and Imports.*—The trade returns show that India exports an average of 63,000 tons of sugar, valued at Rx. 845,000, and imports 135,000 tons per annum, valued at Rx. 3,045,000. There is thus a nett import of some 72,000 tons.

But this amount is inconsiderable in comparison with the total amount of sugar produced. The outturn of raw sugar varies from about 1 ton up to some 4 tons per acre. This latter is, however, only realised in those parts, such as the Bombay Deccan, where the crop is heavily manured. In many parts the system of cultivation is bad, and the amount of manure employed small; the outturn then falls to from ½ ton to possibly 2 tons.

*Cultivation.*—In reference to the cultivation of the cane, I shall only mention that generally the crop is grown either on the flat, as in the case of a cereal crop, or on ridges, as one does for potatoes or roots in this country. The West India practice of planting three cuttings together in pits is but little employed.

The crop has to be artificially irrigated during the hot weather, and in all experiments which have been carried out at the farms to test the point, the advantage of growing the crop on ridges, in which case the stem is always kept out of the irrigation water, has been made manifest by an increased crop.

*Manufacture.*—The crop is nearly always an 11 to 12 months' one in India. Cutting commences when the top leaves begin to turn yellow, and since the whole of the crushing and boiling of the juice is carried out by the cultivators themselves with small appliances, the operations continue for about a couple of months in any one part.

A few words descriptive of these appliances will not be out of place here, since the conditions under which the industry is carried on will then be more perfectly appreciated.

*Mills.*—Until some 15 or 20 years ago the mills employed were either of wood or wood and stone. They were of three types. One consisted of two horizontal wooden rollers. The power was obtained from one or two bullocks which turned a lever arm, and the perpendicular motion thus obtained was converted into horizontal by means of a horizontal toothed wheel working into a perpendicular one. The whole machine was naturally somewhat cumbrous, and those specimens which I have seen at work were very imperfect machines indeed.

A second description of wooden roller mill is employed in the Bombay Presidency, and consists of three perpendicular wooden rollers. The bullock power is transmitted *directly* from the lever arm to the centre roller, and from this to the others through a helical gearing. The cane is put in on one side between one pair of rollers and is passed back again between the second and third rollers. This mill, when well set up, is said to do very good work. It certainly presses the cane, and it is a wonderful example of a powerful wooden contrivance. But the friction must be very great indeed, and it is difficult to keep such a machine sweet and clean. It is being replaced by the iron mills, which I shall presently describe, though much more slowly than has been the case with the other wooden mills.

The third wooden contrivance which has been extensively employed for crushing cane is not a roller mill at all, but a pestle and mortar arrangement; a machine which is still universally employed for crushing oil seeds. The mortar is of either stone or wood, and has a hole in the side near the bottom of the cavity for the liquid to pass out. The pestle consists of a long wooden beam, which is turned by a bullock; pressure is given by a man sitting on a horizontal beam which is attached to it. I have not had an opportunity to test the pressing power of this machine for cane. It presses oil seeds comparatively well for such a contrivance, the oil-cake containing from 10 to 15 per cent. of oil. But although it may have pressed cane fairly well, it is a machine which could not be kept clean, and much sugar must have been lost by fermentation.

These crude wooden appliances have been replaced in most parts of India by small iron mills, consisting of two or three perpendicular rollers, about 12 to 18 ins. high and 6 to 10 ins. in diameter, which are driven by bullocks through a lever arm, and there can be no doubt that this change has meant a saving of many thousand tons of sugar per annum to the cultivators. Those interested in Indian agriculture have to thank Messrs. Burrows, Thomson, and Mylne, of Behar, for perseveringly urging the advantages of the iron mill for some 20 years, before it became generally acknowledged and employed.

It will be sufficient to add here that with these mills from 100 lb. to 500 lb. of cane can be crushed per hour, and that one up to four bullocks are employed at a time.

*Evaporating Pans.*—For the evaporation of the juice, open iron pans, generally of greater depth than width, were until recently employed. Now shallow pans, measuring 5 to 8 ft. in diameter and from 4 to 8 ins. deep, have been adopted in many parts, this again being a distinct improvement. Steam vacuum pans, such as are employed in refineries, are of course unknown to the Indian Ryot. From 200 to 1,000 lb. of juice is evaporated at one time in the shallow pans referred to.

*Skimming.*—The scum which forms on the surface of the juice when it is boiling is skimmed off by an iron, or sometimes wickerwork, sieve.

*Crude Sugar.*—In most parts of India the juice is boiled down until so little water is left in it that it will solidify on cooling. The pan is removed from the fire and allowed to cool, but before the mass is quite solid it is transferred to a mould, and there allowed to remain until quite hard. This sugar has a colour varying from yellow to very dark brown, according to the amount of care which is taken in skimming, &c. It is called "gur" or "gul."

*Output.*—The outturn of crude sugar per acre varies very greatly indeed, depending on the methods of cultivation, the variety of cane grown, and the amount of manure employed. I have seen crops which produced only 500 or 600 lb. of sugar; I have seen others which produced 14,000 lb. of raw sugar. But these are extremes, and it may be said that whilst in the North-West Provinces and Oudh the average outturn will not be more than 1 to 1½ tons, that obtained by the cultivators near Poona is from 3 to 4 tons, and even more.

The methods of refining sugar I shall refer to in a subsequent paragraph. The foregoing will explain the general conditions under which the industry is carried on.

*Field Experiments* are being conducted at four of the experimental farms, namely, Poona, Cawnpore, Dumraon, and Burdwan, the object being to determine the effects of manuring and to test the comparative qualities of varieties of cane. It is considered that these field experiments must be carried on for some years still, before definite conclusions can be drawn regarding them.

I am able, however, to give briefly details regarding the more strictly chemical investigations, which were carried out under my direction.

Before placing the figures before you, it will be well that I should mention that the juice was always analysed at the field. A chemical balance cannot be readily carried about in camp, but many volumetric processes may be executed in a tent. The cane sugar was invariably determined by Clerget's methods, and the glucose by Fehling's volumetric method. The raw sugar was analysed either in camp or later on at the laboratory at Dehra, according as to which was the most convenient.

In the case of the juice, the results are in all cases expressed in grammes of sugar per 100 grammes of juice.

*The Juice.*—The juice of some 30 varieties of sugar cane has been analysed in different parts of India, the cane having been grown under varying conditions, and although reliable information has been obtained in some directions, in others the results cannot be considered conclusive.

One question which is of paramount importance in connection with this, as indeed with most field crops, is "Have we as good varieties as other countries, or will it be desirable to import new ones?" and with a view to obtaining information regarding this point, I determined the cane sugar and glucose in the cane crops in different places, grown both at the farms and also in the villages. The outcome of this work is to show that there are in India exceptionally good varieties, as the accompanying Table I. shows. The figures are average ones taken from a number of analyses.

Table I.

| Place. | Variety of Cane. | Cane Sugar. | Glucose. |
|---|---|---|---|
| | | Per Cent. | Per Cent. |
| Poona | Pundia | 14 to 17 | 1·0 to 1·8 |
| The Deccan | Kari Kabbu | 12 to 16 | 1·0 to 1·5 |
| The Deccan | Hulloo Kabbu | 15 to 16 | 0·1 to 0·5 |
| Cawnpore | Madrasi | 11·5 to 14·0 | 1·0 to 1·5 |
| Cawnpore | Matna | 15·0 to 17·0 | 0·3 to 0·5 |
| Bengal | Samsara | 14·0 to 15·0 | 1·0 to 1·5 |

These are some of the best varieties to be found in India, and I believe they compare so well with what is obtained in the West Indies, that I think one is justified in concluding that there is no need to go to other countries for new varieties of cane.

*Effect of transferring varieties long distances.*—Another question which was raised was whether the qualities of a cane are liable to suffer from its being transferred to another country. The information which has been so far obtained is not altogether conclusive. One of the first cases of this nature which came before me was that of two varieties which had been brought to Poona from the Mauritius. They both had the reputation of possessing a juice containing some 18 per cent. of sugar. They have been cultivated for three years at Poona with liberal amounts of manure, and in land which has proved to be exceptionally suitable to the crop, and with the result that the one has grown a juice containing 12 to 14·7 per cent. of cane sugar and 1·4 to 1·0 per cent. glucose, whilst the other contained 10·0 to 12·7 per cent. cane sugar and 2·0 to 1·5 per cent. glucose. It is to be noted, however, that the proportion of cane sugar has gradually increased in the succeeding crops, the lower proportions mentioned being those of the first year's crops, the higher ones those of the third year's crop.

Again, some of the Poona "Pundia" variety was grown at the Cawnpore and Dumraon farms during the last two years, and its juice has there only contained about 14 per cent. of total sugar, instead of the 16 to 18 per cent. which it contains in its normal condition at Poona. Thus, there is some indication that a variety suffers, at least temporarily, by being transferred. But whether the cause of this is really climate, or whether it is not due, in part at least, to difference of treatment, is not clear at present.

*Effect of manures.*—One of the principal objects of the extensive series of sugar-cane experiments which have been commenced at the Government farms in India, is to test the effect of manures on cane. I need not enter here into the details of these experiments since it is proposed to carry them on for some years longer, but I may mention that a number of different materials besides farm manure are employed in varying amounts at each of the farms. It is hoped that the experiments will eventually show what amount of manure may be most profitably applied, as also the comparative value of the several materials. But I will refer for a moment to the effect which manuring has had on the juice.

Each year the juice from the differently manured plots has been analysed, and for two years no effect could be observed in the juice corresponding to the difference in the manuring. The variety "Pundia," which was cultivated on the differently manured plots at Poona, contained in 1895 from 14·2 to 17·3 per cent. cane sugar, and in 1896 from 14·2 to 17·3 per cent. sucrose, and from 1·0 to 1·8 per cent. glucose in the juice; these being the analyses of the juice from 12 and 15 plots respectively.

A variety, the "Matna," ,which has been grown at Cawnpore on a series of 9 plots with varying amounts of manure, gave a juice in 1895 containing from 13·6 to 17·5, and in 1896 from 15·0 to 17·0 per cent. of sucrose, and from 0·3 to 0·5 per cent. glucose, and here again there was no evidence that manuring had any effect on the cane.

It so happened, however, that in each of these cases the cane had been grown under very much the same conditions as those to which it had been accustomed for generations. The Poona "Pundia" is usually grown with very heavy dressings of manure, the cultivators commonly applying 30 and

40 tons of manure per acre; and it was grown with similarly large amounts of manure in the experiments.

The "Matna" variety of the North-West Provinces and Oudh is commonly grown by the cultivators with comparatively small amounts of manure, say, 3 or 4 tons per acre, and it was likewise grown in the experiments with varying, but small, amounts of manure.

In 1896–97 this same variety, the "Matna," was included, with several other varieties at Cawnpore, in an experiment in which all were very heavily manured, and I was somewhat surprised to find that it had grown very differently. The weight of the crop had been very considerably increased, and whilst previously only 45 to 50 per cent. of juice had been expressed from it by the mills, we obtained an average of 60 per cent. from the heavily manured crop. On the other hand, the percentage of sugar had fallen from 15 or 16 to 11 or 12. The total weight of sugar per acre had of course been much increased. But the point which seemed to me to be so significant was that by placing a variety under entirely new manurial conditions its whole composition appeared to be changed. Some confirmatory evidence was obtained in the case of other varieties at Dumraon, and although this question cannot be said to be at all completely investigated, still the instance quoted showed at once how careful one has to be in drawing conclusions regarding the qualities of any variety, and the necessity there is for judging it when grown under normal and not abnormal circumstances.

Another important question is naturally, whether this effect of manure is only temporary and whether such a variety as the "Matna" will, if grown under the new conditions of liberal manuring, gradually regain its normal state.

*Inversion.*—Working under the crude circumstances which I have already briefly indicated, it is only to be expected that there must be some loss of cane sugar owing to inversion whilst concentrating the juice. The conditions of the industry prohibit the employment of any but the very simplest means for preventing inversion. In some parts the cultivators employ a little wood ashes to neutralise acidity, but this is exceptional. The only experiments which I made were on the employment of caustic lime (more or less carbonated).

Milk of lime was added to a certain proportion of the juice for each pan to such an extent that this portion was rendered slightly alkaline, and then the unneutralised portion was mixed with it so as to render the whole slightly acid. Such a method is one which, I believe, the cultivators could be taught to use. I exhibit in Table II. the results of a series of such experiments which were made in 1896.

TABLE II.

| Juice. | Cawnpore Experiments. | | | | | | | | | | Poona Experiments. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | "Matna" Variety. | | | | | | "Saharanpuri" Variety. | | "Madrasi" Variety. | | Plot 7. | | Plot 9. | |
| Cane sugar | 16·74 | | 16·58 | | 16·03 | | 15·69 | | 14·10 | | 15·96 | | 15·03 | |
| Glucose | 0·31 | | 0·29 | | 0·32 | | 0·98 | | 1·35 | | 1·52 | | 1·72 | |
| Total | 17·05 | | 16·87 | | 16·35 | | 16·67 | | 15·45 | | 17·48 | | 16·75 | |
| Ratio:—<br>Glucose to 100 parts of total sugar. | 1·8 | | 1·7 | | 1·9 | | 5·8 | | 8·7 | | 8·69 | | 10·2 | |
| Amount of Acidity neutralised. | No Lime. | Indefinite. | No Lime. | Alkaline. | No Lime. | $\frac{3}{4}$ | No Lime. | $\frac{4}{5}$ | No Lime. | $\frac{26}{27}$ | No Lime. | $\frac{9}{10}$ | No Lime. | $\frac{9}{10}$ |
| Crude sugar:— | | | | | | | | | | | | | | |
| Cane sugar | 72·04 | 74·96 | 71·85 | 75·76 | 73·80 | 73·94 | 70·92 | 78·43 | 69·06 | 77·41 | 75·95 | 79·88 | 73·67 | 78·00 |
| Glucose | 10·57 | 6·66 | 9·91 | 1·62 | 7·33 | 5·31 | 9·94 | 5·08 | 12·82 | 7·57 | 11·90 | 9·00 | 12·47 | 9·60 |
| Total sugar | 82·61 | 81·62 | 81·76 | 77·38 | 81·13 | 79·25 | 80·86 | 83·51 | 81·88 | 84·98 | 87·85 | 88·88 | 86·14 | 87·60 |
| Ratio:—<br>Glucose to 100 parts of total sugar. | 12·8 | 8·16 | 12·1 | 2·09 | 9·0 | 6·70 | 12·3 | 6·08 | 15·6 | 8·9 | 13·5 | 10·1 | 14·4 | 10·9 |

It will be seen that the formation of glucose was largely prevented. The colour and texture of the finished product was very much better than usual, and these are points which affect the price in the Indian bazaar very considerably.

*Composition of Raw Sugars.*—As mentioned in an earlier paragraph, the usual custom is to boil the juice down until it will set to a solid block, the so-called "gur" or "gul," on cooling. Another, though less usual, practice in most parts consists in leaving rather more water in, and in this case a semi-liquid mass of molasses, sugar, and crystal is obtained on standing. This product is called "rab," sometimes also "gur."

Regarding the composition of the solid "gur," it naturally varies a good deal. The analyses detailed in Table II. are of "gur," which had been very carefully made, and was somewhat above the average. The proportion of sucrose in those samples varied from 70 to 75 when no lime was added to neutralise the acidity of the juice, and it rose to 80 per cent. in some of the neutralised samples. The proportion of glucose varied from about 7 to 12 per cent. in the untreated samples. But the "gur" prepared by the cultivators falls below this standard and will frequently contain only 60 to 65 per cent. of sucrose with 10 to 15 per cent. of glucose. The proportion of ash will vary from 1½ to as much as 4 per cent., and that of water from 5 to 15 per cent.

*"Rab."*—The composition of "rab" differs from that of "gur" simply in the proportion of water which it contains; this will vary from 15 to 25 per cent.

*Refining.*—There is now a number of sugar refineries in India, fitted up with all modern plant and managed by Englishmen; of this industry I need not say very much. I may mention that one difficulty, which is met with by the sugar refiner, consists in the fact that the caste of the Hindoo prohibits him from eating sugar which has been in contact with bone-char. Some of the refineries nevertheless clarify by this means; but others purify by re-crystallisation entirely.

Although I do not know what amount of sugar is annually refined in India, I may say that the demand for refined sugar is not nearly so great as might be perhaps imagined. There is little doubt that the greater part of the sugar produced is consumed in the simple form of "gur."

There is, nevertheless, a not inconsiderable business carried on throughout India in the refining of sugar by native methods. One of these processes consists in applying a wet weed to the surface of a mass of sugar, by which means the molasses are induced to drain away, and a layer of fairly white sugar is left behind. In applying this method the raw sugar is placed in a vessel with a false bottom, the weed is applied, and each day the top layer of refined sugar is scraped off, after which the weed is applied to the next layer, and so on until the whole mass has been operated upon. Sometimes the vessel is a small earthenware one, measuring some 2 ft. in diameter and 2 to 4 ft. in depth. Sometimes large baskets are employed, measuring 8 ft. in diameter and several feet deep. Such sugar is comparatively pure, containing as much as 95 to 97 per cent. of sucrose.

Another method of refining consists in placing bags of the semi-solid "rab" one upon another, and allowing the molasses to gradually drain away. I have no exact information as to the composition of this product, but it varies very considerably indeed, and is often as impure as a good "gur" would be. In carrying out these native processes the molasses are almost entirely destroyed by fermentation. Candied sugar is made, but this ranks as a sweetmeat.

It will be readily appreciated that the crystallised sucrose of the "rab" can be separated in a much more rapid and cleanly manner by a centrifugal machine than by the native processes, and the molasses would of course be saved.

With this end in view, Messrs. Thomson and Mylne, who so perseveringly and successfully introduced the iron sugar mill, have for years endeavoured to induce the people on their large estate to separate the molasses from the sugar crystal in the "rab" by means of a small hand centrifugal machine. It is only those who have come practically in contact with the Indian people who can appreciate the extreme slowness with which they adopt any alteration in their methods or appliances. The iron sugar mill was exhibited for years in different parts of India before it was really adopted, and, although it has replaced the old wooden contrivances in most parts, still one meets with the indigenous machine here and there even now, and similarly with the hand centrifugal sugar separator. No better example of the difficulty which one meets with in the introduction of a novelty to the Indian cultivator could be quoted. Mr. Mylne told me that he worked one of these machines for 10 successive seasons at one of the cross-roads before the first was bought. Now the people in that neighbourhood appreciate its value; and I am convinced that its use will be very largely extended. The usual outturn obtained is about 50 per cent. of sugar-crystal from the "rab." The molasses when boiled down form a "gur" of quite as good a quality as much of that which is made directly from the juice.

This semi-purified sugar has approximately the following compositions:—90 to 95 per cent. of sucrose; 1 to 2 per cent. of glucose; 0·8 to 1·5 per cent. of ash; and 0·5 to 2·0 per cent. of water.

*Amount of Juice remaining unexpressed from the Cane.*—One question which interested me very much was how much juice was left in the crushed cane. This question has doubtless been studied by other chemists, and I do not think it will be surprising when I state that the amount is frequently very large. But whilst I have seen the bare fact mentioned, I have not met with any suggestion that a relationship appears to exist between the amount of juice unexpressed and the proportion of crude fibre in the cane.

In order to prevent confusion, it will be well if I mention very briefly what method I employed in arriving at the figures which are set out in Table III.

The proportion of juice expressed by the mills was readily enough ascertained, since all the cane and juice was regularly weighed throughout the whole series of experiments. The proportion of total juice was, however, not so readily arrived at. The first difficulty is that of obtaining a fair sample, and I came to the conclusion that the only practicable way under the circumstances in which I had to work was to take average-sized canes, and cut thin slices from different portions of them. The "crude fibre" in several hundred grammes was then separated by first washing the slices with cold water for a couple of hours, after which the material was placed in a stout calico covering and pounded in a mortar, it was then again washed in cold water and finally dried. I adopted this method of washing with cold water only since what I wished to weigh was that part of the cane which was solid, *i.e.*, other than the juice. The results obtained were very concordant, and quite sufficiently accurate for the purpose. It was then assumed that what was not crude fibre was juice in the original cane. The results of some experiments carried out in this manner are detailed in Table III.

Table III.

| — | Poona Cane. | | Cawnpore Experiments. | | | | |
|---|---|---|---|---|---|---|---|
| | Plot 2. | Plot 11. | "Dhaul." | "Dikchan." | "Matna." | "Saharanpuri." | "Madrasi." |
| Total juice in cane | 91·6 | 91·5 | 87·3 | 89·0 | 85·2 | 89·6 | 90·0 |
| Crude fibre | 8·4 | 8·5 | 12·7 | 11·0 | 14·8 | 10·4 | 10·0 |
| Juice expressed by mills | 71·6 | 72·2 | 51·2 | 56·0 | 45·4 | 57·4 | 64·1 |
| Juice remaining in refuse | 20·0 | 19·3 | 36·1 | 33·0 | 39·8 | 32·2 | 25·9 |
| Calculated composition of refuse cane:— | | | | | | | |
| Crude fibre | 29·4 | 30·7 | 26·1 | 25·0 | 27·1 | 24·3 | 27·8 |
| Juice | 70·6 | 69·3 | 73·9 | 75·0 | 72·9 | 75·7 | 72·2 |

Now the point which is to my mind so striking in these results is this, that the composition of the refuse cane is so nearly alike in all cases. The Poona samples contained a somewhat lower proportion of juice than those at Cawnpore, but the difference is not great, and it is remarkable how nearly alike is the refuse cane obtained from five varieties at Cawnpore farm, varieties which, I may point out, differed much among themselves in habit, sugar content, &c. The refuse consisted almost uniformly of one quarter crude fibre and three quarters juice. Nor is the explanation of this far to seek. The cane after once being reduced to a pulp in the mill must be assumed to be a simple sponge, from which the juice is only prevented from running by the simple property of adhesion. Consequently a pound of this spongy material will retain approximately the same amount of juice, whatever be the variety of cane crushed. And it also follows that it is only the *surplus* juice which runs away in the mill. The amount of juice which is thus retained by the crushed cane depends, therefore, on the amount of crude fibre in the original cane.

In order to illustrate the importance of this matter to the cane grower, consider for a moment the two extreme cases in the Table III. One, the "Matna" variety at Cawnpore, contained about 15 per cent. of crude fibre and 85 per cent. of juice. The 15 parts of crude fibre retained 40 parts of juice and permitted only the other 45 parts to run out at the mill. The variety "Madrasi" (grown at the same place and crushed with the same mills) contained 10 per cent. of crude fibre and 90 per cent. of juice. These 10 parts of fibre held an almost precisely similar proportion of juice as did the fibre of the "Matna" variety, but this came to only 26 parts and, consequently, 64 parts of juice were obtained at the mill.

It becomes, therefore, a matter of no small importance to grow cane which will contain a low proportion of crude fibre.

### DISCUSSION.

Mr. A. **SMETHAM** asked if it would not be possible to carry on the manufacture on more scientific lines, by means of factories properly situated. Dr. Leather had pointed out that there was no very great difference in the percentage of sugar in the juice of canes manured by different manures, but he had not made it clear to him how far it was profitable to manure the land. Although the composition of the juice was practically constant, the quantity was increased by manuring. He would like to know what was the profitable limit in manuring sugar cane. They knew in England, in manuring crops, the first few hundredweights of artificial manure paid handsomely, and that beyond that, the increased yield due to manure rapidly declined, and he would like to know what return there was to be derived from manuring. Doubtless, Dr. Leather would be able to tell them that the manuring was profitable, and that if the raiyat would only apply larger quantities of manure, a greatly increased yield might be obtained, and so a more profitable cultivation result.

Mr. **SIGMUND STEIN** said that it had been mentioned that the exports from India were less than the imports by some 72,000 tons. He thought that large quantity of sugar which had to be imported into India, and which came from Europe (Marseilles, Trieste, and Hamburg), and increased from year to year, could and should be made in India. Some of the sugar journals abroad lately mentioned the intention of French syndicates to start raw-sugar works in India on a large scale with the newest appliances and latest methods. After what Dr. Leather had told them he was rather anxious to know how those syndicates worked. They had been told that the yield of sugar per acre varied from 1 to 8 tons, but according to the figures given, he calculated the quantity of sugar gained per acre to be about 3 to 5 tons. In Europe the yield was 1·25 tons of sugar per acre; in the West Indies about 1·8. It was astonishing that in the crude state of the sugar industry in India they should produce such a large yield per acre.

Prof. **CAMPBELL BROWN** said that in regard to the manuring of the crops, he was afraid that the native and even the British planter was not willing to spend money on artificial manures, and that practically the manures that could be employed were mainly farmyard manure, waste or spoiled oil-cake, and other organic material. He would like to know Dr. Leather's experience regarding the manure available for Indian agriculture.

Dr. **LEATHER**, replying, said a great deal of the work which he had been engaged upon in India was interesting to chemists generally. Mr. Smetham had raised the question of factories, that was, carrying out the sugar industry on the factory system. The basis of the Indian sugar industry was entirely different from that in other countries, it was carried on by the people almost entirely for themselves and not for export at all. The quantity produced was probably between four and six million tons per annum, and the amount exported was a mere fraction of the whole, similarly the amount imported was comparatively small, and had no important bearing on the industry at large. Years ago there were more factories than there were to-day, but they did not pay and, consequently, that part of the industry had disappeared. What now existed was simply a small number of refineries, with European appliances for the manufacture of a comparatively small quantity of refined sugar to be consumed by Europeans. The question of refining did not play a part at present. Another question which Mr. Smetham had asked was with regard to the amount of manure and the percentage of sugar. He considered the results of the field experiments, which had been obtained up to the present, were premature. He might say they could manure very heavily indeed, and still get an increased profit. The probability was that one could apply from 20 to 30 tons per acre of city manure containing $\frac{3}{4}$ per cent. to 1 per cent. of nitrogen, and find it an economical business. In the greater part of India the sugar cane was not manured to that extent. Mr. Stein had spoken of the large excess of imports over exports. It was a large figure, but when one considered the production of sugar, it was a trifle. A large portion of it was beet-root sugar; but it showed there was something radically wrong with the conditions of the production of refined sugar in India, when one considered that sugar could be grown and made and refined in Europe and sent to India for the use of Europeans there, and still beat the sugar refined by European machinery in India. He did not know how the refineries in India had progressed. Mr. Stein had been surprised at the high yield of sugar per acre; it certainly was surprising. In Bombay people could get from 3 to 4 tons of raw sugar per acre. In most parts of India, however, the yield was much less than this. Professor Campbell Brown had asked about the use of artificial manure. Very little was used in India at all, and that little was made in Calcutta.

## London Section.

*Meeting held on Monday, February 7th, 1898.*

DR. R. MESSEL IN THE CHAIR.

## ON THE CURING OF MALT IN RELATION TO COLOUR VALUE.

BY J. W. LOVIBOND.

BEFORE dealing with the subject-matter of the paper I restate the fundamental laws upon which the power to measure a colour sensation is based.

### *White and Coloured Light.*

1. Normal white light must be regarded as being made up of the six colour rays, "red, orange, yellow, green, blue, and violet," in equal proportion; when the rays are in unequal proportion the light is abnormal and coloured.

2. The particular colour of an abnormal beam is that of the one preponderating ray if the colour be simple, or of the two preponderating rays if the colour be complex.

3. The rays of a direct light are in a different condition to the same rays after diffusion and give rise to a different set of colour phenomena. *Note.*—For the present this limits precise colour work to the measurement of diffused daylight.

4. The vision is not sensitive at the same time to more than two colour rays in the same beam of light; the colours of any other abnormal rays being merged in the general luminosity of the beam.

5. The two colour rays to which the vision is simultaneously sensitive are always adjacent to each other in the spectrum order, red and violet being considered adjacent for this purpose.

6. The vision is unable to appreciate colour in an abnormal beam of light outside certain limits, either—

i. Excess of luminosity may mark the colour, or,

ii. The luminous intensity may be too low for the preponderating rays to excite a definite colour sensation.

7. The length of time required by the vision to appreciate a particular colour is longest for the red, increasing as the spectrum is ascended, the minimum being in the violet.

### *Colour Constants.*

8. The colour of a pure substance at a particular density is constant. *Note.*—This law affords a means of checking the standard colour scales, as by matching them with pure coloured substances of known density any departure from absolute accuracy can be at once ascertained.

9. Every substance has its own specific rate of light absorption, developing definite colour sensations for definite increasing densities.

A wide distinction must be made between the terms colour-depth and colour-character. When we speak of a malt as being "light" or "dark" in colour we only express our idea of the "colour-depth" of the sensation without regard to the colour-character. It is only when a number of malts, similar in colour-depth, but varying in quality, are brought together, and the constituents of the colour are separated by analysis, that we realise how great a variety of colour-character is found under a single expression of colour-depth.

In a paper read before this Society on a kindred subject last session it was shown that the colour-depth of a malt extract was determined mainly by the depth of the orange factor, the yellow factor having comparatively little influence in deepening the colour. The effect of an increase in the proportion of yellow is mainly to alter the colour-character without materially increasing the colour-depth. It was demonstrated also by a series of practical experiments that a malt having a greater proportion of yellow to a particular proportion of orange produced a beer more pleasing in colour, finer in flavour, and having better keeping properties than a malt possessing a lesser proportion of yellow to orange. The colour-depth of the two malts may be the same, their only common feature being equality in the value of the orange factor at one particular stratum of the solution.

Under these circumstances it is of importance to those interested in the malt industry that colour-depth quotations should be based on a common understanding on two points, namely, (1) the use of the same percentage of malt and the same method in preparing the solution, and (2) an agreement as to the thickness of stratum at which the extract shall be measured for the quotation of the colour-depth. The methods adopted for this paper may not be the best possible; they have, however, certain advantages, which will become evident at a later stage.

### *Preparation of the Extract.*

Mash 100 grms. of crushed malt in 350 c.c. of water at 74° C. in a 500-c.c. graduated flask; digest in a water-bath at 63° C. for 48 mins.; cool and make up to 500 c.c. with water. After filtering, measure the colour, beginning at the 1-inch stratum, and progressing at intervals of 1 inch up to 9 inches, or, in the case of high-dried malts, until the colour becomes too deep to read. The results will be obtained in terms of orange and yellow, and black also if present. These may be tabulated or plotted in curves, the colour units supplying the ordinates and the strata thicknesses the abscissæ.

### *Quotation of Colour-depth.*

Discrepancies must always arise when different thicknesses of strata are used in the quotation of the colour-depth. This arises from the varying rate of colour-absorption for different qualities of malt. The 1-inch stratum is most generally used, and is found convenient where colour-depth alone is required. It does not, however, furnish a good basis for classifying colour-character. The 3-inch stratum has been found the most suitable for this purpose, and is adopted here for the following reasons: The general principle that the greatest proportion of yellow to orange produces the best results does not hold with the 1-inch and 2-inch measurements, the order being, in fact, reversed at these two points. Good malts are found to have a lower proportion of yellow to orange in the first two strata thicknesses, and a higher proportion in the terminal measurements than bad malts. The curves of proportions of the various malts must cross at one point or another, the point of crossing being a neutral position in their relations towards each other. An examination of Chart A, which contains the proportional curves of a large number of malts varying in depth of colour, will show that the 3-inch measurement is about the middle of the zone of crossings of the whole, and is, therefore, the most neutral point of the series. It is for this reason that the 3-inch stratum was selected for the quotation of colour-depth, as well as the starting-point for classifying the malts. The points of crossing are so regular in their variations as the colour-depth increases that they almost constitute a curve in themselves, which would be more regular if a wider range of malts had been used for the averages.

### *Colour proportion Curves.*

In the absence of a simple method for co-relating the variations in the colour-measurements with variations in the quality of the malt, it was found that they could be co-related by means of a curve showing the proportion between yellow and orange at the different strata. I have termed this curve of colour-proportions the secondary curve, to distinguish it from the curves of the yellow and orange colours themselves—the primary curves. The secondary curve is made by dividing the figure for the orange into that for the yellow colour at each stratum and plotting the results in a curve, as in any of the charts appended. The continuous line represents the secondary curve of the sample of malt under consideration; the dotted line shows the normal or proper curve for malt of that colour-depth, arrived at experimentally in the manner explained below.

The co-relation of variations in the secondary curves with variations in the quality of the malt was established by direct experiment. It was found that good beer could be brewed from pale malt whose secondary curve was of a bold regular type, similar to Chart A 1. It was found also that good pale ale could not be brewed from a malt whose curve was almost a straight line with an upward trend as in Chart B II. This form we now know to indicate a "hand-dry" but uncured malt. Nor could good beer be brewed from a malt whose secondary curve is inverted. This form is now known to indicate a mixture of cured and uncured malt. The example in Chart E is one of the independent confirmations of the truth of what has just been stated; it was furnished by a friend, who, unfortunately, had to deal with a large parcel of this malt.

There is as yet no evidence that the variations in the colour-curves are co-related to any of the chemical constituents of the malt, except those whose colour is affected by the curing temperatures and conditions; nor has any variation in the colour been found to indicate the nature of the body from which the malt was made. Stout or thin, native or foreign, give similar readings under equal curing conditions. The curves at present are simply records of the character of the curing process.

The work dealt with in the paper is divided into three main sections:—

Section A illustrates the method of establishing a series of working normals for comparisons in practical work.

Section B is a record of the colour changes which took place in a 40-qr. steep of malt, from the green stage before going to kiln, through the processes of drying and curing under ordinary working conditions.

Section C is a record of the colour changes in malt cured in hot air under known conditions of temperature and time. The temperatures chosen were 90°, 100°, and 110° C., whilst the time variations were intervals of one hour.

### A.—*The Establishment of Working Normals.*

The number of different malts used in establishing this series was 74, representing about 15,000 qrs. by four different makers. The measurements were made at intervals during the season 1896–97, as occasion arose, and were brought together for classification at the end of the season. Abnormal samples were avoided by the light of previous experience. A wider range of malt would have made the normals more typical, but these are found to answer practical purposes and have been proved to produce good beer.

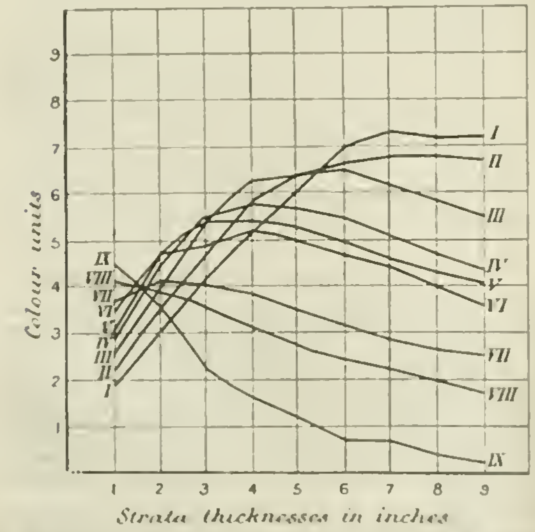
The malts vary in colour-depth from 2·1 to 16·0 of orange in the 3-inch stratum. As the colour-depth increases the secondary curve alters in shape as well as in position, so that it is necessary to have a series of normal curves instead of only one. As this alteration is continuous and almost regular throughout the scale of malts, it would be necessary to have a separate normal for every minute difference of colour-depth if we desired absolute accuracy; but for practical purposes it is found convenient and sufficient to classify all the malts lying between 2 and 3 units of colour in the orange together. Those lying between 3 and 4 units form the next step and so on, proceeding by unit differences. All the malts lying between 2 and 3 units at the 3-inch stratum that have given satisfactory results in practice have been tabulated, and the mean curve for the proportions of yellow to orange at each stratum has been plotted. This curve is called the *normal secondary curve* of all malts of that particular colour-depth; similarly with every colour-depth up to 16 units of colour. In order to save time when comparing the curve of an unknown malt with the normal, a zinc template has been made for each normal curve. The latter is plotted on paper of standard size, the template is placed in its proper position on the paper, and a pencil passed round it gives the normal curve for comparison immediately.

Previous observations have already made it evident that disaster follows the use of an under-cured malt. This is more likely to happen in very pale malts, although it is quite possible by neglect on the kiln to overheat a small portion—sufficient to produce the required colour-depth—and yet to leave a considerable portion under-cured. This state of things would be revealed in the secondary curve by a departure from the normal.

The lowest safe temperature at which very pale malts can be perfectly cured has yet to be determined; Section C, dealing with this part of the subject, not being exhaustive on this point. But experience shows that a temperature which only produces 2·0 to 2·1 units of colour-depth is dangerously near the unsafe point, as shown by the upward trend of the proportional curve before it begins to drop at the 7-inch stratum. No instances between 8 to 9 units colour-depth came under notice during the season. Their absence is indicated by a wider interval between the normals, and when a normal is now required for comparison at this colour-depth a mean is taken between the one above and the one below the theoretical normal.

It is only when the working normals are brought together for comparison, as in Chart A, that their true relation towards each other becomes evident. It can here be traced how the boldness of the secondary curve of the palest of the series gradually decreases as the colour-depth of the malt increases, until in No. VIII. it becomes a straight line. In the next curve (IX.) it begins to curve in the opposite direction, but with a still decreasing proportion of yellow to orange. Although this curve is in a similar reversed direction to the one which indicates an imperfectly cured malt, the two cannot be confounded, as the former (IX.) occupies a different position on the chart.

CHART A.



Nine normal secondary curves of malt for the season 1896-97 at intervals of 1 unit of orange in the 3 in. stratum :—

I. 2 to 3 of orange in the 3-in. stratum (mean of 14).
II. 3 „ 4 „ „ „ ( „ 18).
III. 4 „ 5 „ „ „ ( „ 10).
IV. 5 „ 6 „ „ „ ( „ 7).
V. 6 „ 7 „ „ „ ( „ 5).
VI. 7 „ 8 „ „ „ ( „ 5).
VII. 9 „ 10 „ „ „ ( „ 10).
VIII. 12 „ 13 „ „ „ ( „ 2).
IX. 14 „ 15 „ „ „ ( „ 3).

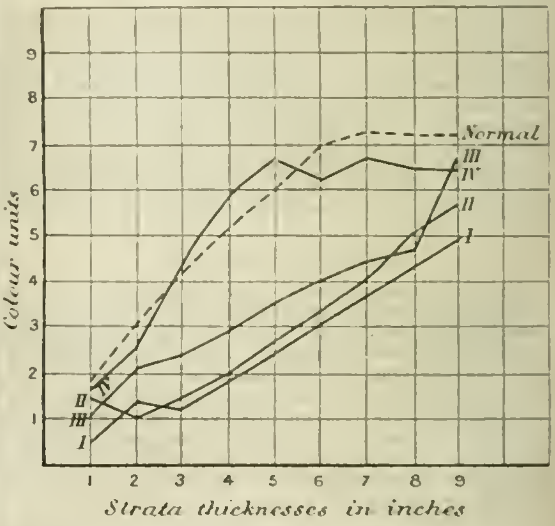
### B.—*Changes during Curing.*

This set of experiments was made with a 40-qr. steep in order to obtain some knowledge of the changes which take place during the drying and curing of malt, under ordinary working conditions.

Sample I. (see Chart B) was drawn eleven days from cistern, just before going to kiln, allowance being made for the excess of moisture present at this stage. The proportional curve is lower than that of any sample met with in commerce.

Sample II. (B) was drawn 24 hours after going to kiln, the last 12 hours being devoted to drying the malt on the kiln in the following way:—A firkin with the two ends out was placed in the middle of the kiln and a layer of malt about 1 inch thick was spread inside. The larger quantity of air which passed through this thinner layer of malt "hand dried" it in 12 hours; the maximum temperature registered was 49° C (120° F). The secondary curve (II.) is practically identical with that of the green malt, showing that simple drying at 49° C. caused no alteration in the colour constituents. A sample drawn 48 hours after going to kiln and "hand drying" in the manner just described gave similar results (max. temp. 50° C).

CHART B.



Secondary curves of green malt during curing :—
I. Green malt.
II. After 24 hours on the kiln at 48° C.
III. Air-dried for 72 hours at 60°. Before curing.
IV. After curing.

A sample (III. B) drawn 72 hours after going to kiln from bulk which was "hand dry" and ready for curing in the usual way (max. temp. 60° C.) The colour-depth of this sample is greater than that of the bulk of the malt (3·8 as against 2·8 units) after curing, and indicates that the curing process had already commenced next the tiles whence the sample was taken. It shows also the uneven nature of the ordinary process of drying up to the operation of curing. This unevenness is shown by the nature of the secondary curve, which is of an exceptionally bad type, associated from an early stage of the investigation with bad brewing quality.

The final sample (IV. B) was drawn from bulk in the usual way before unloading, the maximum registered temperature being 112° C. (235° F.). The secondary curve is a fairly good one, not quite up to normal but well within safe limits. We have here an example of what is effected by curing:—

The registered temperature only means that of the stratum in which the bulk of the thermometer happens to lie, in these instances about 1 in. from the surface of the tiles. The method takes no note of the time during which the temperature was maintained. It will be seen presently that time is a very important element in the curing of malt.

A distinctive feature in the uncured malt is the sharp depression at an early stage of the secondary curve. At first this was attributed to an error of observation, but repeat experiments showed it to be always present in malts which were known to be under-cured. It should, therefore, be considered as one of the indices of unsoundness.

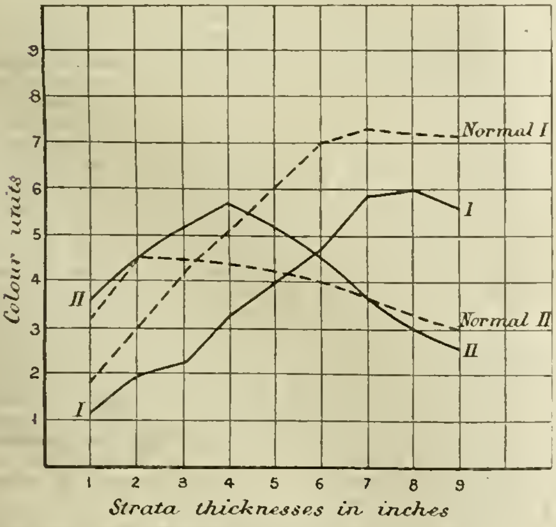
### C.—*The Effect of Time and Temperature in curing Malt.*

This set of experiments was undertaken in order to determine the effect of time and temperature on the curing of malt, for future guidance in the management of the kiln. It was felt that the present method of taking kiln temperatures gives but little insight into what really takes place on the kiln, even when taken at different depths in various parts of the kiln. The malt used in these experiments was from the same steep as in the B series, taken from the kiln 72 hours after kilning, but before curing, so that the artificial curing conditions might be comparable with the practical work.

The hot-air bath consisted of an iron vessel of a capacity of about 18 inches cubed, with a close-fitting lid carrying a thermometer, whose bulb was at the level of the malt. The whole was supported on a tripod and had two intervening false bottoms inside and a disperser outside the bath over the burner, to ensure an even distribution of heat and to obviate radiation from a single hot surface. The malt was heated gradually with the air in the vessel until the desired point was reached, when the time was taken. The vessel was only opened for a few seconds at the end of each hour to permit of the withdrawal of the sample; at no time did the temperature vary more than half a degree from the experimental point.

For the sake of clearness, only the first and last of the secondary curves are shown in each experiment: the nature of the intermediate results will be gathered from the description. Whatever we may learn from the many interesting features of these curves, our present concern is to trace the conditions under which the best type of secondary curve is produced, and to discover the conditions which govern the acquisition of the best colour-character.

Chart C.



Malt cured for 1 hour at 90° C.
" " 5 hours "

The first experiment was conducted at 90° C. (194° F.) with the following results:—

*One hour at* 90° *C.*—The secondary curve (C I.) approximates to the form of the normal, but is lower on the scale. The form indicates that curing has commenced, but has not advanced to obliterate the break at 3 ins., characteristic of under-cured malt. The colour-depth, which is suitable for pale ales, is the same as the malt cured on the kiln, but the colour-character is much inferior, as indicated by the lower position of the secondary curve.

*Two hours at* 90° *C.*—No increase in colour-depth, but the colour-character is slightly improved.

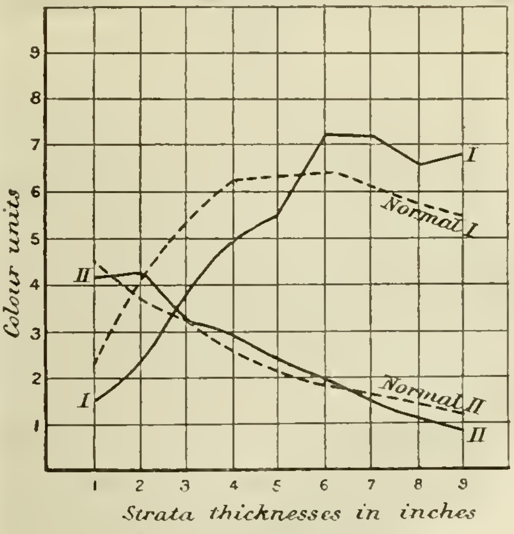
*Three hours at* 90° *C.*—There is a marked discrepancy in this curve with either that above or below, suggesting the probability of an error in marking the sample.

*Four hours at* 90° *C.*—The curve has improved, and is now equal to the normal for this particular colour-depth (6·2 colour units). This depth is low for mild ales, but too high for pale ales.

*Five hours at* 90° *C.*—There is a still further improvement in the character of the secondary curve (C II.), which is now considerably better than normal at the increased colour-depth (8 to 9). This happens to be the one without an example drawn from actual practice, so that a mean has been taken of the one above and the one below. The actual colour-depth is 8·8 units, which is a good working depth for running ales.

It would be unsafe to lay too much stress on a single set of experiments, but so far as they have gone, they seem to indicate that 90° C. is too high for curing pale ale malt. When the secondary curve approaches the normal in character, it is too high for the purpose of the pale ale brewer. It appears to indicate also that 90° C. is a good temperature (with not less than five hours curing) for mild ale malt, as the colour-depth is suitable and the colour-character better than normal. Further experiments with a slightly lower temperature and a longer time in curing may possibly show still better results.

Chart D.



I. Malt cured for 1 hour at 100° C.
II. " " " 3 hours at 100° C.

The second experiment was conducted at 100° C. (212° F.) with the following results:—

*One hour at* 100° *C.*—This gives a full pale ale colour-depth (4·3 units), but an inferior colour-character, as shown by the irregularity of the curve (D I.).

*Two hours at* 100° *C.*—The colour-depth is exactly the same at the standard 3-in. stratum as that of the five hours' curing at 90° C., but a comparison of the curves will show that the character is greatly inferior.

*Three hours at* 100° *C.*—The colour-depth has increased to 14·5, corresponding to the deepest colour of the season's series, and suitable only for very dark ales. The secondary curve (D H.) is a little better than normal.

The inference that may be drawn from the experiments up to this point is that the employment of a longer time and a lower temperature in securing a particular colour-depth gives better results than a shorter time and a higher temperature.

The third series of experiments was conducted at 110° C. (230° F.) with the following results :—

*One hour at* 110° *C.*—This is the only one of the series that falls within the range of ordinary ale brewing, standing as it does at 5·6 units of colour-depth. The colour-character is inferior to the normal.

*Two hours at* 110° *C.*—The colour-depth here has increased to 17·0, which is out of the range of ordinary ales. The secondary curve is interesting as continuing the downward tendency (see Chart A) of the season's malts for increase of colour-depth.

*Three hours at* 110° *C.*—The colour-depth of 43 units found here is beyond the limits of ordinary brewing, and is of more interest to porter malt and caramel manufacturers. A feature of importance is the symmetrical nature of the proportional curves, indicating the gradual change of colour proportions from a yellow orange to an orange yellow.

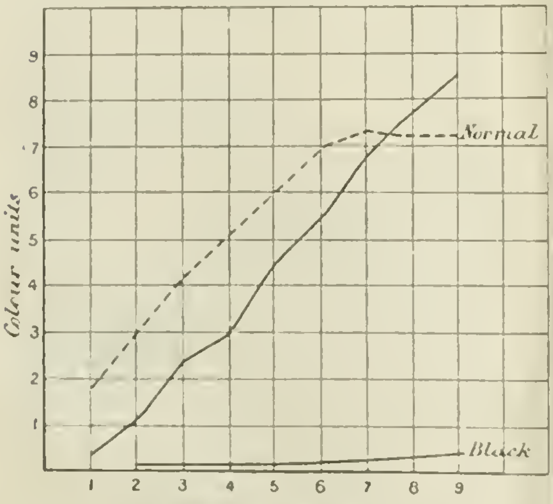
*Four hours at* 110° *C.*—The crossing of the yellow and orange curves occurs here at a colour-depth of 28·4, and the maximum of yellow is reached at 30 units of colour in the 2-inch stratum, where it begins to diminish, disappearing between the 6- and 7-inch stratum, where red takes its place.

*Five hours at* 110° *C.*—In this instance the yellow and orange curves cross at a colour-depth of 39·6, and the maximum of yellow is reached at 48 colour units, when it begins to diminish, disappearing between 7 and 8 inches in favour of red.

### The Black Factor.

This has been found so frequently in unsatisfactory malts and always in uncured malts, as in Series B. (see Chart E.) that it would have been considered a sure index of unsoundness but that it sometimes appears in malts capable of brewing good beer. Two examples have lately come under notice, each containing the black factor. Coke was used to dry the malt in both instances, and it remains for investigation whether the sulphur from the coke, or other fuel containing sulphur, is an element in the production of the black factor. Pending this investigation the black factor may be looked upon with suspicion as possibly representing a point dangerously near imperfect curing.

Chart E.



Independent example of a bad type of malt.

### Discussion.

Mr. E. Grant Hooper said that the author gave no definition of what was a "good malt" or a "good beer," both of which terms very frequently occurred in the paper. He frequently spoke, too, of "unsoundness," but did not explain what he meant by that term. Did he mean what the term implied to most brewers: that the beer developed acidity and want of clearness; or was it merely that it lacked brightness without exhibiting acidity. He could, however, congratulate Mr. Lovibond on having taken up a sounder position on the subject since his last paper. In that he claimed that his process of colour examination really constituted a method of analysis of malts. In the present paper he did not claim that it threw any light on the chemical constitution of malt, but merely that it was a method of arriving at the degree of perfection with which the malt had been prepared. That was a perfectly reasonable position. They could quite understand that colour might represent what had taken place on the malt kiln ; and the announcement of Mr. Lovibood at the end of the paper that, judged according to his standards, a slow but perfect drying at a low temperature yielded better results than a rapid drying at a high temperature, which generally meant destruction of diastase and production of colour, was a conclusion which would meet with very general acceptance. Probably Mr. Lovibond would tell them later what he understood by a good malt. Was it one which converted readily ? If so, it was to be regretted that he had given no standard of converting power with which the colour standards might be compared. Notwithstanding these omissions he felt that the present paper, like all Mr. Lovibond's work, was a distinct advance, however empirical it might appear on the surface ; for if, by a rapid process, brewers could arrive at a conclusion as to whether a malt had been properly dried or not, there might be some advantage over a slower analytical process, for which they would all feel grateful to the author. It was necessary to add, however, that nothing had that evening been said as to the process followed in preparing the malt solutions for the colour determinations. If he remembered correctly the method described by Mr. Lovibond in his previous paper was a brewing process, and this was an important matter where we were concerned with the relative quickness of different processes.

Mr. Alfred C. Chapman thought that the author had so identified himself with this subject, and had so thoroughly absorbed it, that he at times ran the risk of speaking a language foreign to many. It was stated that after seven hours of curing, the "black factor" did not seem to have increased at all. He, for one, failed to understand the meaning of that. It was, of course, well known that one might have two samples of malt from the same barley, possessing the same amount of colour, arrived at in two different ways, by carefully drying up to a comparatively high temperative, on the one hand, or by carelessly drying at a lower temperature on the other. Had the author made any experiments to ascertain whether there was any difference in the nature of the colour curves obtained on examination of malt worts of these different characters? One remark of Mr. Lovibond's he thought was open to severe criticism. He had said that a "good beer" *could not* be brewed with a malt which gave a certain colour curve. What then, did he mean by a bad beer? Was it one which was unsound, or was it one which did not possess a satisfactory flavour ; and if the latter, did he mean one so bad that it could not be consumed. The majority of brewers had at times to deal with unsatisfactory malts, and many of them, from which good beers were brewed, would doubtless show that bad curve to which he had referred. The brewer knew, however, that by altering the conditions of brewing within certain limits he could produce a thoroughly drinkable beer. "Could not" seemed to the speaker to be a stronger term than Mr. Lovibond was justified in using. He would like, in conclusion, to express his appreciation of the interesting paper to which they had just listened.

Mr. Bryan Corcoran, while agreeing as to the very interesting character of the paper as a whole, thought that the author should have given a definition of the word curing. Did he mean the process which did not begin until after the malt was dried? Then the question of the kiln and the time on it were important factors, and the results were affected not only by the temperature attained but also by the volume of the air which was allowed to pass. The hand-dried samples were dried on kiln tiles, and kiln tiles were not, perhaps, the best substance for drying on. Then again, the water-bath would permit of very little air having access to the malt except when the samples were being taken. He thought that if Mr. Lovibond would give definitions of the expressions to which attention had been called he would very materially help members to realise the full value of his results.

Mr. A. R. Ling said that when the tintometer was first introduced by Mr. Lovibond, its use was, he believed, restricted to the determination of the depth of colour of a substance on a purely arbitrary scale. Recently, however, Mr. Lovibond had developed a method by means of which he claimed that the components of a particular colour could be determined in terms of certain "colour units" based on coloured glass. In order to make the coloured glasses exact standards, their absorption spectra should be determined. At all events, he thought, something should be stated as to the way in which these glasses were coloured.

Mr. J. W. Lovibond, in reply, said that Mr. Hooper had asked him to define a good malt. A good malt was one which produced a beer that went bright quickly without artificial aid, was perfectly sound, and generated in itself sufficient carbonic acid gas to keep it good until it was consumed, be the time of consumption long or short. He feared that even Mr. Hooper himself would find it impossible to define the chemical composition of such a malt. Mr. Hooper had also made it a matter of courteous complaint that he had not given a series of analyses with his observations. He could only reply by asking Mr. Hooper to place his laboratory and staff at his disposal for that purpose. The work he already had in hand absorbed 16 hours a day and he could not go beyond that. The assistance of gentlemen like Mr. Hooper, who had a knowledge of these matters far beyond his own, would be invaluable. The question of the proportion of the extract had been raised on a former occasion. It was then asked whether a cold extract was not as applicable as a hot extract. He had made experiments and had found that it was so in most cases, though in a few instances there was a very material difference, and therefore to keep his own series of results uniform he felt that it would be best not to depart from the method he had laid down in his former paper. With regard to Mr. Ling's question as to the colour value of the units, this was really so rudimentary a phase of the question that it could hardly be dealt with then, and he would refer Mr. Ling to his earlier papers on that subject.

Mr. Ling, interposing, said that his purpose was only to suggest that in performing the analyses it was desirable to always use glasses coloured by the same means. Even then he failed to see how anything more than arbitrary values could be obtained.

Mr. Lovibond, continuing, said that was done. If one took the spectrum analyses of his glasses by the light by which they were standardised they would be found to be in exact accord. But if an intense direct light, such as that from an electric arc, was used, they would be found not to accord. Mr. Chapman had asked him to define the black factor. He could not do so. It was always present in uncured malt, and its effect was to make the colour of the extracts dingy. A malt equal to 10 units of red and 10 of yellow was a pure combined colour, but if dulled with one unit of black then the colour was dingy to that extent. Hence he considered that for a malt to be thoroughly sound the black factor ought not to appear; though it might happen that a malt might contain a little of the black factor, and yet be capable of yielding good beer. Mr. Corcoran had asked him to define curing. He had done so in the paper. It was the process after the drying had taken place which demonstrated that the simple drying of the malt made no alteration in the colour curves, but on a small increase of temperature with suitable limits of time one got characteristic colour variations which formed the distinctive difference between the curing of good and bad malt, apart from questions of chemical analysis. If the malt were unsound in the beginning the beer would be bad from other causes, which had not yet been subject to colorimetric investigation. Mr. Corcoran had also referred to the influence of air. He did not know whether Mr. Corcoran had experimented in that direction, and for his own part, he was not prepared to say whether air was or was not necessary. He could only point to the results in the cases of malts cured without a current of air, and that they produced a colour proportion curve higher than the average normal of last season's malts. He had already answered Mr. Chapman's questions with reference to the black factor and would only add that after 72 hours curing, at 110° C. the colour did not increase, nay, it decreased to destruction if the curing were carried on long enough.

Dr. Morris: May not that be due to the presence of an insoluble body? An insoluble body would produce the black factor by simple interference of light, but in these cases it was caused by a substance in solution as the liquids were filtered bright.

Mr. Lovibond, continuing, said he wished Dr. Morris would investigate that point. In his experience the black factor had always been destroyed by perfect curing, so that although it might be present in malt capable of producing good beer it should always be looked upon with suspicion.

The Chairman suggested that if Mr. Lovibond proposed to produce any further paper on the subject, he should put himself in communication with scientists in time to benefit by their collaboration.

## Newcastle Section.

*Meeting held at the Durham College of Science, Newcastle-on-Tyne, on Thursday, March 17th, 1898.*

PROF. P. PHILLIPS BEDSON IN THE CHAIR.

### ON THE SEPARATION AND ESTIMATION OF ARSENIC.

BY J. PATTINSON, F.I.C., AND H. S. PATTINSON, PH.D., F.I.C.

It is well known that in order to obtain perfect precipitation by sulphuretted hydrogen of the metals that form sulphides insoluble in hydrochloric acid, the degree of acidity of the solution containing the metals must be carefully regulated.

Chemists have taken advantage of the greater stability of one or other of the sulphides in hydrochloric acid to effect the separation of these metals from each other, as, for instance, Field's process for separating mercury from antimony by digesting the mixed sulphides of these metals with moderately strong hydrochloric acid, whereby the sulphide of antimony is decomposed and the antimony passes into solution, whilst the sulphide of mercury is left unattacked (J. Chem. Soc. **12**, 132); also Loviton's method for separating antimony from tin by saturating a solution of their chlorides with sulphuretted hydrogen in the presence of an amount of hydrochloric acid which will allow of all the antimony being precipitated as sulphide, but which suffices to prevent the formation of the tin sulphides (J. Pharm. Chim. **17** 361; this Journal, 1888, 403). Rose, too, in his "Handbuch der Analytischen Chemie" (6th ed., vol. 2, 423), states that arsenic and antimony may be separated by gently warming the precipitates of the mixed sulphides with concentrated hydrochloric acid, the antimony passing into solution whilst the sulphide of arsenic remains; and, more recently, Koehler (Archiv. d. Pharm. **227**, 406; this Journal, 1890, 768) has asserted that, in a hot moderately concentrated hydrochloric acid solution of antimonious and arsenious chlorides, the arsenic may be precipitated by saturating the liquid with sulphuretted hydrogen, the antimony remaining in solution.

The method of separating arsenic which we are about to describe depends upon the practically complete insolubility of arsenious sulphide in cold concentrated hydrochloric acid which is saturated with sulphuretted hydrogen. Although there is little in the method that is novel, we think it is worth while calling attention to it, because in many cases it is a really useful and convenient means of separation which, so far, has not been sufficiently taken advantage of in analytical chemistry.

If arsenic is contained in solution as arsenious chloride in concentrated aqueous hydrochloric acid of specific gravity 1·16—1·17, it may be practically completely precipitated as arsenious sulphide by saturating the liquid in the cold with hydrogen sulphide, and if a sufficient quantity of hydrochloric acid be present, arsenic may in this way be easily and perfectly separated from lead, cadmium, tin, antimony, and bismuth, since the sulphides of these metals do not form in the presence of a sufficient excess of hydrochloric acid of 1·16 sp. gr. Arsenic cannot, however, be separated in this way from silver and copper, unless these metals are present in very small quantity, nor from mercury, platinum, and gold, as the sulphides of these metals are very stable in strong hydrochloric acid.

The practically complete insolubility of arsenious sulphide was proved by the following experiments. Three portions each of 0·01 grm. of arsenious acid were weighed and dissolved in a few drops of strong sodium hydrate solution; to each of the portions 200 c.c. of hydrochloric acid, specific gravity 1·17, were added, and the solutions were then saturated with sulphuretted hydrogen. The arsenious sulphide rapidly separated in flocks, and the precipitates were filtered on asbestos in Gooch crucibles. To each filtrate 400 c.c. of water were added, and they were again saturated with sulphuretted hydrogen, but in none of them was there any further precipitate formed, nor even a faint yellow coloration of the liquid, as is the case when a minute trace of arsenious acid is present. After standing some hours, a milkiness due to precipitated sulphur only was formed. The three precipitates of arsenious sulphide were then washed by the following liquids:—

(A.) One precipitate with 200 c.c. of hydrochloric acid, specific gravity 1·17, saturated with hydrogen sulphide.

(B.) Another precipitate with 200 c.c. of hydrochloric acid only.

(C.) Another precipitate with a mixture of 180 c.c. of hydrochloric acid and 20 c.c. of water; specific gravity of the mixture, 1·1526.

After washing, 400 c.c. of water were added to each of the washings, and they were then saturated with sulphuretted hydrogen. In (A) no precipitate or coloration was formed; but in (B) and (C), where no sulphuretted hydrogen had been present in the acid used for washing, a faint coloration was observed, and after allowing to stand overnight and collecting on filter paper the small precipitates, chiefly of sulphur, which had settled on the bottoms of the beakers, and testing these small precipitates for arsenic, a minute amount was found to be present. Further experience has shown that a mixture of 5 parts of hydrochloric acid and 2 parts of sulphuretted hydrogen water may be used for washing precipitates of arsenious sulphide, and that this mixture suffices to prevent the precipitation of antimony when present, without dissolving any of the arsenious sulphide.

We find that sulphuretted hydrogen forms a very distinct precipitate in a solution of 1 part of arsenic (existing as arsenious acid) in 100,000 parts of hydrochloric acid of specific gravity 1·170.

We will now give a few instances of how these facts may be made use of to separate arsenic.

Supposing that in the course of an analysis we have obtained a small mixed precipitate of the sulphides of arsenic and antimony, or of these two metals and of tin we can dissolve this precipitate in as small a quantity of yellow potassium or ammonium sulphide as possible, and then pour this solution of the metallic sulphides slowly into a sufficient quantity of hydrochloric acid to keep the antimony sulphide from precipitating, the acid being constantly agitated until all the solution has been poured in. The volume of acid of sp. gr. 1·16 should be at least three times the volume of the alkaline solution of the sulphides. The sulphides of tin and antimony, which are precipitated at the moment the solution meets the strong acid, are almost instantaneously decomposed, whilst the arsenious sulphide is precipitated.

We must here draw attention to an important precaution that must be taken if a perfect separation of the arsenic be desired. Solutions of the sulphantimonites and sulpharsenites of the alkalis absorb oxygen very rapidly from the air, and thereby become converted into the corresponding sulphantimonates and sulpharsenates. In solutions of the alkaline sulpharsenites there is therefore always more or less sulpharsenate present, the quantity depending upon the length of time that the solution is exposed to the oxidising action of the air. The arsenic existing in this more highly oxidised state is not precipitated at once when the solution is acidified, as is the case with the arsenic which exists as sulpharsenite. To effect the complete precipitation of any arsenic existing as sulpharsenate, the alkaline solution of the precipitate of arsenious and antimonious sulphide should be freshly prepared, and exposed as little as possible to the air; and after the solution of the mixed sulphides in alkali has been acidified in the way described above, the acid liquid should be warmed to about 45° C., saturated with sulphuretted hydrogen by passing this gas through it, or by adding a little zinc sulphide, and allowed to cool before filtering. In this way the whole of the arsenic can be precipitated free from antimony and tin.

The precipitate may then be filtered on asbestos or even on filter paper (some of Schleicher and Schüll's soft papers will stand an acid solution of 1 part of water to $2\frac{1}{2}$ parts of HCl, sp. gr. 1·16), and the antimony and tin may be afterwards precipitated in the filtrate by dilution with water, preferably preceded, when tin is present, by neutralisation of the excess of acid by an alkali. This method of separating arsenic is very handy when dealing with small quantities of the mixed sulphides.

In iron and manganese ores, arsenic may sometimes be readily separated as follows: 3 grms. of the ore are dissolved in 30 c.c. of concentrated hydrochloric acid (a little potassium chlorate or permanganate being added in the case of iron ores) by gentle heating, 12 c.c. of water added, and the liquid filtered on a small filter paper from the insoluble residue, which is washed free from iron, &c., with 30 c.c. of hydrochloric acid mixed with 12 c.c. of water. A further volume of 30 c.c. of hydrochloric acid is then added to the filtrate, and then a strong solution of stannous chloride (made by dissolving 50 grms. of pure granulated tin in hydrochloric acid, and afterwards adding hydrochloric acid until the total volume is 200 c.c.) is gradually poured into it until the iron is reduced to the ferrous state, which is readily seen by the change in colour of the solution, and after this a few drops in excess are added. By this means the arsenic acid is, of course, reduced to arsenious acid, in which state we wish to have it. On saturating the liquid with sulphuretted hydrogen, the arsenic now precipitates free from any tin and from all the metals usually present in these ores, such as lead and antimony, and even from copper, if it be present in only very small quantity, as is usually the case. The precipitate of arsenious sulphide, after vigorous stirring with a glass rod, generally collects into flocks, which allow of easy filtration on an asbestos filter with the aid of the filter pump, and is washed first with a mixture of 5 parts of hydrochloric acid and 2 parts of sulphuretted hydrogen water, and finally with water alone. Occasionally the precipitate assumes a very finely divided condition, which tends to pass through the filter. When this is the case, it may be filtered by stirring up a little ignited asbestos in the liquid before filtering.

One other very useful application of this principle in separating arsenic, we may also give, *viz.*, the separation of arsenic from commercial antimony. Three grammes of the finely pounded antimony are brought into solution in 30 c.c. of hydrochloric acid by gradual additions of potassium chlorate, and without the aid of heat; by constantly rotating the liquid, this may be done in a few minutes. 100 c.c. of hydrochloric acid, sp. gr. 1·16, are now added, and then strong stannous chloride solution (made as described above) until the yellow colour of the antimonic chloride gives place to a colourless solution of antimonious chloride, after which a little stannous chloride in excess is added. Sulphuretted hydrogen is now passed into the liquid to saturation, or the liquid may be shaken up with a little pure zinc sulphide, whereupon the arsenic at once separates as arsenious sulphide, quite free from antimony, tin, or lead, and from the traces of other metals found usually in commercial antimony, and in a flocculent state very suitable for filtration on asbestos with the aid of the filter pump.

The next part of this subject with which we have to deal is the quantitative estimation of arsenic.

Large amounts of arsenic are no doubt best estimated by gravimetric methods. But it frequently happens that the most convenient mode of estimating the small quantity of arsenic that is found in iron ores, manganese ores, pyrites, and in some alloys is to first distil over the arsenic with strong hydrochloric acid by some modification of Fischer's process, and then either to neutralise the acid distillate and titrate the arsenious acid contained in it with a standard solution of iodine, or to precipitate the arsenic as sulphide from the acid distillate. This precipitate may then be purified from free sulphur and weighed; or it may be oxidised and the arsenic precipitated by magnesia mixture and weighed as pyro-arsenate of magnesia; or the precipitate of sulphide may be boiled with a considerable quantity of water until it is all converted into arsenious acid, and titrated with iodine according to the method described by F. Platten in this Journal, 1894, 324.

We find that the arsenic in such precipitates of arsenious sulphide may be very satisfactorily estimated as follows:—

The precipitate is collected on asbestos in a Gooch crucible and washed with cold water until free from hydrochloric acid. The asbestos felt with the adhering precipitate is then placed in a small beaker; the crucible is wiped out with a little clean ignited asbestos, which is also put into the beaker. 10 or 15 c.c. of concentrated sulphuric acid (specific gravity about 1·83), free from arsenic, are then poured into the beaker, which is then placed without a cover on a hot plate or on a wire gauze over a small Bunsen flame in a good draught closet. As soon as the acid reaches the temperature at which it begins to fume, the arsenious sulphide becomes rapidly decomposed; at first both sulphuretted hydrogen and sulphurous acid are given off (if a cover be put on the beaker the mutual decomposition of these two gases causes a deposition of sulphur on the sides of the beaker). The solution of sulphuretted hydrogen is over in a few seconds, but the sulphur dioxide takes longer to expel, depending upon the quantity of arsenious sulphide, and upon the quantity of free sulphur that may have been mixed with the precipitate. As soon as the decomposition of the arsenious sulphide begins, the liquid becomes darkened in colour, and the heating, which may be brought to and kept at the verge of ebullition of the acid, is continued until this dark colour passes away, when it will be found that all the sulphurous acid has been expelled. It is of the utmost importance that all sulphurous acid should be eliminated at this stage. This takes about 10 to 20 minutes with precipitates of sulphide weighing about 0·02 grm. and nearly free from free sulphur. Arsenious acid remains in solution in the sulphuric acid, and the amount is determined by cooling the acid, diluting with water, nearly neutralising the acid with concentrated sodium hydrate solution, and then completing the neutralisation and rendering alkaline with an excess of sodium bicarbonate, and finally titrating with standard iodine solution, using starch solution as indicator. The iodine solution must be standardised against an approximately equal quantity of arsenious acid, to which the same amount of sulphuric acid has been added as was used for the decomposition of the arsenious sulphide precipitate. As sulphuric acid alone usually requires a few tenths of a cubic centimetre of centinormal iodine solution to be added to it before the blue colour of iodide of starch forms, a blank experiment with the stock of acid in use should be made once for all, and the amount of iodine solution required must be deducted both from the quantity of iodine solution required by the solution of the decomposed arsenious sulphide precipitate, and from the amount required by the arsenious acid solution against which the iodine solution has been standardised. We find it the best plan to avoid breaking up the asbestos felt, and if possible to put it in the beaker so that the side on which the precipitate lies is on the bottom of the beaker. This prevents the precipitate from becoming detached from the felt and floating to the top of the acid or creeping up the side of the beaker.

We have used this method for the last six months in the course of our daily work, alongside of determinations made by weighing the sulphide precipitate, or, after having separated the arsenic by Fischer's distillation process, by titrating the distillate (previously rendered alkaline) with iodine solution, and we find that the results are very concordant.

The resultant of the various decompositions that take place when arsenious sulphide is decomposed in the manner described by strong sulphuric acid is probably expressed by the equation—

$$As_2S_3 + 9H_2SO_4 = As_2O_3 + 12SO_2 + 9H_2O.$$

The following experiments show that there is no loss of arsenious acid by volatilisation when arsenious sulphide is decomposed by heating with strong sulphuric acid in the manner described.

Two equal portions of a solution of arsenious acid, each containing 0·01 grm. of arsenious acid, were acidified with hydrochloric acid and saturated with sulphuretted hydrogen. The precipitates of arsenious sulphide were filtered on asbestos, washed free from hydrochloric acid, and decomposed by 15 c.c. of sulphuric acid as described. Another equal portion of the same solution was taken as a standard, and to it was added 15 c.c. of sulphuric acid, without previous precipitation of the arsenic as sulphide. All three solutions were then nearly neutralised with caustic soda solution, the neutralisation completed by sodium bicarbonate, which was added in excess, a little starch solution added, and iodine solution was then run in until a permanent blue colour was produced.

The quantities of iodine solution required were:—

| $As_2O_3$ taken. | Standard. | Decomposed $As_2S_3$ Solutions. |
|---|---|---|
| 0·01 grm. | 26·7 c.c. | (1.) 26·7 c.c. (2.) 26·9 c.c. |

Three portions of another solution, each containing only 0·01 grm. of arsenious acid, were treated in exactly the same way, and required the following quantities of iodine solution:—

| $As_2O_3$ taken. | Standard. | Decomposed $As_2S_3$ Solutions. |
|---|---|---|
| 0·001 grm. | 2·65 c.c. | (1.) 2·65 c.c. (2.) 2·65 c.c. |

The behaviour of arsenious acid with strong sulphuric acid has been occupying our attention for some time, but we need not enter into that subject at present, except to say that we have ascertained that sulphuric acid free from chlorides but containing arsenious acid may be distilled so that the distillate is quite free from arsenic, not a trace of arsenious acid passing over with the distilled acid.

As bearing upon the question of danger of loss of arsenious acid when arsenious sulphide is heated with sulphuric acid, we may, however, quote the following experiment, which shows that there is absolutely no danger of such loss.

Two portions of arsenious acid, each weighing 0·10 grm., were placed in beakers of 150 c.c. capacity and dissolved in 10 c.c. of sulphuric acid, sp. gr. 1·83. A cover was placed on one beaker, and the other was left uncovered, and they were then both heated to just below the boiling point of the acid for three hours, after which the temperature was raised, and the acid was boiled gently for 30 minutes. After cooling, the acid was diluted with water, and neutralised with sodium hydrate solution. The acid in the covered beaker required 39 c.c. of the sodium hydrate solution to neutralise it, and the acid in the uncovered beaker only required 21 c.c., so that about one-half of the sulphuric acid in the uncovered beaker had been evaporated. Another portion of 0·10 grm. of arsenious acid was now weighed into a third beaker, and 10 c.c. of strong sulphuric acid was added to it, and it was warmed gently until all the arsenious acid dissolved, after which the acid was nearly neutralised by sodium hydrate solution. All three portions were now rendered suitably alkaline with sodium bicarbonate, and titrated with an iodine solution. All the portions required exactly the same quantity of iodine solution, *viz.*, 41·0 c.c., showing that there had been no loss of arsenious acid during the prolonged heating and boiling with sulphuric acid.

## ON THE ESTIMATION OF TIN.

BY J. PATTINSON, F.I.C., AND H. S. PATTINSON, PH.D., F.I.C.

The separation and estimation of small quantities of tin in metallic alloys is a problem which generally presents considerable difficulty. We have recently had occasion to determine a small quantity of tin in commercial antimony, and the method we now describe refers more especially to the separation of a small quantity of tin from a large quantity of antimony. The two methods usually employed for effecting this separation are: 1st. The method of fusion of the oxides of the mixed metals with caustic soda, by which antimoniate of soda insoluble in alcohol is formed, the stannate of soda formed at the same time being soluble in alcohol and removed from the antimoniate of soda by filtration and washing. Everyone who has had experience of fusion methods with caustic alkalis knows how troublesome and tedious they are. The second method in general use is Tookey's (J. Chem. Soc. **15**, 462), by which the antimony is precipitated from hydrochloric acid solution by metallic iron, the tin remaining in solution. J. Clark (this Journal, 1896, 256) has shown that the presence of phosphorus in the iron causes the precipitation of a little of the tin as phosphide of tin, which thus escapes determination. Our experience of Tookey's method is that the results obtained by it are not uniform, although the method seems to be an extremely simple one.

We have, however, obtained very good results by the following method, which depends upon the great insolubility of antimony sulphate in strong sulphuric acid, sp. gr. 1·83, whilst stannic sulphate and arsenious acid are readily soluble. Gmelin (Handbook of Chemistry, Eng. ed., vol. 1, page 361) says, "when antimony is heated with oil of vitriol, the products are sulphurous acid, sublimed sulphur, and a white anhydrous residue." This white anhydrous residue is, however, only formed when there is not sufficient sulphuric acid present, for on adding more acid and continuing to boil the solution, the white substance completely dissolves. On the same page, but further on, under the heading "Ter sulphate," Gmelin, quoting Brandes, says that the tersulphate "crystallises in small needles from a solution of the white compound in excess of sulphuric acid."*

By adding a sufficient quantity of sulphuric acid to begin with and boiling, the antimony readily passes into solution without any separation of the white substance above referred to. On allowing such a solution to cool, nearly the whole of the antimony separates out as a sulphate in a mass of white crystalline needles, whilst if tin and arsenic be present, these two metals remain entirely in solution. These facts enable us to treat a large quantity of the antimony so that we have the means of determining tin even when it is present in a very small proportion.

The details are as follows:—100 grains (6·480 grms.) of finely pounded metallic antimony are placed in a beaker of about 300 c.c. capacity, 2½ oz. (75 c.c.) of sulphuric acid (sp. gr. 1·83) are poured in, and the acid is boiled over a Bunsen flame. A cover is put on the beaker to avoid loss by spraying, but care must be taken not to allow the drop of aqueous acid which sometimes condenses on the cover from falling back into the boiling acid, as the sudden generation of steam thereby caused throws some of the liquid on to the cover. By heating up the acid quickly, the antimony can generally be all brought into solution before a drop large enough to fall has collected. After all the antimony has dissolved, which requires about 10 minutes, the gas is turned out and the contents of the beaker are allowed to cool completely. Very shortly after the flame has been turned out, needle-shaped crystals of sulphate of antimony begin to form in the liquid, and soon nearly the whole of the antimony has separated out in this way. When quite cold, the mass of crystals is stirred up gently with a glass rod and transferred to an asbestos filter, and the acid withdrawn by the aid of the filter-pump. The beaker and the crystals are then washed four times with cold sulphuric acid, sp. gr. 1·83. Half an ounce of acid (15 c.c.) is used for this, 1 dram (about 4 c.c.) of acid being used for each washing. After each washing, the crystals are sucked as dry as possible by the pump. The best form of filter is made by placing a glass marble, about half an inch in diameter, in an ordinary funnel, 2½ in. in diameter, and filling in the annular space round the marble by pouring on some ignited asbestos suspended in water. A very thin layer of asbestos—not sufficient to cover the marble—is needed. After all the crystals of antimony sulphate have been transferred to the funnel, the mass should be pressed down with a flat-headed glass rod, and the acid used for washing should be poured on quickly each time so as to completely cover the surface of the mass in the funnel. If this is not done, the washing acid will be drawn through the crystals in channels and will not wash them properly. The filtrate now consists of between two and three ounces of strong sulphuric acid, containing in solution all the tin, arsenic, copper, iron, and other impurities in the antimony (except perhaps some of the lead), and only about half a grain (0·03 grm.) or less of antimony. The filtrate is transferred to a large beaker, or preferably a flask of about two litres capacity, and diluted with about three times its volume of water; it is then best to cool it, after which nearly the whole of the acid is neutralised with ammonia (probably potassium hydrate would be preferable, but it should be free from silica and alumina). When nearly neutral, but still decidedly acid, enough strongly yellow ammonium sulphide is added to completely keep in solution the sulphides of tin, arsenic, and antimony. The sulphides of all the other metals present are thrown down in a flocculent form, and are separated by filtering upon a loose-textured paper, and washed with water containing a little ammonium sulphide until the filter-paper is free from antimony. The filtrate now only contains antimonious and arsenious sulphides and stannic sulphide. It is to be noted that tin, when dissolved in an excess of strong sulphuric acid, goes into solution as stannic sulphate and not stannous sulphate, the stannous sulphate first formed being oxidised at the expense of the sulphuric acid.

The mixed sulphides of tin, arsenic, and the small quantity of antimony are then thrown down from the filtrate, by rendering the solution very slightly but distinctly acid with dilute sulphuric acid (acid of the strength of 1 vol. of acid, sp. gr. 1·83, to 3 vols. of water may be used). The precipitate should be allowed to settle, and is then filtered. The filtrate can be set aside to see if any further precipitate forms, but we have never found this to be the case.

The precipitate of the sulphides is then washed off the paper into a beaker with as little water as possible, and a solution of bromine in strong hydrochloric acid poured over the paper and allowed to run through upon the precipitate in the beaker, and the paper is then washed with hot water. On boiling, the arsenic, antimony, and tin quickly pass into solution, leaving a mass of sulphur. It is now best to decant the clear liquid from the mass of sulphur, which is allowed to remain in the beaker. On pouring a little fuming nitric acid upon it, the sulphur is easily dissolved, and this solution is then united with the main solution of the metals. The greater part of the bromine is now boiled off, and, after cooling, the excess of acid is nearly neutralised with ammonia. The bulk of liquid at the end of this operation is about 150 c.c.

The tin is now separated from the antimony and arsenic by F. W. Clarke's process (Chem. News, **21**, 124), *viz.*, by adding oxalic acid to saturation (about 15 grms. is needed) and precipitating by sulphuretted hydrogen the sulphides of antimony and arsenic in the hot solution. The tin, which is not precipitated, remains in the filtrate, which is first nearly neutralised with ammonia, then yellow ammonium sulphide added until the precipitate of stannic sulphide that forms redissolves, and finally the tin is thrown down by rendering the solution acid with acetic acid. The precipitate collects together after standing a few hours, and is easily filtered, washed, and ignited, and the tin weighed as stannic oxide, $SnO_2$.

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* Our analysis of these crystals shows that the compound is not the tersulphate, but that its composition approximates very closely to that of antimony pentasulphate, $Sb_2(SO_4)_5$.

Three portions, each of 100 grains, of a sample commercial antimony examined in this way, and another portion, also of 100 grains, to which 0·2 grain of pure tin (=0·254 grain of $SnO_2$) was added, also treated in the same way, gave the following results, which show that all the tin added had been separated and determined :—

| Taken. | Grains of $SnO_2$ found. | Equal to Metallic Tin found. |
|---|---|---|
| 100 grains of sample....... | 0·235 | 0·185 |
| " " ....... | 0·230 | 0·181 |
| " " ....... | 0·270 | 0·212 |
| " " + 0·2 gr. tin = 0·254 gr. $SnO_2$ | 0·505−0·254=0·251 | 0·397−0·2=0·197 |

# New York Section.

*Meeting held at the College of Pharmacy on Friday, February 25th, 1898.*

MR. THOS. J. PARKER IN THE CHAIR.

## NEW METHOD FOR THE DETERMINATION OF TARTARIC ACID.

BY JOHN MOSZCZENSKI.

The method used almost exclusively at present for the determination of tartaric acid is Goldenberg's so-called acid method. This method consists in decomposing the materials containing tartaric acid, especially argols or wine-lees, with hydrochloric acid, boiling the filtrate with excess of $K_2CO_3$, whereby lime is precipitated and all acids combined with potassium, finally adding to the second filtrate acetic acid, thus converting neutral potassium tartrate into bitartrate, almost insoluble in strong alcohol, with which it is precipitated and washed. The amount of pure cream of tartar contained in this precipitate is found by titration with caustic alkali. Every molecule of cream of tartar found corresponds to 1 mol. of tartaric acid present in the substance tested.

It is clear that the cream of tartar, into which tartaric acid has been converted by the above method, will be contaminated with all those substances present in the original sample which are soluble in dilute HCl, which from such solution are not precipitated in presence of tartaric acid by boiling with $K_2CO_3$ and are not removed by washing with strong alcohol. Besides iron and alumina, the most important compounds belonging to this class are the so-called pectinic matters, which in argols and lees are found frequently in large quantities, and which probably interfere greatly with the results of the titration, especially if they are previously boiled with $K_2CO_3$, as is the case in the method described.

Those pectinic matters, which are insoluble in alcohol, are kept out of the cream precipitate in the following method, which, besides, has the advantage of being shorter than those used at present.

Materials containing tartaric acid are treated with dilute $H_2SO_4$ and then with alcohol, which dissolves all tartaric acid and free $H_2SO_4$ present. Part of this alcoholic solution is filtered off and alcoholic potassium acetate added to it, which precipitates all $H_2SO_4$ as $K_2SO_4$ and tartaric acid as cream of tartar. This precipitate is filtered off, washed with strong alcohol, and titrated. The substance to be tested must of course be ground very finely and treated with sufficient quantity of $H_2SO_4$ (10—15 per cent.), avoiding at the same time unnecessary excess, not to increase beyond need the bulk of the ultimate cream and potassium sulphate precipitate. This amount of $H_2SO_4$ varies with the nature of tartaric acid compounds to be tested. So 1 mol. of potassium bitartrate needs 1·7 mol. of $H_2SO_4$ (10 per cent.) for its complete decomposition into tartaric acid and $K_2SO_4$; calcium tartrate requires less $H_2SO_4$. With ordinary materials it is safe to use 26 c.c. of 13 per cent. $H_2SO_4$ for 5 grms. of argols, lees, or cream.

It is not necessary to let $H_2SO_4$ act long on the material to be tested; with careful stirring a few minutes' action is sufficient, whereupon the mixture is transferred into a flask, filled up to a certain volume with strong alcohol (not under 90°, preferably stronger), and part of it, after good shaking, filtered off immediately through a ribbed filter. I found it convenient to use 5 grms. of substance, fill up to 250 c.c., and filter off 200 c.c.; but those proportions can of course be changed without affecting the results, using necessary judgment and making corresponding changes in the corrections mentioned below. It is very important, however, not to let the alcoholic solution containing free sulphuric and tartaric acid stand too long, as this would make the results too low, probably because of formation of ethylated tartaric acid, which on addition of potassium acetate would not be transformed into cream of tartar.

The alcoholic solution is transferred to a porcelain dish, sufficient quantity of alcoholic potassium acetate added, and well stirred. To reduce the solubility of the potassium bitartrate thus formed in alcohol, add 5 c.c. of concentrated KCl solution. Working with argols or lees, it is easy to find the point when enough potassium acetate has been added by observing the change of colour from pure red into pale bluish-red which then takes place. After the precipitate has settled, pour off a little of the supernatant liquid in a test tube and add a few drops of potassium acetate solution, which should not form any further precipitate. Having thus ascertained that the amount of potassium acetate used is sufficient, the precipitate is left to stand for at least six hours, washed with strong alcohol, and titrated.

If in filling up to a certain volume with alcohol the volume of solid matters has not been taken in account, a corresponding correction has to be made on the results. In testing lees or argols, the volume correction has, in most cases, been found to be about 1·2 c.c. for 5 grms. of substance.

Another correction has to be made to cover the losses by solubility of cream of tartar in alcohol. Using 5 grms. of substance, filling up to 250 c.c. with 90 per cent. alcohol, and filtering off 200 c.c., those losses amount to 0·320 of tartaric acid, which should be added to the result. The results obtained with this method are in most cases a few tenths of a per cent. lower than those found by Goldenberg's acid method. Which are nearer correct is very hard to decide, as unfortunately it is as yet quite impossible to produce, artificially, mixtures which would correspond in their composition to natural argols, lees, or other impure materials containing tartaric acid, and only on such mixtures could those methods be satisfactorily tested.

In both of those methods the results are considerably interfered with by a good many different substances; but then the same could be said of almost every analytical method.

A mixture containing 50 per cent. of cream of tartar and 50 per cent. of potassium oxalate gave, on testing by the new method, 55 per cent. of cream of tartar.

Another, containing 75 per cent. of cream and 25 per cent. of potassium oxalate, gave 77·25 per cent. of cream; the same with Goldenberg's method, 79·72 per cent. of cream.

The presence of $P_2O_5$ also makes the results too high; and here, as in the previous case, the results of the new method seem to be less affected. Moreover, from the few experiments which I made on this subject, so far it would seem that $P_2O_5$ has a rather quantitative influence on the results of the new method, and increases them by about 1 mol. of tartaric acid for every molecule of $P_2O_5$ present in the mixture

If further experiments should confirm this, it would be possible to correct the error due to $P_2O_5$ in testing materials containing tartaric acid and $P_2O_5$. This would be quite important for testing so-called "old acid liquors"—mother-liquor from tartaric acid manufacturing—as these contain, besides other impurities, considerable amounts of $P_2O_5$.

For testing those liquors and, in general, tartaric acid mother-liquors, the new method is especially convenient, as in this special case it is still simpler than in testing other materials, and as it combines well with the "free sulphuric acid test," which is of importance for those products.

Those liquors containing, as a rule, free sulphuric acid, no addition of such is needed

A certain amount of the liquor is simply filled up with strong alcohol to 500 c.c., well shaken, and filtered immediately; 200 c.c. of the filtrate are tested as above for tartaric acid; 250 c.c. for free sulphuric acid in the usual way.

For comparatively pure mother-liquors no correction is needed; for old acid liquors the volume of the precipitate formed with alcohol may be taken into account; but this changes so much with the methods of manufacturing, that no figures can be given here.

In this way the determination of both tartaric and free sulphuric acid can be made in a short time, and the exactness of the results is very well within the limits of a factory test.

It would seem that, carrying out the determination of tartaric acid as described above, and starting with a known amount of $H_2SO_4$, it would be possible to determine the amount of tartaric acid present as potassium bitartrate according to the equation—

$$2x\ \mathrm{KHT} + y\ \mathrm{CaT} + p\ \mathrm{H_2SO_4} =$$
$$x\ \mathrm{K_2SO_4} + y\ \mathrm{CaSO_4} + (2\ x + y)\ \mathrm{H_2T} + (p - x - y)\ \mathrm{H_2SO_4}.$$

by deducing from the sum of the acidities of tartaric acid and $H_2SO_4$ present in the alcoholic filtrate, the acidity of $H_2SO_4$ added in the beginning, and making the difference equal to the acidity of cream present in the sample; but this method of cream of tartar determination would be longer and more complicated than those used at present. Besides, the large excess of sulphuric acid used in this method, and the, under the circumstances, not quite negligeable solubility of $K_2SO_4$ in alcohol, render the results uncertain.

### Discussion.

The Chairman said that he called to mind a very similar method for the determination of tartaric acid and cream of tartar in mother-liquors, which, as he recollected, was that of the late Dr. Casamajor, and could be used for the quantitative estimation of both potash and tartaric acid and cream of tartar. He would ask Mr. von Moszczenski if he was familiar with the Casamajor method for these determinations. He thought there was a very full account of it in the Journal of the American Chemical Society and perhaps one or two other journals. The manufacture of tartaric acid and cream of tartar formed a very important chemical industry in the United States.

In reply to the Chairman, the author said he was not familiar with the method referred to.

## THE EVOLUTION OF THE SULPHITE DIGESTER.

BY MARTIN L. GRIFFIN.

By sulphite digester, I mean the vessel used for cooking or reducing wood to its ultimate fibre by treatment with solutions of sulphites and bisulphites of the alkaline bases. It is not my purpose this evening to weary you with all the details of construction of the sulphite digester, which have been proposed and patented by inventors and manufacturers since the discovery of this process for treatment of vegetable fibres by Benj. C. Tilghman, of Philadelphia, in 1867. The great mass of such are absolutely of no interest and worthless. I shall endeavour to confine the limits of this paper to the improvement and rational features of the development, without giving exact dates. From Tilghman's patent we learn that "a strong iron vessel of any convenient size and shape, lined with lead, and provided with a steam jacket and with necessary pipes," &c. may be used for this purpose. In this brief description we are introduced to the first sulphite digester. From this point we shall see that the difficulties to be encountered and the development of the digester are both of a chemical and physical nature.

Naturally attention was first directed to materials which would resist the action of the sulphur gases and acids so far as it was possible to decide from experience in other industries. Next, the physical qualifications of such materials. Lead was selected to meet the first of these requirements, but on account of its great weight, its lack of hardness, and tensile strength, it was totally unsuited physically for the purpose. Iron, on the other hand, fulfilled the requirements which lead lacked, but could not resist the action of the acids. Nevertheless, we find, as late as 1880, that Mr. C. F. Cross took out an English patent, No. 4984, for a process of boiling jute, hemp, flax, &c. with sulphites and caustic soda in an iron vessel. The writer has no knowledge that this process was ever put to any considerable use, but ventures to say that such a vessel for cooking with an alkaline solution of sulphites would have quickly failed.

Hence we see in the first digester a combination of these two materials. We have no knowledge of the difficulties which Tilghman encountered with his digester, as he did not make a commercial success of his process. We know what his difficulties would have been had he pursued his work. The unequal expansion and contraction of these two metals, about 1 to $2\frac{1}{2}$, was destined to play a very important part and to present great difficulties to the chemical engineer, and to cost manufacturers hundreds of thousands of dollars in their attempts to yoke them and make them work together.

From this time nothing more of importance relating to this process happened till about the expiration of Tilghman's patent, when almost simultaneously, during the years 1880 to 1885, the process was taken up by Carl D. Ekman and Edw. Partington in England, David Otto Francke in Sweden, Alexander Mitscherlich in Germany, Eugene Ritter and Chas. Kellner in Austria, and also by Chas. S. Wheelwright and Geo. E. Marshall in the United States. From this time there has been great activity in the development of the process and industry.

In general the process of reducing wood to fibre suitable for paper making, consists in subjecting it to a solution of sulphites of the alkaline earth bases in the digester for from ten hours to three days at a steam pressure from 1 to 5 atmospheres. The acid solution contains from 1 to 2 per cent. base, and 3 to 5 per cent. sulphur dioxide according to circumstances. Hence we see what is demanded of the digester.

Ekman's digester, as described in his English patent, July 13, 1881, consisted of a "jacketed boiler lined with lead or a suitable compound thereof, and mounted on trunnions for convenient filling and emptying." The digester was cylindrical in shape, and mounted in either a vertical or horizontal position. You will note that the heat is transmitted to the mass to be cooked by means of a steam jacket. Also that neither Tilghman nor Ekman made any reference to the method employed in lining their boilers with lead. The production of the fibre was their attractive object, and the digester was only a means to this end.

The same year Francke, in Sweden, patented a process for improvements in the manufacture of paper pulp. His digester consisted of a horizontal cylinder revolving upon anti-friction supporting rollers, having a steam jacket on each end connected by lead-covered tubes passing through the interior of the digester, which was lead-lined.

Ekman's digester was 4 ft. in diameter and 12 ft. long, and Francke's was probably comparatively small. In 1883, Alexander Mitscherlich patented a digester and process for reducing wood to pulp radically different from anything hitherto known. The digester, cylindrical in form, was about 12 ft. in diameter and 36 ft. long, stationary, and was supported horizontally upon piers of masonry. It was built as follows:—First the iron shell, $\frac{3}{4}$ in. thick, the interior surface of which was covered with a cement of tar and pitch; next a lining of thin sheet lead with edges burned together. Upon this lead is laid flatwise a course of dense, vitrified bricks, especially made for this purpose, having tongues and grooves edgewise. Upon this is laid another course of bricks edgewise, having their tongues and grooves arranged accordingly. This brickwork was laid in Portland cement.

The cooking was accomplished by indirect steam passing through coils of 1-in. pipe, composed of lead and antimony, lying in loops on the sides of the lower half of the digester.

This was and is to-day the most rational construction of the sulphite digester. From the Gay-Lussac and Glover towers lined, not with lead, but with acid-proof tile or bricks, the transition to the tile- and brick-lined digester was very natural and easy to such an able scientist as Dr. Mitscherlich. So carefully did he work out the details of the lining, size, and shape of his digester, and method of cooking, that though the coefficient of expansion of firebrick is less than half that of iron, the lining has never given any serious trouble, the joints only requiring pointing from time to time. One would have thought that now the lead-lined digester was doomed to a speedy death. Not so; it was destined to struggle on for a dozen years in less scientific hands. We shall observe its course in the progress of this paper.

At this point let us review the probable reasons why the use of lead for lining, and later, bronze digesters, was so vigorously followed up. Lead has long been used in the manufacture of vitriol, though under very different conditions, and hence it was thought suitable for this purpose. Some believed that though the lead lining would give out in time, it could be so used and the digester so constructed as to make the necessary repairs cheaply, and so make its use a commercial success. Still others believed that lead could be so bonded to its iron companion that it would give up its bad habits of expanding extravagantly and then refusing to contract to its former dimensions, and lead a new life. The beautiful pulp produced, when the digester was new, in 15 to 20 hours, and often in less time, with no loss of time in blowing down the pressure, discharging and washing out, and little if any inspection, was a great temptation to its use, when contrasted with the slow process of moving away the discs of wood in the large Mitscherlich chambers, steaming, cooking at low temperature and pressure for nearly a week, blowing down the pressure, and finally washing and shovelling out by hand, &c. Perhaps one of the most potent reasons—in the United States at least—for not adopting the heavy, thick, brick lining and process of Mitscherlich, was because he had protected it by patents and was able to organise a strong company composed of influential men in the Government service during the administration 1884 to 1888. This company owned the exclusive right to operate the process in the United States and Canada. The price of pulp and paper at that time, and the very low cost of producing sulphite fibre, was a sufficient reason for charging a high royalty for the use of the digester and process. This the majority of manufacturers were unwilling to pay, believing that they could circumvent these demands at less expense. Also the slow, tedious European process did not accord with the American idea of progress.

As indicated in the early part of this paper, the unequal expansion between iron and lead was destined to be one of two great obstacles in the use of lead for lining. The expansion of the latter and the failure to contract soon caused the sheet lining to buckle and crack and require constant repairs. This is what happened in the case of Fraucke's and Ekman's boilers.

As soon as this became apparent, a great variety of ways of attaching the sheet lead to the iron were devised.

The second obstacle to the use of lead lining for any length of time was due to the fact that in the process of burning brimstone for making the solution of sulphites, a small amount of sulphides of calcium or magnesium is produced from reducing impurities in the brimstone. Also that there are conditions in the process of cooking favourable to the reduction by the organic matter of a small proportion of sulphites to sulphides of the alkaline base.

The result of this action is that at first there is only a slight blackening of the surface of the lead, which would have no significance, except to the experienced observer. In time the lead will show signs of crystallisation, and on such places we shall a little later observe black scabs, which are composed of sulphites and sulphides of lime and lead. These in turn appear to have a very bad effect, for if we remove these scabs we shall find the lead pitted. The scabs may be scraped off, and the defective places chipped out, and the holes burned up, but the lining is doomed, and in a very short time is worthless. Besides digesters lined with bricks and lead, bronze digesters were used to a considerable extent in the United States.

After this introduction to the three classes of digesters, let us follow the history of each.

The Ekman and Francke digesters were never used in this country, and we pass on to some of their successors.

The Partington digester ran its course from about 1885 to 1890, and was principally used in England and this country. As originally constructed, it was spherical in shape, about 12 ft. in diameter, and mounted on trunnions for ease in filling, emptying, and circulating the cooking acid. The lead lining was applied in spherical lunes, clamped to the iron, and edges burned to each other. The theory was that it would be an easy matter to replace an injured section, and thus to keep the lining intact at comparatively small cost. The shell was perforated in many places to locate leaks in the lining. The result of this clamping was an expansion and buckling between the clamps, and final fissuring of the sheets. The repairs of these digesters were doubtless greater than on any other digester ever built; add to this the time shut down for repairs, we see that the process was very unsatisfactory and costly.

The Ritter-Kellner digester, though a little out of chronological order, should be described in connection with the Partington, as the right to use the two processes in the United States and Canada had been purchased by one company, and the two kinds were at several places installed in the same mill. At first we saw that the production of the fibre was the object sought; now we are fully launched in the struggle to improve the digester linings. The Ritter-Kellner digester was cylindrical, vertical, and stationary, about 10 ft. by 28 ft. high, built up of cylindrical sections 4 ft. wide, a few inches apart, and fastened by heavy exterior bands. The object of this construction was to provide the means for attaching the lead lining peculiar to the process. The spaces between these sections formed annular dovetail mortises. These mortises are filled with an alloy of lead and antimony, and at the ends of a diameter meet similar vertical tenons, to which they are attached. The sheet lining is burned fast to this semi-cylindrical frame. The specific object of this arrangement, as stated by the patentees, was to make the lead lining of the digester durable by providing an arrangement for securing the lining to the casing of the boiler in such a manner that the creeping effect of the soft metal is localised and hence more durable. To further secure this result, hard lead rivets might be used, though I have never seen them so used in this country. The shell was also provided with the leak-locating holes.

Manufacturers are now in the midst of the struggle to bind fast the recalcitrant lead. The second obstacle to its permanent use, previously referred to, has not as yet appeared; when they have fairly accomplished the work of forcibly holding the lead in place, it will appear. We see the advance made by Ritter-Kellner over Partington in the mechanical method of binding the lining. The cooking was accomplished in each case by the direct admission of steam into the mass.

These digesters were certainly more successful than the Partington; but the means, though vigorous and powerful, were as yet inadequate to hold fast the lining, which still buckled and cracked, though over smaller areas. A new phenomenon now appears. The digester being vertical and stationary, the expansion of the lining is downward, in consequence of its weight; the upper parts become thin, and give way. This, of course, could not happen with a revolving digester.

In 1884, Chas. S. Wheelwright, engaged in the manufacture of sulphite pulp and paper in the city of Providence, R.I., patented an improvement in paper-pulp digesters for sulphite, which were built, having double walls, of rolled iron, made in sections, and united section to section and wall to wall by rings. "A suitable metallic lining (preferably of copper) is to be placed on the inside of the inner shell."

The cooking was accomplished by admitting steam into the annular spaces between the outer and inner shell. This construction proved very costly and unsatisfactory, and was followed by the following modification: the shell of the digester was constructed of annular sections of cast iron, having flanges, and these were bolted together. Each section was lined separately with sheet lead, and attached by flanging over the iron flanges. The object of this construction was to facilitate the repair work, as now no one hoped by mechanical means to hold the lead lining in place. Steam for cooking was admitted directly.

Notwithstanding the high price of the pulp produced, the process was not a commercial success, and the company, after a struggle for several years, went out of existence.

I have now detailed some of the early and typical methods for fastening the lead lining and localising the expansive action. A great many other modifications for mechanically attaching the lead followed from year to year even as late as 1891, when it must have been apparent to the most superficial observer that only a waste of time and failure could result. It is needless to perpetuate them further.

There still remains one more chapter on lead lining to be told, and we are done for ever with lead as the principal lining in sulphite digesters. This we will defer while we take up the digester of bronze, which I regret to say was not destined to rank with other works in bronze and become a lasting monument to posterity of the skill and scientific attainments of this age of paper making.

This age of bronze was brief, lasting only a few short years from about 1887 to 1893 or 1894 in this country. It would even now be forgotten except for the death and destruction caused by explosions. It was never seriously taken up, except by one man and one manufacturer of the digester, though they were operated in some half dozen places.

The difficulties of making large homogeneous bronze castings were claimed to be obviated by a secret process employed by the Deoxidised Metal Co., of Bridgeport, Conn., and hence the construction of these digesters fell to them. They were stationary, upright cylinders, 7 ft. in diameter and 22 ft. high, made in sectional castings of this deoxidised bronze, with planed flanges, bolted together, and lead-jointed in assembling. This alloy the designer assumed was sufficiently acid-proof for the purpose without any protecting lining. Steam was admitted directly in cooking.

In a paper read, June 1889, before the American Society of Civil Engineers, by Major O. E. Michaelis, at that time in command of the Kennebec Arsenal, and myself, after an extended tour of investigation of the sulphite processes, he says, in reference to copper-tin alloys, they are "'Kittle cattle'; all authorities agree in testifying to their sensitiveness. Fluctuations in temperature affect their strength, and their constant exhibition under active circumstances in the presence of sulphur compounds cannot fail to produce deleterious effects." The analysis of a piece of one of the deoxidised bronze digesters showed the following composition:—

| | Per Cent. |
|---|---|
| Tin | 7·68 |
| Copper | 91·28 |
| Zinc | 0·89 |

The specific gravity was 8·5579; the computed gravity was 8·76; while that of pure copper is 8·91.

This indicates the difficulty in making perfect castings. From an examination of the castings, it was further evident to us that had they been perfect, it would have made little difference, for neither copper nor any alloy of it is proof against the action of sulphur acids.

The progress of the action of the acid sulphites on the interior of the digester was first to dissolve a film of the surface. This in turn was promptly reduced to the oxide and sulphide by the organic matter, forming a dirty, black scale, which increased in thickness until it became dislodged and contaminated the pulp. It was only a matter of time how long any digester would last, or how long it could be safely operated, as it was a very treacherous thing to inspect.

Finally, after a few explosions, insurance companies would not insure them, and they were quickly abandoned. The manufacturer of the castings had previously abandoned his part of the work as a failure.

The argument employed to introduce the bronze digester was a very plausible one. The promoter declared that though there was some action on the metal, the digester was, nevertheless, as durable as an iron digester for soda pulp. (I suppose he meant the riveted digester then in use.) He also offered to take back the old digester, and pay the market price of lake ingot copper. In this way, he claimed that his digester was the cheapest and most satisfactory of any on the market.

We now come to the last chapter on lead lining. Lining digesters with sheet lead was recognised as a very unsatisfactory method, if not a failure, for the reason that no one had been able to devise means for holding it in place, and keeping it from buckling and cracking. If anyone could be found who could accomplish this, there were those who were still willing to put their money into it.

In 1852, one James Anthony Graham, of London, patented a process for covering iron with any thickness of lead, by a process of cleaning the surface of the iron, heating and covering the same with a flux of zinc chloride, and applying molten lead—in other words, a solder attachment. It seems a little strange that such a patent should have been granted when the manufacture of terne plate had been known so long.

In 1886, one Geo. R. Noble, at that time a resident of England, conceived the idea of constructing digesters with sheet iron covered with lead, according to the Graham process, and was granted a patent therefor.

Manufacturers had been introduced to The American Sulphite Pulp Co., The International Sulphite, Fibre, and Paper Co., &c. But the breadth and extent of the operations of these companies, as indicated by their titles, were not to be compared with those of a company which was organised to operate what was known as the Graham process, and so the title, Globe Sulphite Boiler Co. was selected as most appropriate.

The company expected to build both rotary and stationary digesters. The first rotary revolving on trucks, as Francke's, was 7½ ft. by 22 ft. Later digesters, 8 ft. 4 ins. by 32 ft. 6 ins., were built, the latter producing about 7,000 lb. of pulp to each cook. It was constructed by rolling and assembling the sheets covered with about one-half inch of lead after Graham's patent. The seams were then cleansed, filled in, and burned with the soldered lead coating. The greatest care was taken to obtain the purest Spanish lead and to make the interior surface smooth. At the time I do not know that any importance was attached to the galvanic action due to contact of different metals or the alloying of the flux with the lead. I have reason for believing that it was not even thought of.

Just here are some very interesting points which in this connection were ably presented by my friend, Major Michaelis, in the paper above referred to. I quote from his paper: "Although the promoters of this process do not so call it, it is, nevertheless, soldering, which is authoritatively defined to be 'the process of uniting two pieces of the same or of different metals by the interposition of a metal or alloy, which, by fusion, combines with each.'"

Here the metals to be united are lead and iron; the "metal or alloy" is zinc, or zinc and lead. I emphasize this simple matter because it is claimed that in the Graham process the two metals are united by a special alloy of lead and iron.

I quote from the company's courteous, well-posted expert:—

"Our boiler obviates these defects, and overcomes the difficulties and repairs and constant inspection to which ordinary lead-lined boilers are liable. This is because the lead is united to the iron or steel uniformly by means of an alloy at the point of junction between the steel and lead, and the crystals of the two metals are intimately mixed at that point, so much so that it is impossible with a cold chisel to cut the lead away from the iron or steel without still leaving a thin surface of lead. The junction alloy of steel and lead has also a greater breaking strain than lead itself, it having been subjected to breaking tests."

A standard authority states that "an alloy is a combination by fusion of two or more metals, as brass and zinc, tin and lead, silver and copper, &c." Hence to speak of the "junction alloy of steel and lead" is incorrect, for in the coating operation there is not even an approximation to the melting point of iron.

The alloy of lead and iron appears to be almost unknown in the arts; and that this so-called "junction alloy of steel and lead," or, as I think, of zinc and lead, should possess a greater tensile strength than the lead, is not strange; it is not an unusual property of alloys; thus one consisting of twelve parts lead and one part zinc, hàs six times the tenacity of lead.

I must also put on record my lack of faith in the natural inference that would be drawn from the claim that the two metals (iron and lead) are intimately mixed at this point (the junction or cementation surface), "so much so that it is impossible with a cold chisel to cut away from the iron or steel without still leaving a thin surface of lead." Under favorable circumstances the lead can be "peeled" from the iron—I have done it myself.

I examined the Graham process after the Ritter-Kellner at Cornwall, and it struck me at once as a logical consequence that, if "tacking" the lead on in places only, gave satisfactory results, then complete cementation ought to do even better. Upon reflecting, however, I modified this hasty conclusion, for the governing conditions in the two processes are entirely different. In the Ritter-Kellner, the lead is burned to the circular and vertical tenons.

Burning is defined as "joining metals by melting their adjacent edges, or heating the adjacent edges and running into the intermediate space some molten metal of the same kind." A well-known writer says:—

"The articles burned together being homogeneous, the parts expand and contract evenly by changes in temperature; the solders have a greater range of expansion by given changes of temperature than the metals they connect.

"The solders oxidise more or less freely than the metals they connect, and establish galvanic circuits which destroy the integrity of the joint, especially in the presence of heat, moisture, or acids."

One would almost suppose the author had sulphite digesters in mind.

In the Ritter-Kellner there is little danger of the joint loosening. In the Graham, under periodic expansion and contraction and galvanic action, I should fear a sundering between the lead and iron; if this did take place the boiler would very soon become unserviceable. Even granting the claim that the lining and sheet are indissolubly connected by an alloy of lead and steel, and that the "lead, being the weaker metal, when fastened homogeneously to the iron or steel, has to obey the expansion and contraction of the iron or steel," there still remains an apparently insuperable difficulty. The lead lining is at least half an inch thick, and even supposing that the cylindrical laminæ in homogeneous contact with the iron are docile, and move in military cadence with their inseparable stronger companion, yet the balance of the lead must be subjected to a recurring ebb and flow for which no compensation is provided. I quote from the German expert:—

"The explanation of the iron controlling the lead expansion is easy to understand. Lead is a very malleable metal, its crystals fitting loosely together, *i.e.*, there is comparatively a large space between the crystals as against other metals, and if the crystals of lead were counted over 22 ft. (the length of the digester), and the number divided into one-third of an inch (the linear expansion of lead for 90° in 22 ft.), it would be found that the fractional part of this one-third inch would be considerably less than the space between each crystal, and there is no chance of one crystal pushing another one, as in metals where crystals are closer together."

On this theory I do not understand why lead should expand measurably under ordinary increase of temperature. Further, the common experience of every plumber militates against the correctness of the assumption.

Lead pipes for hot water will "creep" under fluctuation of temperature without perceptible increase of length; this is very evident when the pipe under a metallic clamp is examined: a burnished band is seen protruding beyond the clamp. I have seen this in less than twenty four hours after brand-new pipes were installed.

It is a well-known fact that in hotels, for instance, where a large quantity of hot water is used in the morning and very little during the rest of the day, the pipes being thus subjected to frequent and comparatively great changes of temperature, this very movement of which I have spoken, to use plumber's parlance, "kills" them. That is, they lose all elasticity, become brittle and crack, showing a crystalline fracture. Now, I am inclined to think that there is danger of such a result with the lead lining of the Graham boiler, even if the cementation remains intact. The lead, under this constant, enormous molecular stress, will degrade, become brittle, porous, crack, even drop off.

Then the boiler is ruined, for the lining, under such circumstances, can neither be repaired nor replaced.

I regret to say that Major Michaelis did not live to see the fulfilment of his prophecy, which came true in every particular. The lining of the boiler remained clean, and showed no apparent defects caused by the enormous stress upon it until some 200 or more cooks had been taken off. Then there began to appear little star-shaped defects, showing hard crystals and tiny cracks. These were, of course, cut out and new lead burned in, but soon they multiplied so fast that it was impossible to repair them all. These in turn became black, dirty scabs, and later large masses of them would bulge out at the seams. It was then only a short time until the whole lining became a dirty, crumbling mass, and the digester unsafe. It was impractical to replace this lining, and so the Graham process came to an end.

Upright Graham digesters were placed in one mill with much less satisfactory results.

Reference has been made to the tendency of lead linings in upright digesters to sag. One would hardly have thought it possible for this lining to have stretched to the extent it did. In a very short time it was nearly all in the lower half of the digester, and the top with hardly no protection. Of the many different methods for lining with lead, this looked at first the most promising, but in the end it proved the most disastrous. Of the four companies which put in these digesters, one failed directly from this cause, and the failure of two others must be largely attributed to the same cause, and the Globe Sulphite Boiler Co. wound up its affairs as quietly as possible. With the failure of lead lining, many manufacturers were still looking for something to patch up their old digesters, and from this to discover yet some durable lining and process preferable to the Mitscherlich, which they were still unwilling to adopt. So we find the American Sulphite Pulp Co. lining their upright digesters with glazed tiles about 1 in. thick, laid in cement, and their Partington digesters with about 1 in. coating of Portland cement. This thin tile lining could not, of course, endure the wide range of temperature and pressure consequent to the process, and was quickly given up. The coating of cement was more durable than one would have supposed, but there could be nothing permanent in it on account of its thinness, unequal expansion, and action of the acid upon it.

Although the use of lead for a lining was not again practically attempted, it was not entirely abandoned, for we find in 1887 one Solomon R. Wagg took out a patent for lining a digester with tiles and securing same by tamping the joints with lead. So far as I know, no use was made of this device.

In 1892 Chas. Curtis and Nathaniel Jones patented a process for lining digesters with blocks of artificial stone composed of Portland cement and ground quartz. The blocks were moulded so as to fit the contour of the digester and were laid in cement. Several mills were equipped with this style of lining, and the inventors thought they had at last found the long-sought lining. When they discovered that there was action of the acid on the blocks, they argued that it was slight, and that it was an easy and inexpensive lining to replace. It proved very unsatisfactory, however, as the dislodged quartz would get into the pulp and the blocks would become saturated with acid moisture and repairs become frequent; and the lining was abandoned in many instances.

About 1890, a digester, known as the "Solomon Brunger," was offered to manufacturers by a prominent company engaged in building paper- and pulp-making making machinery. It consisted of a double walled or jacketed vessel of iron, the sheets, nozzle, and manhole connections all welded, as by the process employed by the Continental Iron Works, of Brooklyn, in the manufacture of digesters for the soda process. It was expected and claimed for them that the indirect transmission of heat by means of the jacket would deposit a protecting coating of lime and magnesia on the interior walls of the digester, and so protect the iron. Under such conditions a coating is formed, but not one which can be relied upon to protect the iron shell. I omit details, as the digester never attained any prominence and could not endure. Some two or three mills were induced to make trial of them, little was ever heard of them, and they are now almost forgotten.

We have now come to the present state of the art, which is simply a vindication of the Mitscherlich lining; and although some slight changes have been made in the method of lining whereby two companies have ventured to build and offer this digester for sale, the honour belongs entirely to Mitscherlich, and he should be defended in his rights. It must not be forgotten that during the last 12 years, since the first Mitscherlich plant was built in Alpena, Mich., this digester has not failed to fulfil the claims of the inventor, and the companies using the process have as a rule been successful.

The construction at present in vogue, and copied after Mitscherlich, consists in a steel shell ½ in. thick, rivets countersunk on the inside, vertical, stationary, and about 14 ft. by 38 ft. to 40 ft. high, though smaller sizes have been built. This shell is first lined with sheet lead, having no points of attachment whatever (this is one of the essential claims), so as to provide for a "free movement of the lining" under variations of temperature. It would be interesting to hear the claimant explain how this "free" movement takes place with two heavy courses of bricks laid in and backed up with Portland cement and crushed quartz, and 80 lb. pressure upon same. However, it is interesting to reflect that only a few years ago these same men were straining every nerve to bind fast this unmanageable metal, suffering defeat, as all who rebel against nature's laws must. They would now give it "free movement."

As just intimated, two courses, about 8 to 10 ins. of bricks, are laid in cement against the lead lining. These bricks are made from a bastard fireclay, and are partly vitrified, hence are not impervious to moisture and acid fumes. The bricks have no tongues and grooves, but are of shapes adapted to the dimensions of the digester. The joints are finally pointed with glycerine and litharge. The shell of these digesters is required to be perforated with small holes, by the insurance companies.

For the work performed they are giving very good satisfaction, but the lead interlining, though protected by 10 ins. of masonry, is still rebellious, and leaks are of not infrequent occurrence, showing conclusively that destructive agencies are still at work on the lead. It is a wonder to me why manufacturers of sulphite will not study every detail of construction, rather than make costly and indifferent ventures. Why should an absorbent brick be used for lining? Mitscherlich, in his first digester, used a beautiful, dense, and vitrified brick, which would absorb no moisture.

The present digester for quick cooking must yet be much improved. The bricks must be vitrified and the lining must be much thicker, so that the shell will not be warmer than about 100° F., and the difference in temperature between the inside and outside will equalise the expansion. This is the only way the life of a lead interlining can be prolonged. The digester will cost more, but repairs and stoppages will be reduced to a minimum. To all of these points Dr. Mitscherlich carefully adapted his process, and time has proven the wisdom of his construction.

In this brief review of the history of a very important part of one of our largest industries, I have endeavoured to give the principal facts, as a matter of interest to all, and trust that they may be valuable to some of our chemical and engineering friends.

The history of the sulphite digester emphasizes again one most important point, conspicuous in the Graham process—that a device, machine, process, or individual, no matter how strong and powerful it may appear, when subject to an undue strain or stress in any of its essential parts, cannot endure; it will fail. Beware, therefore, men of science, to compute carefully the maximum load to be carried in every chemical and mechanical operation in which you engage; whether it be the lining of a digester, the burning of bricks, the transmission of electricity, steam, water, &c., the building of a steam boiler, bridge, or arch, there are no favours to be bestowed, no exceptions to be made.

## Discussion.

The Chairman stated that he had had the pleasure of attending the recent banquet of the American Paper Manufacturers' Association, and was surprised to learn that in the neighbourhood of New York there were about 250 tons of wood-pulp paper used every day, and about 150 tons exported. The enormous amount of spruce timber used for this manufacture must have been observed by everyone who knew the section of the country where that description of paper was made,—in Maine and elsewhere,—and in fact they were drawing now on the far-away forests of West Virginia for spruce for this business.

The matter of the cracking and creeping of lead was most interesting to all who had to do with chemical industries. They all knew what an intolerable nuisance it was. For many years he had endeavoured to control it, but had given it up as hopeless. It would seem that the method last suggested of equalising the temperature of lead and steel would be the most feasible one. It would seem that the masonry lining, if made of impervious brick, would prevent the undue temperature of the cook from affecting the lead. Possibly by water-jacketing the iron, the lead might be further cooled so that the temperature would be more equalised.

It might be of interest to mention that he had been asked his opinion of the feasibility of manufacturing sulphur dioxide, liquefying it and transporting it in steel cylinders to the paper people for digestion with the spruce wood chips.

Mr. M. Toch said he would like to know what the extremes of temperature were in the processes of digestion.

Mr. R. C. Woodcock asked why porcelain iron would not do for a digester.

Mr. Geo. A. Prochazka asked if the tiled lining used by the American Sulphite Pulp Co. at first was embedded in cement.

The Author said, in reply, that in the acid process the cooler the lime water the more easily it would absorb the gas, and that the range of temperature was from 80° F. to 310° F. The cooking temperature, at the end of the cook, was about 300° to 310° F., and the acid went on to the wood at from perhaps 75° in the winter up to about 100° in the summer, when the water was warm.

In answer to Mr. Woodcock, the porcelained iron would be very thin, and in a very short time it would crack from the undue unequal expansion. The first tile lining was used by the American Sulphite Pulp Co., and was only 1 in. thick—much greater than was found in glazed iron, and yet it was of no use whatever.

In answer to Mr. Prochazka, the tiled lining was embedded in cement; and then afterwards, in other digesters which they operated, the spherical rotary, they simply put on about 1 in. coating of cement, plastered it all over, and yet they found cracks in them in a very short time, owing to the expansive action of the iron.

## AMERICAN CHEMICAL GLASSWARE.

BY E. C. UHLIG.

While the superiority of American plate- and bottle-glass is acknowledged by all who are acquainted with the industry, it is not generally known that the manufacture of laboratory glassware is being carried on in this country, and the product compares very favourably with European manufactures in price, quality, and appearance. Acting on the suggestion of Dr. Schweitzer, I have prepared a few specimens for your examination this evening. This glass is made from the purest materials obtainable, and from analyses of the Bohemian product. The objects sought after are, resistance to the action of reagents, durability in use, and cheapness.

The materials used in the manufacture of this glass are sand, sodium carbonate (as Brunner, Mond, and Co.'s 53 per cent. soda-ash), potassium carbonate (as pure crystallised salt), and lime (as oxide, prepared from a high grade of dolomite). The sand comes from the Juniata river in Pennsylvania, and showed on analysis—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 99·75 |
| Alumina, $Al_2O_3$ | 0·04 |
| Ferric oxide, $Fe_2O_3$ | 0·03 |
| Moisture and organic matter | 0·18 |
| | 100·00 |

The soda-ash showed as follows:—

| | Per Cent. |
|---|---|
| Sodium carbonate | 98·00 |

the other constituents being divided between sodium sulphate and chloride, with traces of iron, alumina, and lime. Some moisture was also present.

The percentage of carbonate usually varies, this variation being usually due to the increased moisture.

The lime is known as Toledo lime, and contains—

| | Per Cent. |
|---|---|
| Calcium oxide, $CaO$ | 57·80 |
| Magnesium oxide, $MgO$ | 40·37 |
| Carbonic anhydride, $CO_2$ | 0·38 |
| Water, $H_2O$ | 1·29 |
| Iron and alumina | 0·11 |
| | 99·95 |

The potassium carbonate showed as follows:—

| | Per Cent. |
|---|---|
| Potassium carbonate, $K_2CO_3$ | 83·05 |
| Water, $H_2O$ | 16·75 |
| | 99·80 |

Potassium chloride was occasionally present in a minute proportion.

The materials were mixed in such proportion, and fused, as to give a glass of the following composition:—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 72·00 |
| Alumina, $Al_2O_3$<br>Ferric oxide, $Fe_2O_3$ | 2·28 |
| Lime, $CaO$ | 5·50 |
| Magnesia, $MgO$ | 3·97 |
| Potash, $K_2O$ | 6·20 |
| Soda, $Na_2O$ | 9·90 |
| | 99·85 |

A Bohemian beaker, supposed to be from the manufacture of Joseph Kavalier, showed as follows:—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 72·22 |
| Alumina, $Al_2O_3$<br>Ferric oxide, $Fe_2O_3$ | 1·46 |
| Lime, $CaO$ | 7·98 |
| Potash, $K_2O$ | 8·32 |
| Soda, $Na_2O$ | 9·53 |
| | 99·51 |

I have also analyses of two specimens of Jena beaker glass. One, known as "normal glass," had the following composition:—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 67·47 |
| Alumina, $Al_2O_3$<br>Ferric oxide, $Fe_2O_3$ | 3·70 |
| Lime, $CaO$ | 0·35 |
| Magnesia, $MgO$ | 2·68 |
| Soda, $Na_2O$ | 13·57 |
| Oxide of zinc, $ZnO$ | 4·88 |
| Boracic acid, $B_2O_3$ | 7·31 |
| | 99·96 |

The small proportion of CaO, together with the presence of $B_2O_3$, were matters of surprise to me. But I ascribe the presence of the latter to the possibility of the mineral boracite or magnesium borate being used instead of lime.

The other Jena glass, known as "Resistance Glass," had the following composition:—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 67·54 |
| Alumina, $Al_2O_3$<br>Ferric oxide, $Fe_2O_3$ | 0·98 |
| Lime, $CaO$ | 10·85 |
| Magnesia, $MgO$ | 0·09 |
| Soda, $Na_2O$ | 19·09 |
| Potash, $K_2O$ | 1·62 |
| Manganese oxide, $MnO$ | 0·33 |
| | 100·50 |

In the American glass the high percentage of silica is a good feature, and the presence of magnesia replacing part of the lime is, I believe, another aid to the resisting properties of the glass. The increased proportion of the alumina is due to the action of the mixture on the melting pots, and cannot well be avoided. In making this glass, the quality of the Bohemian ware is taken as a model, and every effort is made to have the product as nearly perfect as possible. In the mechanical qualities, uniform thinness was sought after the most, together with careful finish.

The apparatus for volumetric analysis are graduated by weight, using distilled water at 60° F., thus obtaining results identical with actual laboratory practice.

This brief review will exhibit in an imperfect manner, to the members of the Society, the efforts which are now being made in this country to build up an industry which has been so successful in Germany.

### Discussion.

Mr. M. L. **Griffin** asked if the glass annealed here would stand differences of temperature as well as glass of foreign manufacture, and whether the price was any higher.

Mr. R. C. **Woodcock** asked if his people were able now to make a full line of chemical apparatus, as they were offering to the trade all sorts of glassware.

Mr. **Geo. A. Prochazka** asked where this glassware was made.

Mr. **Wm. J. Schieffelin** asked if it was on the market in New York, and by whom it was sold.

Dr. **Virgil Coblentz** said he thought, considering the immense quantity of glass used by the institutions—the teaching institutions—throughout the country, and considering they had been for a number of years importing such tremendous quantities, it was gratifying to know of makers taking this matter up. It was surprising to him that it had not been done before. To his knowledge they had been working at this for five or six years, and they certainly deserved commendation for having developed the matter as far as they claimed to have done. The only question in his mind was, whether they could get this glassware or not. They had had a great deal of experience in handling test tubes, beaker glasses, and flasks. They all knew that if they sent in their order on the first of May to be imported, it was a question whether they could get it in September, and if they did get it then whether it would be of the quality and kind they wanted

Last fall they imported at least several gross of funnels, and there was not one of them that could be used for practical filtering purposes on account of the inequality of the sides. They had found that the glass was unequally thin, and he hoped the quality of glassware made by Mr. Uhlig's firm would be all they claimed for it. He certainly thought they deserved to be patronised by all the members of the Society and those generally using such articles, as they were trying very hard to replace foreign ware.

Dr. H. Schweitzer remarked that for the last few years he had used nothing else but Whitall and Tatum glassware, and had found it just as good as Bohemian glassware in every respect, even if some of the pieces looked perhaps a little heavy. They stood the most violent heating, and the price of Whitall and Tatum glass was very much cheaper than that of Bohemian glass.

The Chairman said that in another direction this matter of glassware interested him very much—that was the burette. Some years ago he had occasion to use some very accurate burettes, and tried at a certain place and found very few were even fairly accurate. They all measured 100 c.c. or 50 c.c. correctly, as the case might be, but between the 1 c.c. gradations of the scale they were not accurate. He went to the dealer and told him they were not right, and he would like to get some that were. The dealer took him into another room, where a man was engaged in doing something with burettes. He asked him what he was doing, and he said he was "picking out the accurate burettes"; he told the man that was the kind he wanted, but the man replied he could not have those, as a certain professor took them all and paid him a higher price for them; from which fact he judged that foreign burettes were made to sell.

Mr. Parker said that if accurate burettes could be made at home and sold at a lower price than the foreign, he hoped the manufacturers would give special attention to the burette question, and give them tubes accurately calibered and graduated.

Mr. Griffin said he thought it was a very bad thing to trust to anybody's burette, no matter how well it was made. A man in his mill came near getting him into trouble on account of using one he did not correct by weighing himself.

The Chairman said he thought it was better to get some nearly accurate, rather than have something admittedly inaccurate, if they had any on the American market which could justly claim to be correct. He had found many which claimed to be correct were very inaccurate.

Mr. Van Ingen said, in regard to the American glassware, that he had used it very satisfactorily for two or three years, and the only objection was the shape of the beakers. The lip was not inclined enough, and if one put a rod in the beaker, one could not put a glass cover on top. That was the only objection he had to them. He could speak highly of them for strength, and did very satisfactory work with them. He had only used a few assay flasks; but if that correction were made in the shape of their beakers, and the shape of the Bohemian glass were copied more closely, they would be all right. The glass was an excellent glass for ordinary laboratory purposes.

The Chairman stated that during his college days they were not permitted to use a beaker with a lip to it.

Mr. M. L. Griffin stated that, as the author had so accurately described copying the composition of the Bohemian glassware, he would like to know, as a matter of curiosity, whether they copied the mechanical operations and annealing processes as accurately.

The Author said, in reply, that they tested the beakers by boiling water in them over a naked flame, and, after throwing the water out, immersing them in ice water; they stood that test. Of course they would not guarantee them to do that; that was simply a sample of the way they tested them. The price was very much lower than the imported price They were not making a full list of chemical apparatus, but the principal glassware used for ordinary laboratory work. This glassware was made in Millville, N.J., midway between Cape May and Philadelphia It was on the market in New York, and sold by the makers, Whitall, Tatum, and Co.

In regard to the burette question, the price was higher than the imported ones, but that was due to the great care taken in making them. The tubing was certainly about as accurate as possible. Each burette was suspended over a balance, and the water weighed out by 10 c.c., and proper corrections made in order to obtain it at 60° F., and then into smaller divisions put in by measurement. That was the best way it could be done consistent with the moderate price. As to the mechanical operations, the men who made those beakers were from Bohemia, who had come to the States, and the other workmen learned from them. So far as annealing was concerned, he believed they had the same methods as the Germans.

# Scottish Section.

*Meeting held at Edinburgh on Tuesday, March 1st, 1898.*

MR. GEORGE BEILBY IN THE CHAIR.

## THE EDUCATION AND TRAINING OF BREWERS.

BY WILLIAM FREW, PH.D. (MUNICH).

We have come together this evening under exceptionally interesting circumstances, which will, I trust, remind us of certain things sometimes forgotten by the trade in whose special interest this meeting has been planned. In the prospectus of this Society we read that "the Society of Chemical Industry was founded in 1881, for the advancement of the study and practice of applied chemistry; for the discussion of *all* matters connected with the chemical industries, and for the diffusion of information on applied chemistry and chemical engineering among its members." In my opinion there is no industry which can more fitly be described as a chemical industry than that of brewing—for with what is the brewer occupied? His work can be very briefly and comprehensively described by saying that he occupies himself with breaking down complex organic bodies into simpler compounds. The Society never had any doubt of the relation in which brewing and the kindred industries of distilling and wine-making stood with regard to itself, for they are included in the long list of industries in whose interests those valuable abstracts are published month by month in this Journal. This being so, it is clearly the duty of brewers to support the Society of Chemical Industry in every way in their power, and in the first place, by becoming members of the Society. In this connection it will be well to recall what was said by Dr. Armstrong in his presidential address to the Chemical Society in 1894:—"Whilst it is our function to deal with papers embodying discoveries in chemical science, the Society of Chemical Industry deals with the applications of chemical science to industry; may I venture to utter a word of warning and supplication regarding the increasing discontinuity of operations in this field? Surely it is of high importance that this Society should focus all information concerning applications of chemical science to industry carried out in British works, &c." Dr. Armstrong then went on to deplore the fact that the dyers and colourists had started an independent journal and that Institutes of Brewing had started up in several places in England. He stated that these bodies were publishing valuable papers, many of which were of interest, not only to themselves but to chemists generally. "No time," he said, "should be lost in affiliating those bodies for the purposes of publication; if their members have not sufficient public spirit to support the Society of Chemical Industry as a whole, let reprints of the papers in this Journal which concern the members of the different bodies be sent out to them; but I trust the broad view will prevail . . . . . Admirable as is the growth of the feeling of local self-government, unless care be taken to avoid the sub-division of interests which it necessarily involves, it may have consequences from which it is the general desire to escape—undue specialisation and narrowness of purview."

Dr. Armstrong's eloquent protest is as much needed to-day as it was four years ago. It may safely be said that, in nine cases out of ten, the papers read at meetings of the various Institutes of Brewing would meet with a much more adequate and satisfactory discussion at a meeting of this Society, where they would be submitted to the criticism of representatives of the other chemical industries. Societies such as the Institutes of Brewing are very apt to acquire that narrowness of view of which Dr. Armstrong spoke, and their discussion of questions has not that many-sidedness so characteristic of the discussions at any meeting of the Society of Chemical Industry.

Mr. Beilby, in his opening address this session, showed very clearly the advantages of belonging to a Society representing all branches of chemical industry :—"As a manufacturer who has had considerable experience in working new processes, both patented and unpatented, I have, as might be expected, the utmost sympathy with the right and natural desire to conserve the results of brains and experience for the primary use of their possessors, but I can personally testify to the great help which my own work has received from the stimulus of meeting workers in other departments than my own, as well as from the unique stores of information contained in this Journal. . . . . The Society does not want yours or any other man's secrets, but it does urgently want you to join in the intelligent discussion of the scientific principles which underlie each and every manufacture, and it can promise you that if you give to the common fund some of your wisdom and experience you will receive in return the powerful stimulus of criticism which may lead you to do your own work better. And, further, by contact with the active minds in other branches of industry, your own mental horizon will be widened, and you may find that principles which have been worked out in some widely different branch of industry have a valuable application to your own special work."

The present meeting is one of a series representing a new departure in the methods of the Scottish Section. In order to stimulate that interest, which every right-thinking person connected with the various chemical industries ought to feel in this Society, the Committee have arranged that meetings shall be held at the various centres of industry, and that, so far as possible, the papers read and discussed shall be concerned with those industries. To-night we are met together in the interests of the brewing industry at its great centre in Scotland. We have no Institute of Brewing in Scotland, and I trust now that the brewers have found their way to our meetings they will bring forward for discussion the many interesting problems which arise continually in their practice. Successful brewing depends upon the intelligent application of certain great fundamental laws and their adaptation to varying conditions; there are, therefore, fewer secrets in our industry than in almost any other. Local conditions play so great a part in all the processes in brewing, from the malting of the barley to the final storing of the beer, that a special way of carrying out any one of these processes which may be most successful in A's brewery, may prove quite unsuited to the conditions obtaining in B's brewery not many yards away. To a brewer who has received a thorough education in the scientific principles underlying brewing, and has had a wide and varied practical training, the suitable modification of the various processes is simply a matter of experiment. In many cases, however, we find in place of well-planned experimenting, a reckless and unintelligent varying of conditions, which only at times and by accident leads to a solution of the difficulty. It is impossible, as every scientific man knows, to carry out a research in any line without a thorough knowledge of the main principles involved, and a very comprehensive practical training in methods of work. Without those qualifications, both in scientific work and in brewing, we can only grope in the dark hoping, almost against hope, that fortune will ultimately favour us.

It is on this account that I have chosen to speak about the education and training of the brewer, feeling, as I do most strongly, that neither of them has received sufficient attention in this country up to the present time, and I have chosen a meeting of the Society of Chemical Industry at which to ventilate my opinions, because there is, as our Chairman said at our opening meeting this session, no other body in the country so eminently qualified to discuss the vital question of technical education.

Now I venture to assert that the brewing industry in this country has not, as yet, realised the importance of a thorough education and training for brewers. There is not, as yet, a single brewing school of any kind whatever, founded and generally recognised by the brewers of England or Scotland. This is equivalent to saying that there is no definite standard of education and training required of brewers as it is, for example, in the case of engineers. Of course, I am speaking to-night only of those intending to become head or under brewers, not of the ordinary workman. Just think for one moment of the high standard of theoretical knowledge and practical training required of a man desirous of taking up a responsible position as an engineer; look at the wide range of subjects he must study, and the arduous apprenticeship through which he must pass before he can hope to obtain any such position; and then consider what the average education and training of a head or under brewer amount to. Under no circumstances would you entrust the building of a locomotive to a man who had simply attended a course of lectures on mechanics and worked in the drawing office for a few years, and yet, is this not an exactly parallel case to that of the embryo brewer in too many instances? He attends a course of lectures on brewing, chemistry, or works for a short time in some laboratory, after which he is attached as a pupil to a brewery for a few years. His education and training are then in too many instances considered complete, and he is let loose, for I can use no other words, as a fully-equipped brewer. We all know the advertisement which appears so often in the trade journals: "Young gentleman, aged 23 years, desires position as brewer or under brewer, has a thorough knowledge of malting, brewing, chemistry, and the use of the microscope and polarimeter."

We must in the future insist on a more thorough theoretical knowledge of the scientific principles upon which brewing rests, and, at the same time, on a real apprenticeship being served by brewers. We want to get rid of the idea that the operations of malting and brewing can be learned in the brewers' room, or by walking through the brewery looking at the workmen. Brewing is far less mechanical than engineering, and there is, therefore, a much greater necessity for taking part in the actual operations; to slightly alter Goethe's words, the brewer "sieht mit fühlendem Aug', fühlt mit sehender Hand."

To sum up, I believe that the following are the principal deficiencies in the education and training of brewers in Great Britain at the present time :—

1. There is no guarantee that the would-be brewer has had an adequate preliminary education to enable him to make a proper use of the special knowledge imparted to him during his pupilage.

2. The theoretical training is given at the wrong time in most cases. A few years' actual work in the malthouse and brewery should in all cases precede any scientific training.

3. The range of studies is too limited to be of any great use, and the young brewer is too often taught what is fondly called "brewing chemistry" before he has grasped the elements of chemical science. It is as if an infant were to begin spelling words of four syllables before learning its alphabet.

4. The practical training is by no means long enough, is in many cases not actual working at all, and is not for the most part varied enough. Before a young man is considered fit for the post of brewer he should have worked in several breweries, and that work should have included actual malting.

5. We have at present no adequate guarantee that the education provided by the various private schools is of sufficient quality or that it is even suited to the requirements of brewers. I will, of course, be told that there are the technological examinations in brewing held by the City and Guilds of London, and that by them we can test the young brewer's knowledge. To this I answer that no written examination can give a proper indication of the

fitness of a certain individual for a position such as that of brewer. There is such a thing as cramming even in brewing chemistry. I do not for a moment mean to say that the men at present engaged in teaching are not fitted for the work—on the contrary, we number amongst them some of our best known scientists. I only insist that we have no proper standard by which to judge a brewer's training.

To make my position more clear and to show how the advance of brewing in Germany and Austria has gone hand in hand with improved methods of training and educating brewers, I propose now to briefly sketch the development of the very complete educational system in those countries and to consider its present position.

To do this I must ask you to go back with me to the year 1832 and glance at the state of the brewing industry in Germany at that time. In that year Gabriel Sedlmayr of the Spatenbraurei in Munich, in company with Dreher of Vienna, visited England for the purpose of obtaining information regarding the brewing process. At that time brewing was at a very low ebb indeed in Germany. To quote the words of Dr. Holzner, in a memoir of Sedlmayr published in the "Zeitschrift für das gesammte Brauwesen" in 1892, "The whole brewing process was carried out on rules determined by the brewer himself. No explanation was forthcoming of the changes which took place, and the estimation of the output of the malt was altogether unknown . . . In Bavarian breweries there was no machinery and it would have been considered madness to have talked of ice-cellars." The use of the saccharometer was practically unknown.

Sedlmayr and Dreher had a great deal of difficulty in gaining admission to any brewery in England; but they got into communication with some brewery hands and, at last, they were able to make arrangements whereby they were allowed to visit a brewery for *one* hour daily. Before leaving the country they visited Glasgow, Edinburgh, and Burton-on-Trent and then returned to Germany by way of Holland, Belgium, and France.

In England the brewing industry had made great strides, and to show how different the state of the industry was in the two countries I will quote from a letter of Sedlmayr to Dr. Holzner written in the year 1880:—"Your love of investigation has decidedly over-estimated my services to brewing, which, for the most part, consisted in my being one of the first German brewers to travel in England . . . . In England we first learned the true principles of brewing; nowhere else had we found them, although we had, shortly before, travelled through Germany, Austria, and the Netherlands . . . We found everything new in England and quite different from what prevailed at home—a fact which can only be realised when one knows that our practice in the 'thirties' was still very empirical, even although it was at times successful." Sedlmayr goes on to enumerate what they learnt in malting, mashing, in the use of the saccharometer, in the conduct of the fermentation process, and in the use of machinery and improved plant; and then goes on to show how this information was utilised by Dreher and himself on their return to their own country.

I have related those highly interesting facts not only to indicate the relative positions of Germany and Great Britain some fifty years ago, but also to show how the improvement in the practice of brewing in Germany dates from the return of Sedlmayr and Dreher. Gabriel Sedlmayr was during his whole life a firm believer in the union of science and practice, and he helped in every way in his power in the establishment of stations for research in brewing science and of schools where brewers could receive an adequate training for their life-work. No one was more alive to the value of scientific research, and the Spaten Brewery in Munich was the scene of many important experiments. Thus, for instance, in 1873 Sedlmayr placed his brewery at the disposal of Dr. Carl Linde for the purpose of experiments in the production of cold, and to these experiments we owe to a large extent the ingenious and highly efficient ice-machines of the present day.

The results of his enterprise and broadness of view were not long in showing themselves. The practice of brewing in Germany improved rapidly, and interest in the scientific side of the industry increased greatly. In the year 1862 we find the first beginnings of the "Brauerbund" for the promotion of the common interests of the German brewers, and in 1871, at a congress of brewers held in Dresden, the "Bund" was formed, and Sedlmayr was chosen as the first president. At that meeting one of the speakers quoted the immortal words of the chemist Joh. Nep. von Fuchs: "Die Wissenschaft ist der goldene Leitstern der Praxis; ohne sie, nur ein blindes Herumtappen in dem unbegrenzten Reiche der Möglichkeiten." ("Science is the golden leading star of practice; without that guidance there can only be a blind groping in the unbounded realm of possibility.") To me these words seemed to have formed the motto of the German brewers, as of the German nation as a whole. No nation has so faithfully obeyed that priceless injunction as Germany; in no country do science and practice go more faithfully hand-in-hand; and nowhere have such rapid strides been made in all branches of industry in so short a space of time. It was a good omen for the future that at this same meeting Dr. Carl Lintner, sen., delivered an address on the advantages proceeding from the union between science and practice. With the name of Dr. Lintner, the revered Altmeister of the science of brewing, is associated the most important period in the history of brewing science in Germany—nay more, in the world. We find him again addressing the International Congress of Brewers in Vienna in the year 1873, on the results of certain zymotechnical researches on many points in practical brewing. In the following year Dr. Lintner published the programme of a scientific station for brewing which he, in company with Reischauer, had begun as a private concern in order that it might afterwards be developed and supported by a combination of brewers.

To Dr. Lintner, then, belongs the credit of having founded the first important station of this kind, and of instituting a departure the tremendous results of which cannot readily be estimated. In the same year, perhaps instigated by the example of Lintner, Captain Jacobsen, of Copenhagen, conceived the idea of founding a magnificent research laboratory at Alt-Carlsberg, and in 1876 this splendid foundation was in full activity. Since that time scientific work of the utmost importance has been carried out in this laboratory, and the results have been freely published to the world. I need not remind you that Alt-Carlsberg is the home of Hansen, and his epoch-making work on the yeasts is the best proof that "endowed research" is not *invariably* a failure.

Thanks to the efforts on its behalf made by Dr. Lintner, the now well-known Scientific Station for Brewing at Munich came into existence, and from that time to this has had the warm support of the brewers of Bavaria. Since that time many other similar stations have been founded in all parts of the Continent, and they have all proved of immense advantage to the brewing industry. They are in some instances subsidised by the State, but depend for the most part on the general support they receive from the trade.

Dr. Lintner had at the same time been devoting his energies to developing the brewing academy founded by him at Weihenstephan, and now so well known throughout the world. This school became the model for the many similar schools of brewing existing in Germany and elsewhere, and thus Lintner laid a further claim to the respect and gratitude of every right-thinking brewer. I do not think that I exaggerate when I say that to Lintner, more than to any other man, do we owe the great advances made in brewing generally since 1876, for, perhaps, in no other man have science and practice been so thoroughly combined.

The development of the German brewing industry has gone hand-in-hand with the increase of scientific knowledge and the better education of the brewers until a country which, when Sedlmayer made his historical journey to England, exported practically no beer whatever, is a very formidable competitor of the British brewer in many, if not all, foreign markets.* In the case of Bavaria it is probable that at the present time Munich itself produces more beer in a year than the whole of the country did in 1834.

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* At the present time Germany exports almost as much beer as Britain; thirty years ago it exported, to all intents and purposes, no beer whatever.

Having thus briefly sketched the development of the brewing industry in Germany, and having shown how closely that development has been associated with scientific research and thorough educational methods, I now pass on to describe in detail the education and training necessary for brewers desirous of making their way in the German brewing world. But before doing so I would like to point out how closely identified the great brewery proprietors were with the movement for the foundation of research laboratories and brewing schools. They saw that what was good for all was good for each one, and recognised, therefore, that to be of any use the work done at the different stations must be freely published to the world. By their hearty co-operation with the brewing academies they have so raised the standard of quality in the case of brewers that the brewing director in most of the large breweries in Germany may very fairly be classed along with the technical director of (say) a chemical work as regards scientific knowledge and practical training.

At present there are at least ten brewing schools in Germany and Austria, of which the four most important are :—

1. The Royal Agricultural and Brewing School at Weihenstephan, near Munich. Of this school Dr. Carl Lintner, sen., was for many years the director, and it is to his untiring energy that its present position is due. It is the oldest of all the brewing schools, and has, more or less, served as the model for the more recent foundations. It is subsidised by the Bavarian Government, and its diploma is highly prized by brewers throughout Germany. It is served by a large staff of professors and teachers, and has attached to it a small brewery and a research station.

2. The Brewing School attached to the Experimental Station for Brewing in Berlin, which is upheld by the "Verein Versuchs- und Lehranstalt fur Brauerei in Berlin."

3. The Brewing Academy at Worms, which was founded in 1861, and is thus in point of age next to Weihenstephan.

4. The Academy for the Brewing Industry in Vienna, which was founded in 1895 by the "Verein der oester. Versuchsstation fur Brauerei und Mälzerei in Wien," and which stands in organic connection with the Experimental Station and the Royal and Imperial Technological Museum.

Besides these there are smaller schools in Augsburg, Munich, Worms, Vienna, and Prague. These schools, small as well as large, are provided with class-rooms and laboratories, and have in all cases experimental maltings and a brewery attached to them.

Thus we find, in the case of the Vienna Academy, that the block of buildings containing the lecture-rooms and laboratories covers 385 square metres and is two stories high. In this block are lecture-rooms, chemical, physical, and photographic laboratories, reading-rooms, and a permanent exhibition of machines and appliances for use in breweries and maltings. The experimental brewery adjoins the Academy, and comprises floor and drum maltings, a malt-kiln, a brewhouse fitted with direct and steam-heated coppers, fermentation and lager cellars, and a complete plant for the continuous culture of pure yeast. This Academy, of which Professor Schwackhöfer is director, possesses a teaching staff of 21 members, besides the maltster, brewer, and engineer in the experimental brewery, and may be regarded as a very complete example of a modern brewing school.

It is worthy of note that the Weihenstephan, Berlin, and Vienna Brewing Schools are in connection with experimental stations, and are thus kept in touch with the brewing industry in general.

We must now turn our attention to the scheme of education adopted by the three principal schools, which is more or less followed out by the minor institutions. I will take the case of a young brewer intending to enter the Academy at Weihenstephan in order to obtain its much-coveted diploma. In applying for admission as a student he must satisfy the authorities of the school that he is over seventeen years of age, and that he has had at least two to three years of practice in a brewery. I am told by the friend (Mr. J. Klingler) to whom I am indebted for this information, that the average number of years served by those studying at Weihenstephan is from five to six, and that their ages vary from 24 to 27 years. This practical work is real, and does not mean playing about in a brewers' room, as is too often the case in this country; the young brewer enters the brewery as a workman, and passes through the maltings, mash-house, fermenting room, and cellars, doing all the work side by side with the other employés. He serves an apprenticeship like any other workman, and acquires a good practical knowledge of the malting and brewing processes, which enables him to take full advantage of the education offered to him by the various brewing schools.

But practical training alone is not sufficient; he must also show that he has had an adequate preliminary training at school. The Weihenstephan authorities require of the intending student that he shall have at least the scientific training qualifying for the "einjährig-freiwilligen Militärdienste." To understand what this means we must look for a moment at the course of study in the "Realschulen"—the "modern" secondary schools, as opposed to their "classical" analogues, the "Gymnasien." The German boy passes from the elementary school to the "Realschule" at the age of ten or eleven years, and remains there for six years. During the whole of that time he is more or less occupied with the German language, literature, geography, English and French, ancient and modern history, arithmetic, and mathematics. The last-named subject includes algebra (logarithms and equations up to the third degree), plane geometry, trigonometry, &c. During two years he takes physics (including mechanics, acoustics, optics, heat, and electricity); lineal drawing, including model drawing; and freehand drawing from copies and plaster ornaments. In his second last year at school he takes botany, whilst zoology and mineralogy are relegated to the last year along with inorganic chemistry.

It will be seen from this very hurried glance at the education given in the "Realschulen," that by it the boys are well fitted out for their future technical or commercial careers.

If the brewer is able to satisfy the authorities of the school with regard to these various points, he is admitted as a student and begins his work there. It is, however, not unworthy of note that he has in most cases served his one year in the army, and has there learned those habits of obedience which are of such great value to all workmen and students. To quote the words of Mr. David Howard in introducing the discussion on technical education at the London Section of this Society in 1889 :—"One did not find that the English lad of eighteen was taught those lessons which the German lad of eighteen was taught, and which were not commonly called technical education. The German lad was taught military service. They might ask, What had that to do with chemistry ? to which he would reply that intelligent obedience was the foundation of all successful manufacture."

The course of study at Weihenstephan lasts for a year, which is sub-divided into a winter and a summer session. During the winter session there are lectures on physics, general machinery, brewery machinery, inorganic chemistry, botany (with special reference to yeast) hops, brewing practice, attenuation theory and control of work, book-keeping and the theory of exchange, taxation of beer. There are also practical courses in the chemical laboratory and on the use of the microscope, besides practical work in the maltings and brewery attached to the school. In the summer session lectures are given on brewery machinery, organic chemistry, fermentation chemistry, zymotechnical analysis, barley, brewing, faults in working, pure yeast culture, architecture, theory of exchange. Then there is practical work in the chemical laboratory (zymotechnical analysis); in the physiological laboratory (pure yeast culture); and in the maltings and brewery as before. Besides all this the student may also, if he so wishes, hear lectures on law, outlines of political economy, commercial geography, and distilling; but these are not obligatory.

At the end of the summer session examinations are held in the various subjects and the successful men receive their diplomas. Here it is well to note that there is a great difference between this system of examination and the technological examinations of the City and Guilds of London. In the first case, the students' work for a whole year is taken into account and is thrown into the balance along with his written examination, thus rendering the cramming system more or less useless.

After leaving the brewing school the brewer works for a year or two in different breweries so as to get the maximum of experience; or else he may take the position of brewer ("Braumeister") in one of the smaller breweries. He then gradually works his way up, perhaps taking a position as maltster ("Obermälzer"); foreman in the fermenting room ("Gahrfuhrer"); or mash-room man ("Biersieder") in one of the larger breweries: until at last he attains the aim of his ambition and is chosen as brewer or brewing director in one of the large breweries.

Besides the ordinary course of study, which can only be taken advantage of by those with a sufficient preliminary education, there are in many of the schools short summer courses for working brewers. There are also short practical courses of about ten weeks in length, in pure yeast culture and the control of work, which are intended for those brewers who wish a more thorough knowledge of the methods for the production of pure yeast on the large scale and who desire to be able to control the working of their breweries in all departments.

As regards the cost of education I may state that at Weihenstephan a Bavarian student pays about 7*l.* 10*s.* in fees for the winter session and 5*l.* for the summer session. For the ten weeks' practical course in pure yeast, &c., at the Berlin school the fees amount to 12*l.* 10*s.*, with the addition of 1*l.* per month for a room, as those taking the course usually live in the brewery building.

This, then, is a very brief description of brewing education in Germany at the present day. It is hardly necessary to point out the differences between it and the *want of system* in our own country. In Germany, a brewer wishful to take a good position in his profession must be an exceedingly well educated man in all directions to begin with; he must have served an apprenticeship as an ordinary workman for at least two years before entering the brewing school; at that school he must pass through a thorough course of technical education in matters relating to brewing, and, finally, he must again work in several breweries before he can hope to become head brewer.

How differently we manage things in Britain! Here, we do not insist on a sufficient preliminary education for our intending brewers; we give them, in some cases, a few months at a private "school for brewers" or at some brewing laboratory, from which they migrate, if they are rich enough, to the brewers' room. During their stay there they walk about the brewery and look at things being done by the workmen, instead of working side by side with them. If we admit for one moment that the German system of education and training is a good one, we must with the same breath acknowledge that our own condition with regard to this important matter is one of semi-barbarism.

What, then, are the causes which have so hindered the progress of education and of really adequate research in this country? To this there is but one answer—the silly jealousy of the great brewers one of another, their failure to recognise that what is beneficial to all is beneficial to each one. Of the truth of these assertions there can be no better proof than the fact that no single British brewer has attempted to imitate Captain Jacobsen in the institution of a laboratory for research in matters relating to brewing; that no group of brewers has ever attempted to found a scientific station for brewing; and that there is not in the whole of Great Britain one single brewing school with an adequate equipment or a sufficient staff of teachers.

Now it has seemed to me that here in Scotland we have a perfectly free hand. We are not hampered with any Institute of Brewing, we have no supposititious vested interests to consider. I would, therefore, ask the Scottish brewers to seriously consider the possibility of founding a school for brewing in the capital of Scotland. It might be found possible to make arrangements with the Heriot Watt College whereby part of the education might be imparted in that institution. This is not the time to bring forward any special scheme, that could only be done when the brewers act together in this matter. It must be remembered above all things that no school can possibly be successful unless it is supported by a majority of the great brewery proprietors. Is it too much to ask them to throw aside that childish jealousy which has for so long prevented any advance in this matter?

I have just noticed that the brewers of Birmingham are moving in this matter, and that they are considering the possibility of founding a school for brewing in connection with Mason's College. I shall feel more than repaid for any small trouble caused by the preparation of this paper if I hear of the Scottish brewers moving in the same direction.

My sincere thanks are due to my respected teacher, Professor Aubry, Director of the Munich Scientific Station for Brewing, and to Mr. Klingler of Wellpark Brewery, for their kind assistance in the preparation of this paper.

### Discussion.

Mr. Stenhouse expressed his approval of Dr. Frew's suggestions, and endorsed his statements regarding the education of brewers.

Mr. Irvine asked if the author could give some idea as to the cost of establishment of such a college or chair as he had suggested, having for its object the special instruction of students in matters relating to brewing.

Dr. Frew in reply said that it was difficult to give a definite answer to that question. An efficient brewing school would require a small brewery and maltings, and he thought that 20,000*l.* would be sufficient to provide these along with facilities for teaching. The additional expense would depend very largely on the nature of the laboratory fittings, but as they would be able to make use of the classes at the Heriot Watt College in Edinburgh for instruction in some of the subjects he was of the opinion that the above-mentioned sum would represent the initial expense, but would not include any endowments.

Mr. John Hunter asked if the beer from these experimental breweries was usually sold, and on receiving a reply in the affirmative, said that he did not think the Edinburgh brewers were likely to subsidise them.

Mr. Howard remarked that, as every subscriber would probably be also a brewer, this difficulty was easily disposed of, if each subscriber took a share of the beer and passed it through his own brewery.

Mr. Falconer King said that Dr. Frew had made the very interesting and striking statement that Germany now exported almost as much beer as Great Britain, whereas this country practically supplied the whole world fifty years ago, and asked how he accounted for this. He asked if Dr. Frew considered this to be entirely or even principally due to the quality of the beer brewed in Germany or to the superior way in which the German commercial men conducted their business. It was frequently stated that this country was losing ground in many manufactures not on account of any inferiority in the quality of the products, but because the wants of customers in foreign countries were not sufficiently considered, the English in many cases did not speak and correspond in the language of the country, and did not bring the goods into the particular form desired. He thought that this partly explained the great increase of the German beer export.

Dr. Ingle pointed out that the Skinners' Company had founded a chair and equipped laboratories at the Yorkshire College, Leeds, for the purpose of providing instruction in tanning, and that these had proved a great success. He also mentioned that the Cloth-workers' Company in London had established a research laboratory in connection with dyeing, and had appointed in connection therewith an assistant whose duties were chiefly to engage in and direct research work in dyeing. He thought that these examples might well be followed by the brewers in Scotland.

Mr. Hunter said that Dr. Frew had referred to the examinations of the City of Guilds of London Institute as being somewhat unsatisfactory as compared with those of the German schools. He (Mr. Hunter) had no hesitation in saying that the City and Guilds examiners were as capable and searching in their work as any body of examiners in Germany, and that their certificates were as highly prized in England as the German ones were in Germany. He was of the opinion that the brewers in Scotland received a good practical training, going as they did through all the details of brewing, and he considered that as a result of this training they turned out as good brewers as those of any other country.

Dr. Frew, in reply to Mr. Falconer King, said that he did not think that the superior commercial methods of the Germans afforded a sufficient explanation of the increase in their export, because in America, where a few years ago there was hardly a lager beer brewery in existence, practically all were lager beer ones at the present time. He thought it due more to a demand for a lighter and more refreshing beer. He admitted that Mr. Falconer King's view might to a certain extent explain Germany's position, but would point out that they in this country had formerly all the advantages in the matter of export. He thought if the beer of this country had been better, that the supremacy would have been retained. The manufacture of colours was a case where practically everything had been lost and where Germany had taken up the whole of the industry. That was entirely due to the very thorough system of technical education that existed there and was in fact the result, as he had stated in his paper, of the intimate and constant connection between science and practice which was so apparent in Germany.

In reply to Mr. Hunter, Dr. Frew said that he (Mr. Hunter) had misunderstood his remarks regarding the City and Guilds examinations. In comparing these with those of the German schools, he did not think he had cast any reflections whatever upon the examiners. The difference which he emphasised was that, while in the latter the practical work which had been done during training was taken into account, this was not the case in this country. The examinations here were solely in practice and theory; no consideration could possibly be given to the work done during the years of training, and it was consequently not possible to arrive at such an accurate estimate of a man's capabilities as was obtained by the method of examination followed in Germany.

Mr. A Forbes Watson pointed out that the founding of a brewing school would involve not only the initial expenditure of a considerable amount of money, but also a large current expenditure, because he felt sure that the fees they might expect to get would not meet the current expenses. The school could not be self-supporting, and he did not know where the funds were to come from unless from the brewers themselves. Would a brewer, he asked, look upon the expenditure as a profitable investment? Would he derive any benefit, direct or indirect, from such an institution? If he himself possessed a brewery with a number of tied houses he would have some difficulty in seeing wherein the advantages lay, as he would have a profitable enough business and would probably brew a tolerably good beer; if anything went wrong he would call in expert aid from London and this would be set right again. If, on the other hand, he possessed a brewery with a general trade, he would be in one sense in a more enviable position, and in another sense in a more precarious one, because in that case the sale of his beer would depend upon its merits. He would then be extremely careful as to the qualifications of his brewers, and would make sure that they were thoroughly acquainted with the science and practice of brewing. It would be necessary for him to educate the men himself, but he would regard the money thus expended as a kind of insurance premium against accidents. By employing only such men as had received some sort of systematic scientific education he would minimise the risk of anything going wrong. The intimate connection between chemistry and brewing science rendered a thorough training in chemistry necessary, but he would not care so much for actual chemical knowledge as for the possession of a trained head and hands. When he had secured a man possessing these qualifications he would make him spend a year or two in the brewery, working along with the workmen and going through all the practical processes there until he became thoroughly and personally acquainted with them. When the brewer had reached this stage it would be necessary for him to specialise, but this specialised education, as Dr. Frew had pointed out, could not be obtained in this country, and he thought that this defect should be remedied by the formation of a brewing institute of some sort. The practical advantages of such an institute had already been stated; he might point out further that analyses for the brewers might be carried out in the institute, and that as most brewers had at one time or another felt the want of an experimental plant, they would recognise the value of this, as they did not care to make experiments on a large scale in the brewery. He thought that the principal of such an institute should possess a thorough practical knowledge of brewing, and should be constantly in close personal contact with the brewers themselves, so that he might consult with them as to the work to be done.

## AN IMPROVED ELECTROLYTIC STAND.

BY HUGH MARSHALL, D.SC., F.R.S.E.

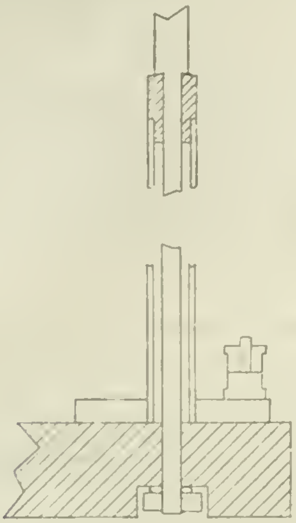
The improvement in the electrolytic stand here shown consists primarily in the method of making connection with the electrodes. In most stands the conducting wires are brought directly to the supporting arms, which are insulated from one another by means of glass rod forming part of the supporting rod or of one of the arms. The wires in such a case are very apt to get in the way, especially when space is limited, and the stand may be shaken or upset by the wires becoming entangled with other apparatus, &c., while being shifted. This is avoided in the present stand by bringing the wires to fixed terminal binding screws on the base, which is made of suitable insulating material, and these terminals are in direct connection with separate parts of the supporting rod bearing the electrode arms.

The base of the stand consists of a slate block 6 ins. × 4 ins. × 1 in. The lower part of the vertical support consists externally of a brass tube 6 ins. × $\frac{1}{2}$ in., having at its lower end a flange about 2 ins. in diameter and about $\frac{1}{4}$ in. thick. The under side of this flange has two projecting pins which fit into corresponding holes in the slate. The upper end of the tube is fitted with a vulcanite collar through which passes a brass rod 15 ins. long. Above the collar this rod has a diameter of $\frac{3}{8}$ in.; the remainder is only about half that thickness. The narrower portion passes axially right through the brass tube and through a hole drilled in the slate base near one end. The under side of the slate is countersunk to allow of a brass nut being screwed on to the end of the rod. A binding-screw terminal is fitted on the base at the side, being held in position by a nut also sunk into the under side of the slate; it is connected with the central rod by means of a stout copper wire or strip also embedded in the slate. The other binding screw is fixed directly on the flange of the brass tube.

To put the parts of the apparatus together the bottom of the flange is first covered with waterproof cement and placed in position over the hole in the base; the vulcanite collar is inserted into the upper end of the brass tube, and the rod passed through so that its shoulder rests on the collar and the screw end fits into the hole in the slate; the loop of the connecting wire or strip is placed over the end of the rod, and the nut finally screwed tightly on, binding all firmly together. The lateral binding screw is fixed in position in a similar manner, after which the hollowed-out parts on the bottom of the slate are filled in with wax or cement to protect the connections.

The figures (see next page) represent sections through the two principal parts of the apparatus—the vulcanite collar and the end of base.

In this way the central rod is insulated from the surrounding tube and held in position by the slate and the vulcanite collar, and each part of the support is connected with one of the terminals. The electrode supports consist of a brass ring with boss fitting fairly closely on the brass tube, and of a brass rod with boss similarly fitting the upper part of the vertical rod. The cathode ring has the usual three small platinum studs to support the basin. The extremity of the anode rod is composed of a binding screw and washer to clamp the anode wire. All non-contact parts are lacquered, or, better still, the whole external metal surface may be gilt or platinated.



The electrode supports can be freely moved about their respective parts of the vertical support without the least trouble from the wires. The terminal connections being close together and in a fixed position on the stand, a flexible insulated double lead may conveniently be used for joining up. All binding screws are "Post Office pattern."

# Obituary.

## FERDINAND HURTER, Ph.D.,

Formerly Chairman of the Liverpool Section of the Society of Chemical Industry, and Member of the Publication Committee of the Society since 1883.

As it is the earnest wish of the Liverpool Section of this Society, to do special honour to the memory of Dr. Ferdinand Hurter, it is proposed that a full Review of his Life and Work shall be presented at the next meeting of the Section. Hence, it is not considered desirable to give in this place more than a brief reference.

Dr. Hurter had been out of health for some time past with bronchial catarrh, but of late he appeared considerably better. On the evening of Saturday, March 5th, he had just finished tea with his family and was very happily chatting with them, when death came instantaneously upon him, through the rupture of a blood vessel of the heart (aneurism of the aorta).—W. S.

## HORACE KOECHLIN,

Member of the Society of Chemical Industry, Editor of the "Revue Generale des Matieres Colorantes," &c.

When the united names of Horace Koechlin and Léon Léfèvre appeared so short a time ago as the early part of last year, as joint editors of their new journal the *Revue Générale des Matières Colorantes*, it was little thought that that union, so soon as the opening of the new year, would be broken by death.

That that journal should have at once assumed a pre-eminent position in the world of journals devoted to the tinctorial arts and industries was, however, but the natural outcome of the application of a mind richly stored with scientific and practical experience and enriched by learning.

The following is a brief sketch of the career and work of the subject of the present notice:—

Horace Koechlin was born in Glasgow in 1839, and returned whilst still a child to Mulhouse with his parents. Here, later on, he received his chemical training in the École de Chimie under Schützenberger. The theoretical studies of young Koechlin being completed, he received practical instruction from his father, Camille Koechlin, and under his guidance he soon became an expert calico-printer. Horace Koechlin subsequently occupied various positions in Rouen, Glasgow, Mulhouse, and Wesserling, and was for many years the technical director of the firm of Koechlin-Baumgartner and Co. in Loerrach, to the world-wide renown of which he materially contributed. Relinquishing his position in Loerrach, he went some years ago as partner in a silk dyeworks to Lyons, but this not turning out satisfactorily, he undertook the management of a factory in Rouen, where he remained till the last.

It is a singular fact that Horace Koechlin represents a third generation in which genius for the application of dyestuffs by printing and dyeing has been maintained, he being the son of Camille Koechlin and grandson of Daniel Koechlin, two men to whom the calico-printing industry owes a number of most important and interesting discoveries.

Though afflicted for some years past by a terrible malady, his indomitable energy bore him up, and thus he continued a busy worker until the last day of his life.

Though H. Koechlin's published works are all contained in the *Bull. de la Soc. industrielle de Mulhouse*, yet these do not at all represent the scope of his scientific and technical activity, for many of his discoveries have not appeared in print, and are only known through personal communications to friends and colleagues. But not only in the fields of bleaching, dyeing, and printing have his skill and powers of observation borne fruit; in the production of dyestuffs he was also successful.

Horace Koechlin discovered Gallocyanine, and, in conconjunction with Witt, the Indophenols (this Journal, 1882, 66 and 255—258). The production of the Phenocyanines, perfected by de la Harpe in the firm of Durand, Huguenin and Co., was due to the result of one of his discoveries. Among his more important technical achievements may be mentioned the introduction of the chrome mordants in dyeing and printing, the shortened Turkey-red process (in conjunction with G. Galland), the direct production of azo dyes in dyeing and printing, also the compound metallic mordants, and the chlorination of wool, &c. In recognition of his services, he was, in 1889 (Paris), made a Knight of the Legion of Honour, and the industrial societies of Mulhouse and of Rouen honoured him with their highest distinctions.

By his great amiability, the readiness with which he communicated the results of his wide experience to friends and colleagues, and lent a helping hand to beginners, he made a large number of friends, who will now mourn his death, not only in Mulhouse and Rouen, but in all parts of the world. His decease took place on January 16th.—W. S.

## SIR HENRY BESSEMER, F.R.S.

The founder of the Bessemer process for the manufacture of steel died on Tuesday last, March 15th, at his residence, Denmark Hill, S.E., at the mature age of 86. When 18 years of age, Bessemer came to London from Charlton, and found employment in engraving patent-medicine labels on steel with a diamond point, and also in modelling in clay. About this time, public attention was drawn to a practice which had arisen of transferring deed stamps from old to new documents, and thus defrauding the revenue. Bessemer devised a stamp which could not be used twice, and he then placed himself in communication with the authorities. His stamp could not be transferred, was impervious to moisture, and would not yield a duplicate. One of his first ventures was the manufacture of gold paint. He, his wife, and his brother-in-law used to compound the mixture with their own hands in order to keep the method of manufacture a secret.

He was thus early shown to be the possessor of inventive faculty. But he also gave evidence of shrewd business habit, perception, and judgment, qualities which are too frequently not associated with inventive power. To this somewhat uncommon union of faculties his great success in life was no doubt due in large measure.

But Bessemer's great claim as an inventor is in connection with his metallurgical work; for, by the improvements he effected in the manufacture of steel, he brought about a world-wide revolution. Prior to this, iron was decarbonised by the process of puddling. Bessemer conceived the idea of stirring up or agitating the molten iron by means of the very reagent used—*viz.*, air—for oxidising the carbon in the iron, and thus decarbonising the metal.

The new process was not well received by scientific workers; and a paper which Bessemer read before the British Association at Cheltenham was not thought worthy of being even summarised in the official report of the proceedings of the Association. It was generally regarded as a visionary project, and would probably have been forgotten but for that other faculty which Bessemer possessed, *viz.*, business shrewdness, habit, and energy. The discredited inventor now established works at Sheffield, wherein his own process was carried out. He was here also assisted by Robert Mushet, who introduced that improvement of essential importance in this process—the addition of a definite amount of spiegeleisen to the charge, after having blown out all the carbon. Bessemer was then very soon able to adduce that most cogent argument in favour of his improved process, whether for the scientific man or the ironmaster—the production of Bessemer steel at a price some 20*l.* per ton lower than any other steel!

After this followed recognition and honours. Bessemer was elected a Fellow of the Royal Society in 1879, was knighted the same year, and died wealthy and full of honours.—W. S.

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* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

## I.—PLANT, APPARATUS, AND MACHINERY.

*Iron Plates and Tubes that have failed in Use, Composition of.* Finkener. Mitt. aus d. k. tech. Versuchsanst. zu Berlin, 1897, **15**, [6], 277.

*See under* X., *page* 248.

### PATENTS.

*Mixing together Liquid and Gaseous Bodies, Improved Method of, and Apparatus therefor.* A. J. Boult, London. From H. H. Dikema, Brussels. Eng. Pat. 1843, Jan. 23, 1897.

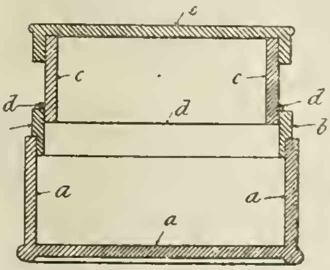
The invention is applicable for the impregnation of beer, wine, alcohol, effervescing water, &c., with carbonic acid, oxygen, &c., the liquid and gas being mixed by atomising or spraying the former into the latter. The apparatus employed consists of a series of closed vessels, suitably arranged, and supplied with the gas, each vessel being provided with an atomiser through which the liquid is discharged into the vessel. The liquid is passed through all the vessels of the series, and is discharged from the last vessel into a collecting tank. Each atomiser consists of a spiral or tube formed of a coil or coils, or of separate rings of wire, &c., and is fixed at the end of the inlet tube, its extremity being closed by an air-tight cap (a kind of blank flange), so that the liquid is forced to pass through the interspaces between the coils or convolutions of one coil. The size of the interspaces is regulated in accordance with the pressure of the liquid, and can also be adjusted by hand by means of a screw spindle bearing on the closing cap.
—R. A.

*"Coffey's" Distilling Apparatus and the like, Impts. in and relating to.* D. A. Blair, Glasgow. Eng. Pat. 4612, Feb. 20, 1897.

The tubes through which the "wash" or liquor passes, are fitted to their frames, so that the individual tubes of each frame can be removed or replaced without displacing the frame.—R. A.

*Separator for Use in connection with Granulated Substances, Liquids, and the like, An Improved.* R. H. J. G. Huet, Haarlem, Holland. Eng. Pat. 29,576, Dec. 14, 1897.

The separator consists of a receptacle *a*, fitted with a ring *b*, and a cylinder *c*, over the end of which is placed a sieve *d* of silk gauze, &c., the sieve being held in position by the ring *b*. The upper end of the cylinder is closed by the cap *e*. In use, a sieve of comparatively large mesh is first employed, and for the further separation of the matter which passes into the receptacle *a*, this sieve is replaced by another of finer mesh. A series of sieves of suitably graduated mesh may thus be employed.—R. A.



## II.—FUEL, GAS, AND LIGHT.

*Illumination by Flame, The Modern Development of.* H. Bunte. Ber. 1897, **31**, [1], 5—25.

Since the introduction of gas lighting at the beginning of the century, gas and petroleum flames had become almost the only artificial sources of light until the advent of the arc light. Then the competition between flame illumination, or the chemical light, and the electric light commenced. F. Siemens introduced regenerative gas burners, and Auer von Welsbach invented the system of gas lighting by incandescence. The reason of the high illuminating effect of Welsbach mantles has hitherto evaded discovery. Lewes ascribed it to a transition of the materials of the mantle to a crystalline state, and Drossbach to a peculiar resonance of the earths towards waves of light. These and the other explanations offered have been unsatisfactory. Killing, however, showed that the mantles were capable of exercising contact or catalytic effects, but his theory attributed a special power of emitting light to the materials. In April 1896 the author expressed the view that very high temperature alone was the cause of the very strong luminous effect, and observation has since confirmed that view.

The Welsbach mantles consist of 98—99 per cent. of thoria and 1—2 per cent. of ceria, and are made by dipping purified gauze in a solution of the nitrates of thorium and cerium, and subsequently incinerating the fabric. The incineration of thorium nitrate yields the oxide in the form of a very voluminous fibrous mass, whereas ceria similarly obtained is merely a fairly porous material. It was shown, by exposing small prisms of magnesia coated with each of the oxides and with the Welsbach mixture alongside similarly-shaped prisms of magnesia and carbon in a tube of carbon heated by an electric current to at least 2,000 C., that neither of the oxides nor the mixture possessed any peculiar power of emitting or radiating light when the phenomena of combustion were absent. Thoria was also shown to have no influence on the combustion of hydrogen and oxygen, as the temperature of ignition remained about 650° C., whether thoria was present or absent. But ceria lowered the temperature of ignition to 350° C., or in other words, induced combination at a temperature 300° C. below that at which it would naturally have occurred. It may be safely assumed that ceria exerts a similar action at the temperature of the flame gases, and provokes rapid combustion of the previously heated gas. A mantle, consisting wholly or largely of ceria, is nevertheless useless. An examination of the behaviour of platinum, which likewise shows powerful contact action, elucidates this apparently anomalous behaviour of ceria. Platinum gauze or thick wire glows only to a moderate extent in the Bunsen flame, but very fine wire may be melted; thus showing that a temperature of at least 1,800° C. prevails. The conductivity of the gauze and thick wire prevented any particle attaining aught but a moderately low average temperature. Evidently platinum, ceria, and other catalytic substances, must be insulated in the flame on a bad conductor, in order that the maximum temperature and intense incandescence may be obtained. The thoria, in virtue of its refractory yet voluminous nature, makes an excellent insulating material for the particles of ceria distributed over it in the Welsbach mantle. The catalytic action of the ceria thus has full play, and the particles of it attain very high temperatures—far above 2,000° C.—instead of the whole mantle remaining at a uniform moderate temperature.

The volume of the luminous part of a gas flame having an hourly consumption of 150 litres and an illuminating power of 20 Hefner units, is about 2 c.c. at 0° C. It may be calculated that about 54 mgrms. of carbon are separated in the flame from 1 litre of coal-gas, and thus it appears that the 20 Hefner units illuminating power are brought about by the presence in the flame at any one moment of only 0·1 mgrm. of incandescent carbon. The ceria in a Welsbach mantle amounts to about 4·0 mgrms., or about 40 times the weight of the carbon in an ordinary luminous gas flame. Therefore the small percentage of ceria in a Welsbach mantle appears to be ample to afford the illuminating power of about 70 Hefner units. The analogy between the incandescent gas light flame and the ordinary luminous gas flame is thus apparent. At present, no efficient substitutes for thoria and ceria in the production of mantles are known. It is essential that a sufficient supply of air for the complete combustion of the gas should reach the mantle. The 2 to 2½ volumes drawn up the burner tube only suffice to disillumine the flame, and the extra supply of air needed must reach the flame externally. Chimneys with perforations for the admission of air to the mantle give a considerably higher efficiency than do ordinary chimneys, which create only a draught parallel to the walls of the mantle. The use of gas or air under high pressure also gives an increased efficiency to incandescent gas burners.

Acetylene ignites at a lower temperature (480° C.), and affords a higher temperature (2,420° C.) on combustion than any other combustible gas. All mixtures of acetylene with air, except those containing less than 5 per cent. of acetylene or less than 20 per cent. of air, are explosive, whereas only mixtures of coal-gas and air which contain from 7 to 30 per cent. of coal-gas can be exploded. The rate of propagation of the combustion is very high with acetylene. The combustion does not, however, extend backwards in a tube of 0·5 mm. internal diameter, and the most explosive mixtures of acetylene and air may be consumed at ordinary burners from such a tube. Acetylene is an endothermic compound, and at more than two atmospheres pressure is very liable to explosive resolution into its elements. The danger is especially great with the liquefied gas, but since 1 kilo. of calcium carbide occupies a space of only 0·45 litre, while the 300 litres of gas obtainable from it occupy on liquefaction double that space, it is evident that carbide is a better form than liquefied acetylene for transport. Carbide appears specially suitable for storage on railway carriages, provided a simple and safe means of generating the acetylene from it is devised, and a method of burning the gas without the burners becoming choked with carbon is discovered. To light a railway carriage by 200 candle-power for 10 hours requires about 15 cb. m. of coal-gas, 8 cb. m. of oil-gas, but only 1·5 cb. m. of acetylene. This volume of acetylene would be obtained from 5 kilos. of carbide. Compared weight for weight with lead accumulators for electric lighting, carbide gives 30 times the lighting power. Acetylene may be safely generated from carbide by a stream of acetylene or other gas saturated with steam. The continuous current carries away heat and prevents the temperature rising unduly.

Gas in Berlin costs 16 Pfennige per cb. m. A light of 20 Hefner-units power at that price of gas costs: in flat-flame and Argand burners, 2·4 Pf.; in Siemens' regenerative lamps, 1·1 Pf.; in the incandescent light with the latest pattern of mantles, 0·53 Pf., or with the gas at high pressure, 0·32 Pf. Acetylene at 125 Pf. per cb. m. gives the same light for 1·63 Pf.; petroleum at 25 Pf. per kilo., for 1·5 Pf.; and spirit at 30 Pf. per kilo. (in incandescent burners), for 1·8 Pf. Notwithstanding the immense increase in the light obtainable from a given quantity of gas, the production of German gasworks has increased at a greater rate in the last quarter of a century than at any earlier period.

—J. A. B.

*Carbides of the Metals of the Alkalis and Alkaline Earths, and of Carbide of Magnesium; Conditions of Formation of the.* H. Moissan. Comptes Rend. 1898, **126**, [4], 302—308.

Certain carbides, such as those of the metals of the alkalis, cannot be prepared by reduction of the oxides or carbonates by carbon in the electric furnace. Berthelot obtained the compounds $C_2HNa$ and $C_2Na_2$ by exposing sodium to acetylene at a moderate heat (Ann. de Chim. et de Phys., 4th series, **9**, 385). The conditions of formation were therefore studied.

Potassium carbide was not formed from a mixture of potassium carbonate and carbon in the electric furnace with a current of 900 ampères and 15 volts. The mixture of black flux and carbon used for the preparation of potassium, gave, under a current of 350 ampères and 45 volts, a coarse powder, which, on contact with water, disengaged gas containing 66·6 to 70 per cent. of acetylene. Very little gas was disengaged, and it was clear that the temperature of the electric furnace was much too high for the preparation of potassium carbide. Potassium was observed to be slowly attacked by acetylene in the cold and at ordinary pressure. 17 c.c. of acetylene in 49 days thus afforded a residue 8·35 c.c. in volume, and containing 4·18 c.c. of hydrogen. The volume of acetylene absorbed was therefore about double that of the hydrogen produced, thus confirming the equation $C_2H_2 + K = C_2HK + H$.

The product $C_2HK$ thus obtained was white, and on contact with cold water disengaged 7 c.c. of pure acetylene. Partial polymerisation had reduced the volume to rather less than that indicated by theory.

Sodium carbide was not formed by the reduction of sodium carbonate by carbon under a current of 1,000 ampères and 70 volts. A mixture of soda and sugar charcoal in a closed crucible under a current of 350 ampères and 45 volts gave a black powder, which disengaged a little acetylene on contact with water. Acetylene gas is absorbed to a very slight extent by sodium in the cold. A sealed tube containing sodium and dry liquid acetylene was exposed for several days at the temperature of the laboratory, and the metal was completely converted to a yellowish-white substance. On opening the tube, free hydrogen was detected in the excess of acetylene. The white compound on contact with water yielded acetylene, and was found to correspond with the formula $C_2HNa$. This compound may be obtained from compressed gaseous acetylene instead of the liquid, but the rate at which the reaction proceeds appears to be a function of the pressure. Heat hastens the change, and at 70° C., a small fragment of sodium was converted completely to the compound $C_2HNa$ in 15 days. This compound, when heated in a tube of Bohemian glass in a vacuum, turned brown, and yielded pure acetylene; then, as the heating was continued, the colour became deeper, and a small quantity of liquid hydrocarbons condensed. The heating was discontinued shortly before the glass showed signs of deformation; the solid residue remaining, disengaged acetylene on contact with water, and was the carbide $C_2Na_2$. But if the heating was continued, the evolution of gas ceased, and charcoal remained, while a ring of metallic sodium was formed on the cool upper part of the tube. This decomposition of the carbide by heat explains why it cannot be produced in the electric furnace.

Lithium carbide decomposed at a temperature intermediate between the temperatures of decomposition of sodium carbide and calcium carbide. Calcium carbide was decomposed by a powerful current; thus, when a small quantity was prepared by a current of 1,200 ampères and 60 volts, a residue of graphite containing only traces of carbide was left at the end of 10 minutes. Condensation of calcium was observed, but not of the carbide. Magnesium carbide obtained by warming magnesium dust in a current of acetylene, on exposure to a current of 600 ampères and 60 volts for six minutes, yielded a black substance which did not evolve gas on contact with water. The decomposition of the carbide at this temperature explains why it is not formed when magnesia is melted in carbon crucibles.

The experiments showed that the intermediate compounds of sodium and potassium, *viz.*, $C_2KH$ and $C_2NaH$. are formed by the action of gaseous or liquid acetylene with or without pressure, and that on a rise of temperature they decompose, yielding acetylene and the carbides $C_2K_2$ and $C_2Na_2$. A further rise of temperature dissociates these carbides into the metals and carbon. Similar dissociation is observed, but at a higher temperature, with the carbides of magnesium and of the metals of the alkaline earths. It was proved that the carbides of potassium, sodium, and magnesium could not be produced at the temperature of the electric furnace.—J. A. B.

### *Acetylene Gas, The Analysis of.* W. Hempel and L. Kahl. Zeits. f. angew. Chem. 1898, [3], 53—55.

Phosphuretted hydrogen is the chief impurity, besides atmospheric oxygen and nitrogen, in acetylene obtained from calcium carbide. The alkalinity of the other products of the decomposition of the carbide precludes the possibility of the presence of sulphuretted hydrogen in the acetylene. The small quantities of sulphur found by some observers in acetylene probably were in organic compounds. Lunge proposed (Zeits. f. angew. Chem. 1897, 651; this Journal, 1897, 1046) to determine phosphuretted hydrogen in acetylene by passing the gas as evolved through solution of sodium hypochlorite, and then determining gravimetrically the phosphoric acid formed; but this method necessarily is tedious, and the authors have endeavoured to find a volumetric one.

The absorption effected by 1 c.c. of various reagents on 100 c.c. of both phosphuretted hydrogen and acetylene was observed. A solution of 15·6 grms. of copper sulphate ($CuSO_4$, $5H_2O$) in 100 c.c. of water, to which were added 5 c.c. of dilute sulphuric acid (1 vol. of concentrated acid to 4 vols. of water), was found to be the best absorbent for the separation of phosphuretted hydrogen and acetylene. 1 c.c. of the solution absorbed 8·8 c.c. of the former and 0·2 c.c. of the latter from 100 c.c. of each gas. It was found that in the presence of mercury and excess of the solution of copper sulphate, the diminution of volume resulting from the absorption of phosphuretted hydrogen from acetylene, exactly corresponded to four times the volume of phosphuretted hydrogen present. The results were different in the absence of mercury, though the solution absorbed exactly the same quantity of acetylene free from phosphuretted hydrogen whether mercury was present or not. Phosphuretted hydrogen produced by heating an alcoholic solution of caustic potash with ordinary phosphorus, was mixed in various known proportions with nitrogen, and the phosphuretted hydrogen determined by means of the copper sulphate solution. The percentage of phosphuretted hydrogen in the gas produced from the alcoholic potash and phosphorus was from 50·0 to 57·6, according to a number of tests. The differences were caused by the ready decomposition of the phosphuretted hydrogen by the oxygen dissolved in the water present, under the influence of light. The burette had to be kept moist in order to avoid errors through the tension of aqueous vapour. The mean value was 52·4 per cent., and mixtures of known volumes of this phosphuretted hydrogen and acetylene were examined by agitating the mixture for three minutes, in a pipette sealed with mercury, with 3 c.c. of the copper sulphate solution. It was assumed that the phosphuretted hydrogen amounted to one-fourth of the diminution of volume. The following results were obtained:—

*Percentage of Phosphuretted Hydrogen in Acetylene.*

| | | | | | | |
|---|---|---|---|---|---|---|
| Present........ | 2·9 | 3·9 | 1·20 | 2·70 | 1·67 | 2·82 |
| Found......... | 2·8 | 3·4 | 1·15 | 2·65 | 2·00 | 2·75 |

—J. A. B.

### *Benzene as an Agent for Denaturing Alcohol for Illuminating Purposes.* Zeits. für Spiritusind. 1898, **21**, [6], 55—56.

The necessity of another denaturing agent to be added to alcohol used for illuminating purposes is pointed out, if alcohol as an illuminant comes into general use, since the employment of pyridine bases in this respect is limited on account of the decrease in their production. Benzene appears to be suitable, as it is soluble to a considerable extent in alcohol of 86 per cent. (by volume) strength. Although the benzene can for the greater part be separated by diluting the mixture with water, yet the alcohol retains the taste and smell of benzene sufficient to render it useless for drinking purposes. The two cannot be separated by distillation on account of the close proximity of their respective boiling points. In order to ensure success in denaturation, Krämer suggests the addition of 0·1 per cent. of pyridine as well as benzene to the alcohol, the quantity of pyridine at present used being 0·5 per cent.

Some experiments with the Phœbus lamp carried out by Krämer show that 5 per cent. of benzene added to alcohol causes no alteration in the intensity of the light, whilst the lamp burns with a steady and non-smoky flame. An addition of benzene brings about a diminution in the amount of alcohol burnt; this is, however, unimportant in quantities less than 5 per cent.- J. L. B.

### *Gas Liquor, Distillation of, for the Production of Liquid Ammonia.* O. Pfeiffer. J. für Gasbeleuchtung, **41**, [5], 69—72.

Gas liquor resulting from the hydraulic main, condensers, and scrubbers is an aqueous solution of ammoniacal salts containing the following substances:—Ammonium carbonate, sulphide, sulphate, thiosulphate, chloride, thiocyanate, and ferrocyanide. Ammonium carbonate and sulphide constitute about nine-tenths of the total salts. Ammonium cyanide, the existence of which in gas liquor is mentioned in all text-books, could not be detected. According to the character of these salts, the terms "fixed" and "volatile" ammonia form an important distinction in the practical working up of gas liquor. The latter, forming the compounds of ammonia with carbonic anhydride and hydrogen sulphide, can be removed from their solutions by boiling, whilst the salts in which the ammonia is fixed, comprising about 10 per cent. of the total, can only be freed from their ammonia by heating in the presence of lime. Moreover, when making liquid ammonia, directly from gas liquor, sufficient lime must be added from the commencement to retain carbonic anhydride and hydrogen sulphide. This addition forms a weak point in the manufacture, entailing much trouble in consequence of crusts of lime forming on the walls of the stills. The amount of lime should therefore be limited as much as possible. It is customary to estimate the total ammonia and add the equivalent quantity of lime ($17NH_3$ : 28CaO), it being assumed that the ammonia is present wholly in the form of primary salts. The author, however, shows that this is not the case, a large proportion existing as bicarbonate, which requires the use of more lime. In all modern types of apparatus, the gas liquor is subjected to a preliminary heating process having for its object the removal of a large proportion of the carbonic anhydride and hydrogen sulphide with simultaneous enrichment of ammonia before the introduction of lime. It is shown that during this operation the liberation of acid gases is considerably more than the quantity represented by the mere conversion of ammoniacal salts from secondary into primary compounds, so that eventually a large excess of free ammonia is present, the separation of which can be effected without the use of lime. The saving thus effected is considerable, as the following figures indicate :—

| —— | Gas Liquor sampled May 5th, 1897. | | Gas Liquor sampled May 16th, 1897. | |
|---|---|---|---|---|
| | Ordinary. | Heated. | Ordinary. | Heated. |
| Ammonia, total | 2·68 per cent. | 1·97 per cent. | 2·23 per cent. | 2·10 per cent. |
| „ volatile | 2·38 „ | 1·75 „ | 2·10 „ | 1·92 „ |
| Carbonic anhydride | 2·20 „ | 0·52 „ | 2·19 „ | 0·79 „ |
| Hydrogen sulphide | 0·15 „ | 0·06 „ | 0·186 „ | 0·113 „ |
| 1 cb. m. of gas liquor requires lime containing 74 per cent. of CaO. | 47·9 kilos. | 15·2 kilos. | 46·9 kilos. | 20·1 kilos. |
| Lime required for 1 per cent. of $NH_3$ in gas liquor | 17·9 „ | 7·7 „ | 20·1 „ | 9·6 „ |
| Saving in lime through heating | .. | 57·0 per cent. | .. | 52·2 per cent. |

The composition of heated gas liquor is given in the following table :—

| Heated Gas Liquor sampled October 11th, 1897. | a. | b. | Average. | Calculated into Ammonium Salts. | Fixed Ammonia. | |
|---|---|---|---|---|---|---|
| Ammonia, total | 1·931 | 1·945 | 1·935 | .. .. .. | .. .. .. | |
| „ volatile | 1·775 | 1·775 | 1·775 | .. .. .. | .. .. .. | |
| Carbon dioxide | 0·583 | 0·592 | 0·588 | 1·293 $(NH_4)_2CO_2$ | 0·454 | |
| Hydrogen sulphide | 0·580 | 0·055 | 0·056 | 0·086 $NH_4HS$ | 0·030 | |
| Thiosulphuric acid | 0·098 | .. | 0·098 | 0·130 $(NH_4)_2S_2O_3$ | 0·030 | |
| Sulphuric acid | 0·046 | 0·055 | 0·050 | 0·082 $(NH_4)_2SO_4$ | 0·021 | 0·612 |
| Hydrochloric acid | 0·122 | 0·115 | 0·118 | 0·178 $NH_4Cl$ | 0·057 | |
| Thiocyanic acid | 0·131 | 0·133 | 0·132 | 0·174 $NH_4CSN$ | 0·039 | |
| Ferrocyanic acid | 0·014 | 0·019 | 0·016 | 0·034 $(NH_4)_4FeCy_6$ | 0·011 | |

These figures are based on the analytical methods described by Dyson (this Journal, 1884, 688). For the estimation of thiocyanates, a colorimetric method was used, depending on the coloration imparted to gas liquor by a ferric salt and hydrochloric acid as compared with a similar coloration obtained with standard solutions of ammonium thiocyanate and ferric chloride. The free ammonia in the above table is equal to 66·8 per cent. of the total. The quantity of lime required for 1 cb. m. of gas liquor for the complete removal of ammonia amounts to 14·3 kilos. (74 per cent. of CaO) if based upon the fixed ammonia, or 43·1 kilos. if calculated from the total ammonia. To ascertain the requisite amount of lime, a measured quantity of milk of lime of known percentage is boiled with heated gas liquor until the ammonia has been entirely removed, after which the excess is titrated back with normal hydrochloric acid and phenolphthalein.—D. B.

## PATENTS.

*Bye-Product Coke-Ovens, Impts. in.* H. Simon, Manchester. Eng. Pat. 1194, Jan. 16, 1897.

Simon-Carves and like coke-ovens, such as that described in Eng. Pat. 554, 1893 (this Journal, 1884, 507), have, according to this invention, their air supply heated by passage through or along the outside of a metallic tube or tubes which are contained within a conduit or channel, or a number of conduits or channels, through which the hot gases are caused to travel in an opposite direction on their way to the chimney. Or the air may pass through the conduits, and the hot gases through the tubes.—R. S.

*Coke, Impts. in or relating to the Manufacture of.* R. Dixon, Crook. Eng. Pat. 8007, March 29, 1897.

Relates to the heating of the retort ovens employed in making coke, and consists in leading the waste gases discharged from beehive ovens, or hot waste gases from other sources, as from blast furnaces, to the underflues of the retort ovens, whereby the latter are heated without the expense of recuperators.—R. S.

*Burning of Pulverulent Fuel, and Apparatus therefor: Impts. relating to the.* A. Freitag, Amsterdam. Eng. Pat. 3129, Feb. 5, 1897.

The pulverulent fuel is discharged from a hopper through a valve in its base into a tube, into which air, which may have been previously heated, is forced. The air and fuel then pass into a casing containing rapidly rotating beaters, from which, after a thorough mixing, they pass through fine apertures in the wall thereof into the furnace or fire tube, where they are burned.—R. S.

*Furnaces, Impts. in or connected with.* W. Brothers, Rawtenstall. Eng. Pat. 4646, Feb. 20, 1897.

Improvements in the furnaces of the kind described in Eng. Pat. 3724, 1897, are claimed, the object being to render that invention suitable for heating kilns, ovens, stills, retorts, saltpans, and chemical and other furnaces, other than steam generators. This furnace is provided with an interior furnace chamber or chambers to receive the fuel, and with an air chamber around and on the outside of the furnace chamber, with which it communicates by passages. The air chamber is provided with regulator valves for the admission of air, and communicates with a combustion chamber arranged behind the furnace.—R. S.

*Fuel, Impts. in.* W. P. Thompson, Liverpool, &c. (From The Compressed Coal Co., New York.) Eng. Pat. 29,605, Dec. 14, 1897.

The production of a mixed fuel which will be impervious to water, will not break up in handling and transporting, or whilst burning, and which will burn freely without smoke. Briquettes are formed of finely-divided coal and some binding material which will not absorb moisture, and will supply some oxygen for combustion. Such a material is lime partially slaked in a solution of sulphate of iron and liquid saccharine matter, or, in place of the latter, bichromated gelatin.—R. S.

*Bunsen Burner for Liquid Combustibles, Improved.* R. Adam, Kœnigsberg, Germany. Eng. Pat. 16,733, July 14, 1897.

The inner side of the wick tube, which extends above the wick, is flanged outwards and perforated so that part of the liquid combustible used, is vaporised by the heated metal when the lamp is lighted, the vapour passing into a capped central chamber where it becomes mixed with air, and, under the influence of the natural draught of the lamp, is drawn through the upper perforations and caused to impinge on the flame, which thereby assumes a tulip form. Owing to the saturation of the vapour, the combustion is not complete until the zone of the outer air current, directed upwards to the outside of the flame through suitably arranged caps surrounding the wick ducts, is reached. The flame may be rendered luminous by a mantle suspended over and enclosing it in the ordinary manner.—C. S.

*Oil and other Lamps for Illuminating Purposes, Impts. in.* H. H. Lake. From La Cie. Continentale d'Incandescence et de Chauffage, Brussels. Eng. Pat. 27,233, Nov. 20, 1897.

The aim of the invention is to produce, automatically, a constant pressure of oil during the time the gasifying burner is lighted. This it is proposed to effect by heating charcoal in an enclosed chamber above the burner, and conducting the air thereby evolved into the oil reservoir, where its pressure forces part of the oil up into the burner and another part into a second receiver at a higher level. The burner is then lighted and the heat therefrom keeps the temperature of the charcoal chamber constant, and thus ensures constancy of pressure of the oil so long as any remains in the upper chamber. To extinguish the flame, the air pressure is relieved by opening a valve in the air tube. The charcoal, in cooling down, re-absorbs air, and is then ready for use again.—C. S.

*Incandescence, Impts. in Lamps for use with Oil or Spirit and Operating by.* E. E. Abrahamson, London. Eng. Pat. 2765, Feb. 2, 1897.

There is claimed "in a lamp for use with oil or spirit and operating by incandescence, wherein a vaporising chamber is disposed above the fuel reservoir and connected therewith by means of a capillary wick-tube, the employment of a small capillary wick-tube or holder closed at its outer extremity and provided with a perforation or perforations through which inflammable vapour issues in sufficient quantity to support a flame, whereby the vaporising chamber is heated and the oil or spirit rendered gaseous." The vapours burn in a Bunsen burner within a mantle as usual.—H. B.

*Incandescent Lighting Purposes; An Improved Fluid and Process of Manufacturing same for Impregnating Bodies or suitable Fabrics, Threads, Fibres, or the like, for.* B. Puchmüller, Schoeneberg, Germany. Eng. Pat. 3811, Feb. 12, 1897.

The essential constituents of the improved fluid are suitable soluble salts (nitrates, &c.) of zirconium and strontium. To the solution of these is then added a solution containing magnesium nitrate, oxide of antimony (dissolved in tartaric acid solution), uranium nitrate, chromic acid, and silver nitrate, and in addition (*a*) for yellowish-white light—ammonium tungstate and vanadate; (*b*) for greenish-yellow light—ammonium molybdate; (*c*) for reddish-yellow light—platinum chloride, ammonium tungstate and vanadate. These are to be added to the zirconium-strontium solution only to the extent of "milligrammes and fractions of milligrammes" per mantle.—H. B.

*Incandescent Lighting Purposes; An Improved Fluid, and Process of Manufacturing same, for Impregnating Bodies or Suitable Fabrics, Threads, Fibres, or the like, for.* B. Puchmüller, Schoeneberg, Germany. Eng. Pat. 49, Jan. 1, 1898.

(See preceding abstract.) The claim is: "In the manufacture of a fluid . . . for incandescent lighting purposes, by the process set forth in my previous patent No. 3811/97, the addition to the oxides or nitrates of strontium and zirconium and the light-giving oxides in the said process, of the oxides or nitrates of barium and aluminium."—H. B.

*Incandescence Lighting, Impts. in Oil Lamps for.* J. Moeller, London. Eng. Pat. 6159, March 9, 1897.

The lamp to which the improvements are applicable, is of the ordinary Argand form, having a circular wick surmounted by a concentric deflecting disc and surrounded by a conoidal shield, to direct an air-current externally against the wick. A metal ring is fixed externally to the top of the outer wick tube, and inside the inner wick tube there is fixed a metal ring, the upper edge of which projects a little above the wick-tube. These rings thicken the upper parts of the tubes "and retain local heat," and the projecting lip of the inner ring forms a small annular recess, which retains oil overflowing from the wick till it is vaporised. The conoidal shield is extended downwards in the form of a trumpet-mouth, and is fitted at its base with air-inlet gratings. The cone carries the chimney, mantle support, &c. The combination of these parts in an Argand oil lamp is claimed.
—H. B.

*Incandescence Gas Lighting, An Impt. in Bunsen Burners for.* J. Moeller, London. Eng. Pat. 7010, March 17, 1897.

Over the burner-tube of a Bunsen burner of the usual laboratory type is inverted a deep, thimble-shaped cup of refractory material, so that the gaseous mixture is deflected at the top of the tube and descends the annular space surrounding the tube towards the closed mouth of the cup at the bottom, issuing by lateral orifices, so as to form an annular flame surrounding the cup. The mantle surrounds the latter, but is separated from it by a small annular space. The refractory cup imparts heat to the gaseous mixture descending within it. The burner as described, is claimed.
—H. B.

*Incandescence Mantles, A Process for Stiffening.* R. Schoder [R. Schröder], Streatham, S.W. Eng. Pat. 7330, March 20, 1897.

The burnt mantle is dipped into pure paper-pulp (such as that of chemical filter-paper) suspended in water to which has been added a little less than 1 per cent. of nitric acid. On withdrawing the mantle and drying it, a stiffening coat of cellulose remains. Instead of water, a volatile liquid, such as alcohol, is preferable. The water may contain about 1 per cent. of starch, or gelatin, and glycerin; and the alcohol may contain a little shellac.—H. B.

*Incandescent Lighting and for Heating, A Petroleum Burner for.* M. Bernstein, Berlin. Eng. Pat. 25,057, Oct. 29, 1897.

The lamp is of the usual circular-wick type. In the interior of the upper part of the wick-tube is fastened a tube, variously notched or perforated, terminating at its upper end in a perforated cap, and from which, at its junction with the cap, a perforated flange projects immediately over the top of the wick. The notches or perforations divide the central air-current and cause it to whirl, and the perforated cap preheats it. The mouth of the wick-tube and the perforated cap are surrounded by a cylindrical jacket, over which the lower edge of the mantle slips. The jacket may consist of a number of small vertical tubes arranged in a circle, which are kept together at the top and bottom by hollow horizontal rings, all the tubes being perforated; or it may simply consist of a perforated metal shield. It serves to heat the outer air-supply.—H. B.

*Calcium Carbide, A New and Improved Receptacle for Safely Storing.* F. S. Thorn, North Brixton, and C. Hoddle, North Brixton. Eng. Pat. 2261, Jan. 28, 1897.

This receptacle consists of a vessel partly covered by a dome-shaped top, in the centre of which is an aperture for charging or withdrawing the carbide, and which has two upstanding concentric rims, between which may be placed any suitable luting. The lid or cover is similarly provided with two concentric rims, which fit respectively over the outer rim and between the two rims on the vessel.—R. S.

*Calcium Carbide, An Improved Method of and Apparatus for the Production of, applicable also to other purposes.* [*Incandescent Core.*] H. Maxim, London. Eng. Pat. 2894, Feb 3, 1897.

This electric furnace consists of a rectangular chamber containing electrodes of less diameter than either dimension of the chamber, and provided with a vertical shaft having a diameter approximate to the diameter of the electrodes, and through which the carbide forming material descends in a column of less diameter than either dimension of the furnace chamber. Between the electrodes is a conducting core formed of a carbon cylinder filled with calcium carbide, and it is the incandescent heat of this molten conductor which converts the material fed into the furnace into carbide, which is drawn off through one of the electrodes as it rises above a certain level. The raw material, consisting of bituminous coal or other carbonaceous substance and lime, is pulverised and compressed into corrugated blocks, and fed into the shaft of the furnace, and the gases given off during its combustion can be utilised for giving a further heating to fresh material, or for other purposes.—G. H. R.

*Calcium Carbide [Treatment with Oil], Impts. in the Treatment of.* F. Bosca, Turin (at present of London). Eng. Pat. 4675, Feb. 20, 1897.

Calcium carbide, after it has been made by any process and allowed to cool, is caused to absorb any fatty or oily substance, as by immersing it for a short time in lubricating oils, varnish, pure or diluted tar, &c. It is then dried and is afterwards unalterable in air, and also decomposes slowly and regularly in water.—R. S.

*Gas, Atmospheric, from Liquid Hydrocarbons; Apparatus for Producing.* P. B. Watson, Falkirk, N.B. Eng. Pat. 2738, Feb. 2, 1897.

The apparatus consists of a scrubber wherein air is carburetted with liquid hydrocarbons, the supply of air being controlled by passage through a meter, the spindle of which actuates an oil elevator with adjustable cups. In this manner the proportions of oil and air can be regulated as desired, so as to enable mixtures burning with either a heating or illuminating flame to be produced.—C. S.

*Gas for Burning and Illuminating Purposes, An Improved Process for Producing.* K. Stattler and A. Strejz, Vienna, Austria. Eng. Pat. 1128, Feb. 18, 1897.

This invention relates to a process for mixing calcium carbide with substances or mixtures liberating carbon dioxide when brought into contact with water, such as sodium bicarbonate and tartaric acid, the object being to dilute the acetylene gas, so that it may burn with a brilliant flame without the formation of smoke and soot. By this means the danger of explosion is also said to be less than with pure acetylene.—D. B.

*Gas Furnaces and the Burners for Heating them, Impts. in.* J. H. Gartrell, Penzance. Eng. Pat. 5473, March 2, 1897.

This invention relates more particularly to nickel muffle furnaces for firing porcelain for dental purposes. The furnace is made in two parts of an iron casing, lined with fireclay. The upper part opens on a hinge, so that the muffle, held between the two parts, can be readily removed and replaced. It is made of sheet nickel, alloyed with about two or three per cent. of manganese, pressed to the shape of a flattened tube. The novelty of the burner is the construction of the gas valve for instantly changing the flame from a Bunsen burner to a blast produced by air under pressure. The Bunsen flame is used first to dry and heat the material placed in the muffle, and then by a turn of the gas-valve the burner is instantly changed to a blast which strongly heats the material.—D. B.

*Water-Gas, Carburetted, Impts. in Apparatus for the Manufacture of.* L. L. Merrifield, London. Eng. Pat. 7013, March 17, 1897.

The object of this invention is to render impossible the escape of gas, due to lack of water in the seal box, either from evaporation or from some of it being driven out by excessive gas pressure, more particularly when the apparatus is not at work or when it is blowing up for a heat. For this purpose the end of the dip pipe in the seal box (between the take-off pipe of the superheater and the pipe leading to the oil heater, condenser, and scrubber) is fitted with a valve, which is connected with the stack valve by means of intermediate gear, so that when the latter is open the seal valve is closed, and *vice versâ*.—D. B.

*Petroleum, Ligroin, Spirit, or the like; Improved Apparatus for the Vaporisation of.* C. A. Green, Stockholm. Eng. Pat. 9277, April 12, 1897.

The vaporising chamber consists of an annular tube, with either a slot or a number of apertures in its upper surface, and surmounted by a metal crown—consisting of radial pieces of sheet metal, rods projecting through the apertures in the annulus, &c.—which transmits heat from the burning gas to the vaporising chamber, and so gasifies the contained liquid.—C. S.

*Air, Carburetting; An Improved Method of and Apparatus for.* F. I. Lothammer, Brussels. Eng. Pat. 19,889, Aug. 28, 1897.

The method relates to what is generally known as cold-air carburisation, and is characterised by the automatic nature of all the successive carburetting operations, and also by the facts that the air at all times enters the carburetter at a constant pressure and that the feeding of the same takes place on a level, which is also at all times uniform in proportion as the gas or carburetted air is consumed. The air distributor consists of a number of pipes of variable lengths terminating in bell-mouth or trumpet-shaped enlargements arranged for the dispersion of air in the state of infinitesimally small bubbles. By this means a very complete carburisation is secured, and consequently gas of a more permanent character is stated to be obtained.—D. B.

*Gas, The Manufacture of; Impts. in Apparatus for.* A. H. Poppel, Hamburg, Germany. Eng. Pat. 25,866, Nov. 6, 1897.

An apparatus is described for generating gas by a cold process in which only the quantity of gas is produced which is required for a certain consumption for illuminating or heating purposes. The gas, produced by forcing air into a carburetter of special construction and partly filled with gasoline, is caused, before it reaches the place of consumption, to pass through a gasholder consisting of two chambers separated from each other by a partition, its object being to regulate the generation of gas in accordance with the consumption. The gas collects in the lower chamber until a certain pressure is reached, and then passes a check valve (which is covered over by a net or strainer) into the upper chamber, which takes up only as much gas as is required at the respective place of consumption. The carburetter is provided with transverse partitions, which fit closely to the right and left side walls of the vessel alternately, and terminate at their other ends at short distances from the corresponding walls of the vessel, so that air forced in is caused to flow in a zigzag course through the carburetter over the surface of the gasoline.—D. B.

*Gas, The Manufacture of; Impts. in and relating to.* W. H. and G. E. Russell, New Jersey, U.S.A. Eng. Pat. 29,231, Dec. 10, 1897.

The present invention has for its object the decomposition of water by electrolysis and the carburisation of the resulting gases for the production of a cheap gas of low specific gravity and high illuminating power. The gas produced is not more dangerous than ordinary coal-gas. The apparatus employed is well adapted for small private plants for dwelling-houses, factories, and the like. The method consists in generating gas in a continuous manner from acidified water by electrolysis and chemical reaction, whereby heat is produced in the generating apparatus, and simultaneously and continuously forcing carburetted air into the hot generator, whereby the gases are combined and fixed under the influence of the heat, and finally recarburetting the mixed gases before they pass to the burners. —D. B.

*Petroleum, Vaporised; Impts. in Apparatus for Burning.* D. Kempe, Nacka, Sweden. Eng. Pat. 29,421, Dec. 11, 1897.

To ensure automatic regulation of the pressure, forcing the supply of oil to the vaporising burner, the inventor employs liquefied carbon dioxide, which has the additional advantage of occupying but a small space. When the pressure in the oil reservoir falls below the normal, the valve of the carbon dioxide bottle opens automatically and readjusts it to its proper level.—C. S.

*Acetylene Gas, Apparatus for the Automatic Generation of.* E. G. Villejean and H. J. J. Frossard, both of Paris. Eng. Pat. 2554, Jan. 30, 1897.

The supply of water to carbide in a generating chamber takes place from a superposed reservoir through a capillary tube of dimensions calculated to govern the water supply so that the pressure of the generated gas is kept constant. At the lower end of the capillary tube is a hollow chamber containing a ball float, which closes the inlet to the chamber and stops the admission of water when the pressure of the gas equals that of the column of water in the tube. A capillary nozzle attached to the valve chamber allows the water to trickle on to the carbide.—J. A. B.

*Acetylene, Impts. in or relating to Means for Producing and Burning.* E. Gossart and H. Chevallier, both of Fampoux, France. Eng. Pat. 4424, Feb. 18, 1897. (Under Internat. Convention.)

The improvements refer to the apparatus claimed in Eng. Pat. 27,574 of 1896 (this Journal, 1897, 1007). Lime of a higher degree of hydration than $CaO, H_2O$, is stated to act upon carbide, and the aim of the invention is to prevent the formation of superhydrated lime. To this end the carbide in the generator is placed in layers upon superposed sieves, and the water drops from a capillary tube into a funnel, which discharges it on a distributing cone above the bottom layer of carbide. The gas evolved passes through the sieves of carbide, and any moisture condensed in the coiled exit pipe is splayed by a distributing cone on to the upper layers of carbide. The exterior of the generator is coated dead black to favour dispersion of heat. —J. A. B.

*Acetylene Gas, Impts. in Lamps for Generating and Burning.* W. H. Wheatley, London. From C. W. Beck, Chicago, U.S.A. Eng. Pat. 22,850, Oct. 5, 1897.

A receptacle for carbide has set centrally within it a water-distributing chamber in free communication with an overhead tank. The wall of the distributing chamber has some perforations, which communicate with perforations in a concentric sleeve within the chamber when the sleeve is in certain positions. The sleeve is rotated by a ratchet device, to bring one or more perforations in communication, or is lifted automatically by a diaphragm when the pressure of the gas in the apparatus is excessive and overcomes the resistance of a spring on the diaphragm. When thus lifted no perforations are in communication, and the supply of water to the carbide is arrested. An elastic relief reservoir beneath the generator receives the gas generated after the supply of water is arrested. The gas passes from the generator through a reducing valve of special type to a burner above the water tank. 22 claims are made. —J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* J. McConechy, Glasgow. Eng. Pat. 24,301, Oct. 21, 1897.

Producers, each consisting of a carbide-container surrounded by water, and fed with water from an annular tank containing sufficient water for reacting with the charge of carbide and no more, are connected to a main leading to a bell gasholder. A tap regulates the flow of water to the carbide.—J. A. B.

*Acetylene-Gas, Improved Apparatus for Generating.* J. and J. Mücke, Berlin. Eng. Pat. 26,435, Nov. 12, 1897.

A bell with a dome for the gas-outlet pipe rests in a tank, to which water is supplied by a siphon from a cistern, which is filled through a cock operated by the bell of a gasholder into which the gas passes. Beneath the bell in the tank are carbide receptacles placed at different levels, and having covers with down-turned edges, and inner bells which cover the carbide, and force the gas which is evolved when the water flows into the receptacles, to pass through the milk of lime also formed. A bend or trap on the outlet-pipe receives condensed water.—J. A. B.

*Acetylene Gas, Impts. in or relating to Burners for.* O. Falbe and E. Borchardt, both of Berlin. Eng. Pat. 27,536, Nov. 23, 1897.

To prevent choking of the mouths of burners by deposits, a hood or cap is placed over the burner. Corresponding to each orifice or slit in the burner is a larger orifice or slit in the cap. The gas is said to burn at the larger orifice or slit without choking it; while, in case choking has occurred, the larger orifice in the cap is easily cleansed. —J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* J. O. O'Brien, Manchester. From E. M. Boule, Versailles, France. Eng. Pat. 29,054, Dec. 8, 1897.

A bell surmounted by a hopper for carbide dips into a water tank, the upper part of which is enlarged and divided into two concentric annular chambers. The valve regulating the discharge of carbide into the water in the tank is operated through rods by an annular float which rises and falls with the level of the water outside the bell. The level of the water depends on the pressure of the gas confined in the bell; in the event of the pressure being excessive, water is forced into the outer annular chamber, whence it returns later through a siphon.—J. A. B.

*Acetylene Gas Lamp, An Improved.* F. J. Bergmann, Neheim-on-the-Ruhr, Germany. Eng. Pat. 29,258, Dec. 10, 1897.

A central container for carbide is removable through the base of the lamp, the body of which is in two superposed compartments in communication through a dip pipe. The bottom of the carbide container is in the form of an inverted cone, and a ball closes an orifice in it, or when the pressure of the water in the body of the lamp is greater than the pressure within the container, the ball lifts and allows water to pass into the carbide and generate gas. Surplus gas is stored in the lower compartment, from which it forces water to the upper.—J. A. B.

*Acetylene Gas, An Improved Generator for.* J. St. C. Legge and A. S. Cooper, both of Dublin. Eng. Pat. 30,690, Dec. 28, 1897.

A holder for carbide is placed in one end of a cylindrical casing containing water, which is admitted to the carbide through a perforated pipe opened by a cock operated from outside the casing. A gas-exit pipe and safety valve are provided.—J. A. B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Ozokerite Industry of Galicia, The.* Chem. Ind. **21**, [4], 81—82.

According to statistics published in "Nafta," there were, at the close of 1896, 44 ozokerite mines—with 289 shafts in work, 32 in process of sinking, and 413 abandoned shafts—in active work in Galicia, 28 of the mines being at Boryslaw, 8 at Starunia, and 6 at Dzwiniacz, the whole producing 72,100 (metric) cwt. per annum. Private advices from Boryslaw, however, estimate the production at 830 railway truck-loads instead of 72,100 cwt. Of late years the depth of the shafts has been increased from a range of 27—176 metres in 1892 to 74—214 metres in 1896.

Since the Länderbank acquired a controlling interest in the majority of the Boryslaw mines, great improvements in the methods of working have been introduced, and the application of electricity for haulage and winding has resulted in an increased output, two men being now able to raise 120 skips of 135 kilos. per diem of 10 hours, whereas formerly four men were required to raise 60 skips of 50 kilos. each in the same time. It is expected that the installation will pay for itself in 12 months.

The barren rock excavated with the ozokerite is freed from small pieces of the latter by washing, and then employed for packing the old workings. If, however—as at Starunia—the rock is impregnated with matter containing paraffin, the latter is extracted by the aid of boiling water. At Dwiniacz, in particular, a quantity of oil collects in the workings, and is drained into the shaft sump, whence it is raised by pumping in large works or by hand labour in small mines. In some properties, boreholes are sunk for oil, but if a stratum of ozokerite is struck, the winning of the latter is proceeded with as the principal object.

The crude ozokerite is worked up for ceresin, paraffin being extracted from the residue, which then forms "ozokerite pitch." In distilling for paraffin, a burning oil comes over at 150° C., the fraction 200°—300° C. consisting of a mixture of heavy oil and paraffin, and being worked up for the latter or for vaseline, whilst the chief yield of paraffin comes from the fraction 300°—350° C. The average yield of the various distillation products is, at Boryslaw:—Light oil (up to 150° C.), 6 per cent.; heavy oil (150°—300° C.), 32 per cent.; paraffin, about 55 per cent.

Whilst the whole of the crude ozokerite is melted on the spot, only a very small portion is further treated there, the bulk being exported for refining. Germany takes about 31 per cent., Austria-Hungary 35·5 per cent., and Russia 27·4 per cent. of the total output. In 1897 the exports totalled 5,743 truck-loads, 1,796 of which went to Germany.

The manufactured ozokerite products are classified into: Normal (m.p. 65° C.), high-prime, special high-prime—50 per cent. of the total—seconds, and hard. The distinctive characteristic of Galician ozokerite is its high paraffin-content and low percentage of resinous matter, the latter not exceeding 2—5 per cent.; whereas American, Russian, and Roumanian ozokerite contain as much as 30 per cent.

—C. S.

*Petroleum in Newfoundland.* Eng. and Mining J. 1897, **64**, [26], 759.

It is stated that the petroleum-producing district of Newfoundland extends over an area of about 250 square miles. It consists geologically of a tract of limestone and conglomerates thereof, shales, and sandstones. The crude oil gathered from the surface depressions had the following composition:—Illuminating oil, 14·5 per cent.; lubricating oil, 82·5 per cent.; solid residue, 3 per cent.; sulphur, 0·098 per cent. The oil obtained from a depth of 1,000 ft. was much lighter and yielded a larger proportion of illuminating oil. The crude oil was dark olive green in colour, of aromatic odour, and had a specific gravity of 0·844 at 60° F. It yielded on analysis:—Naphtha, 7 per cent.; illuminating oil, 56 per cent.; lubricating oil, 34 per cent.; coke, 3 per cent. The quality of the oil compares favourably with that of the best American oils. The illuminating oil is water-white, of high flashing point, and burns with a brilliant flame.—A. S.

*Benzene, A Combination of Phosphoric Anhydride with.* H. Giran. Comptes Rend. 1898, **126**, [8], 592.

*See under XXIV., page* 283.

*Ethylsulphuric Acid [for Alcohol] from Coke-Oven Gas, Production of.* P. Fritzsche. Chem. Ind. **21**, [2], 27—34.

In 1894 the author made a series of laboratory experiments on the absorption of ethylene by means of sulphuric acid, which showed that by working under pressure, the absorption is greatly accelerated. He then made some trials on a large scale, the object being to discover, if possible, a process for the removal of ethylene from coke-oven gases by the use of absorption apparatus of comparatively small dimensions. Having found that the absorption can be effected with results nearly approaching theory, a series of estimations was made to determine the amount of ethylene in coke-oven gases from which the benzene had been previously removed. For this purpose, a well-stoppered glass bottle, holding from 6 to 9 litres, was filled with the gas and well shaken for five or six hours with 100 to 120 c.c. of sulphuric acid containing from 93 to 95 per cent. of monohydrate. The mixture was then diluted with water, transferred to a retort, and 80 per cent. of the water distilled off. The distillate was redistilled several times, potash being used in the first and second distillations to retain sulphuric acid. The following results were obtained:—

| No. | Date. | Temperature | Barometer. | Gas used. | Gas used, reduced to 15° C. and 760 mm. | Distillate obtained. | Specific Gravity of Distillate. | Absolute Alcohol in Distillate. | Absolute Alcohol in 1 cb.m. of Gas. | Ethylene found. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | C. | Mm. | C.c. | C.c. | Grms. | | Grms. | Grms. | Per Cent. by Vol. |
| 1 | 6 Nov. 1894 | 19 | 764 | 6,331 | 6,227 | 3·2491 | 0·99250 | 0·13671 | 21·8 | 1·12 |
| 2 | 12 „ „ | 20 | 744 | 6,331 | 6,086 | 3·9480 | 0·99682 | 0·13468 | 22·1 | 1·13 |
| 3 | 29 „ „ | 17 | 765 | 8,613 | 8,606 | 6·7600 | 0·99535 | 0·17170 | 19·9 | 1·02 |
| 4 | 7 Feb. 1895 | 14½ | 757 | 6,250 | 6,225 | 9·6003 | 0·99712 | 0·13256 | 21·3 | 1·10 |
| 5 | 13 „ „ | 14 | 760 | 6,340 | 6,316 | 11·1387 | 0·99723 | 0·16573 | 26·2 | 1·34 |
| 6 | 21 „ „ | 15 | 759 | 6,340 | 6,332 | 12·1340 | 0·99754 | 0·15890 | 25·2 | 1·29 |

The experiments for the absorption of ethylene under pressure were conducted in horizontal cylinders fitted with stirrers similar in construction to the vessels used in the manufacture of mineral water. They were made of stout cast iron, and consisted of two flanged cylinders bolted together, a perforated cast-iron disc separating the cylinders. For every experiment two such vessels, each holding about 100 litres, were used, the pipe for introducing the gas being at the top, near the back end of the cylinder, whilst the outlet pipe was attached to a dome near the front of the vessel. The washing and drying of the gas before use was effected in a pair of cast-iron pots lined with lead and containing sulphuric acid. The compressor was capable of pumping 2·49 litres of gas per stroke, it made about 40 revolutions per minute, and worked at a pressure of from 4 to 5 atmospheres. The following results were obtained:—

| Number. |  | Date. |  |  |  | Percentage of Alcohol in the Acid. |  |  |  |  |  |  |  | Absolute Alcohol obtained. |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Series. | Experiment. | Day. | Hour. | Gas used. Cb. m. | Gas Pressure. Litres per Minute. | Apparatus I. | Apparatus II. | Ethylene in the Gas. Per Cent. by Vol. | Ethylene in the Gas leaving the Apparatus. Per Cent. by Vol. | Ethylene absorbed. Per Cent. by Vol. | Amount absorbed, calculated as $C_2H_5OH$ in Grms. per cb. m. | Amount absorbed, calculated in per Cent. | Number of Revolutions made by Stirrers. | Total Grms. | Grms. per cb. m. |
|  |  |  |  |  |  | By Vol. | By Vol. |  |  |  |  |  |  |  |  |
| I. | 1 | 16/5 | 9.15 | 97·4 | 35·0 | 2·03 | 1·94 | 1·039 | 0·539 | 0·500 | .. | .. | 100 | .. | .. |
|  | 2 | 20/5 | 10.0 | 294·6 | 35·0 | 4·14 | 4·82 | .. | 0·849 | .. | .. | .. | 100 | .. | .. |
|  | 3 | 24/5 | 5.15 | 506·0 | 35·0 | (4·10)? | 6·07 | .. | 0·892 | .. | .. | .. | 100 | .. | .. |
|  | 4 | 28/5 | 5.0 | 723·9 | 35·0 | 5·69 | 7·52 | 0·959 | 0·911 | 0·048 | .. | .. | 100 | 940 | 1·1 |
|  |  |  |  |  |  | by weight | by weight |  |  |  |  |  |  |  |  |
| II. | 5 | 26/6 | 5.0 | 98·8 | 16·3 | 3·36 | 2·67 | 0·924 | 0·411 | 0·513 | 10·00 | 55·5 | 150 | .. | .. |
|  | 6 | 1/7 | 3.15 | 204·6 | 15·9 | 4·71 | 4·74 | 1·049 | 0·695 | 0·354 | 6·84 | 33·6 | 150 | .. | .. |
|  | 7 | 6/7 | 3.0 | 298·1 | 14·9 | 6·02 | 6·06 | 1·361 | 1·120 | 0·241 | 5·30 | 20·0 | 150 | .. | .. |
|  | 8 | 12/7 | 9.0 | 394·8 | 14·75 | 6·10 | 8·49 | 1·330 | 1·029 | 0·301 | 5·85 | 23·0 | 150 | 1,706 | 4·3 |
| III. | 9 | 26/7 | 6.0 | 92·47 | 16·06 | 5·68 | 2·91 | 0·852 | 0·133 | 0·719 | 14·00 | 84·3 | 150 | .. | .. |
|  | 10 | 31/7 | 5.35 | 195·65 | 15·69 | 8·83 | 8·13 | 0·981 | 0·400 | 0·581 | 11·30 | 59·1 | 150 | .. | .. |
|  | 11 | 5/8 | 6.0 | 299·45 | 16·04 | 9·32 | 11·85 | 0·899 | 0·709 | 0·190 | 3·70 | 21·1 | 150 | .. | .. |
|  | 12 | 5/8 | 7.45 | .. | .. | .. | .. | .. | 0·594 | 0·305 | 5·50 | 31·4 | 220 | .. | .. |
|  | 13 | 10/8 | 9.0 | 395·15 | 16·03 | 9·39 | 14·80 | 0·849 | 0·566 | 0·283 | 4·90 | 30·2 | 220 | .. | .. |
|  | 14 | 10/8 | 10.30 | .. | .. | .. | .. | .. | 0·672 | 0·177 | 3·10 | 19·1 | 150 | 1,288 | 10·8 |

It will be seen that on attempting to calculate from these data the capacity of the apparatus necessary for the complete removal of ethylene from a certain amount of gas, the dimensions assume large proportions. Even assuming that by perfecting the process of purification and increasing the efficiency of the apparatus, it were possible to completely remove in one minute the ethylene from 16 litres of gas, every cubic metre of gas produced per minute would require an absorption apparatus holding 12·5 cb. m. A plant carbonising 300 tons of coal per day produces at least 50 cubic metres of gas per minute, and although by compressing the gas to eight atmospheres, the absorbing power of the plant is doubled, other difficulties are introduced, more especially the greater want of resistance which iron offers to the action of ethylsulphuric acid. The production of ethylsulphuric acid from gas containing ethylene is, however, more feasible when the above data are brought in harmony with the observations recently made by the author, which showed that temperature materially influences the absorption of ethylene without incurring any loss (increasing it at least 30 times).—D. B.

*Pentanonal, the Aldehyde of Levulinic Acid; Conversion of Sylvan [Methyl Furfuran] into. (Investigations on the Constituents of Beechwood Tar.—I.)* C. Harries. Ber. **31**, [1], 37—47.

The isolation of a substance with the formula $C_5H_6O$ from the low boiling portions of the tar oils obtained by the dry distillation of pinewood (*Pinus Sylvestris*) was effected by Atterberg some time ago; he regarded it as α-methylfurfuran, and named it "Sylvan." Fischer and Laycock (this Journal, 1889, 274), in the course of their investigations on the oils obtained by the distillation of sugar, found dimethylfurfuran and higher methylated furfurans not only in these oils but also in the oils obtained from wood tar. The author has recently isolated the same product from the fraction of beech-tar creosote boiling at 60°—70° C. Its conversion into pentanonal (levulinaldehyde) by treatment with dilute hydrochloric acid in methyl alcohol confirms Atterberg's view regarding the constitution of sylvan. Atterberg's substance had a specific gravity of 0·887, and boiled at 63°·5, whilst the author's sylvan boiled at 65°, and had a specific gravity of 0·827 at 18°.

*Pentanonal*, $C_5H_8O_2 = CH_3.CO.CH_2.CH_2.CHO$, is a colourless, very mobile, strongly refracting liquid. It solidifies at −21°, has a specific gravity of 1·0156 at 16°, is miscible in all proportions with water, alcohol, and ether, and volatilises when boiled with water. It boils at 66° C. under a pressure of 8·5 mm., and at 186°—188° under the ordinary pressure with slight decomposition.—D. B.

*Acetone, Oil of, and Methylethylketone; Manufacture of, from Fleece Washings.* A. and P. Buisine. Comptes Rend. 1898, **126**, [4], 351.

*See under XX., page 266.*

*Camphor Oil, Light, as an adjunct to Petroleum.* L. Gans Chem. Rev. Fett- u. Harz Ind. **4**, [22], 304.

*See under XX., page 268.*

### PATENT.

*Ammonia, An Improved Method of Producing.* C. H. Mehner, Charlottenburg, Germany. Eng. Pat. 2654, Feb. 1, 1897.

A mixture of coal with an alkali, or alkaline earth, or a carbonate of either, is heated in an electric furnace with admission of air, producer gas, or other gaseous mixture containing nitrogen. The cyanide vapours formed, with the other gaseous products, are caused to pass below the zone of the electrodes through an air-tight passage into a close receiver charged with coal or coke, in which the eyanides condense. When the charge in the furnace is exhausted, steam is admitted to the receiver to decompose the cyanides, with production of ammonia and re-formation of alkaline or alkaline earth carbonate. The receiver is arranged above the furnace, so that its contents may be discharged into the latter on drawing a slide, and the alkali is thus made to serve continuously in the production of cyanides and thence of ammonia; but it is preferred to leave a portion of the regenerated charge of coal, treated as described, in the receiver, which is protected against cooling by radiation, and to add fresh coal at the top of the receiver. The air or producer gas admitted to the furnace, is preferably heated.—E. S.

## IV.—COLOURING MATTERS AND DYES.

*Natural Indigo-Group and the Synthetical Product "Indigo-Pure."* Carl Bucher. Färber Zeit. **9**, [3], 33—35.

The chief constituents of the indigo plant, besides indigotin, are indigo-red, indigo-brown, and indigo-gluten, bodies which bear a close relationship to indigotin. The fastness and tone of an indigo-blue, are more or less influenced by the presence of these bodies.

In combination with "indigo-pure" (the synthetic product), it is preferable to employ soluble albuminoids. These glutinous substitutes (for indigo-gluten) form a cement between the cell-wall (of the cotton-fibre) and the dye, as well as between the individual particles of dyes themselves. If these glutinous substitutes are coagulative bodies, then a colour-lake of great resistance is formed. The author explains this by the close relationship of the albuminoids to indigotin.

The makers also recommend additions of soap and Turkey-red oil, the advantage of which is to cause better absorption of the dye-solution by the fabric.

Indigo-red should not be under-estimated. It has pronounced dyeing properties, and behaves analogously to indigo-blue. It changes the tone of the colour substantially. Hence, when necessary, indigo-red can be substituted for other products.

Indigo-brown is only injurious when free alkalis are used in dyeing. It is particularly objectionable in direct indigo-printing. The indigo-discharge upon goods dyed with "indigo-pure" is much more beautiful, and the white portions are especially clear.—J. E. H.

*Dyestuffs, Classification of.* W. Schaposchnikoff. Chem. Zeit. 1898, **22**, 55—56.

The classification proposed, which is given in the table following, is intended to express both the chemical character of the dyestuff and its relation to the fibre as well as the method of its application; but, as in other classifications, no hard-and-fast rule can be applied to the dyestuffs of the various groups. For instance, whilst the phthaleins dye silk from an acid bath directly, they require a chrome or tannin mordant on cotton.

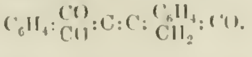
| I. Dyestuffs with Acid Characteristics. | | II. Dyestuffs with Salt-like Characteristics. Substantive Dyestuffs. | III. Dyestuffs with Basic Characteristics. | IV. Indifferent Dyestuffs. | |
|---|---|---|---|---|---|
| *Group* 1. | *Group* 2. | *Group* 3. | *Group* 4. | *Group* 5. | *Group* 6. |
| Dyestuffs Dyeing from an Acid Bath. | Dyestuffs which Dye on Metallic Mordants. | Dyestuffs which Dye from a Neutral or Alkaline Bath. | Dyestuffs which Dye from a Neutral Bath. | Dyestuffs which are formed on the Fibre. | Dyestuffs mechanically attached to the Fibre. |
| 1. Nitro-dyestuffs.<br>2. Most of the azo-dyestuffs.<br>3. Triphenylmethane sulphonic acid, Induline, and Quinoline dye-stuffs, Indigo-carmine.<br>4. Hydrazone dyestuffs.<br>5. Chromotrope. | 6. Some azo-dyestuffs.<br>7. Oxyquinone and quinone oxime dyestuffs.<br>8. Some quinone imide dyestuffs.<br>9. Oxyketone, xanthone, and flavone dyestuffs.<br>10. Phthalein and rosolic acid dyestuffs.<br>11. Natural dyestuffs. | 12. Some natural dyestuffs.*<br>13. Tetrazo dyestuffs.<br>14. Thiazol dyestuffs. | 15. Basic azo-dyestuffs.<br>16. Di- and triphenylmethane dyestuffs.<br>17. Most of the quinone-imide dyestuffs.<br>18. Quinoline and acridine dyestuffs. | 19. Metals.<br>20. Insoluble azo-dyestuffs.<br>21. Mineral colours.<br>22. Indigo and indophenol.<br>23. Aniline black.<br>24. Catechu, Manganese-brown, Canarin. | 25. Albumin colours. |

* Bixine, Curcumine, Carthamine.

See also this Journal, 1897, 137.—T. A. L.

*Anhydrobisdiketohydrindene Dyestuffs.* C. Liebermann. Ber. **30**, [19], 3137—3141.

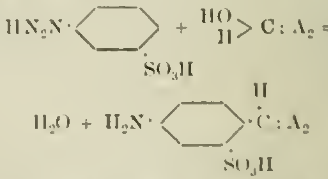
The dyestuffs obtained by the author and Flatau (Ber. **30**, 234; this Journal, 1897, 233) by the action of aromatic amines on cœrulignone do not appear to be of any technical value on account of their slight affinity for the fibre and their instability to acids and reducing agents. The cause of this behaviour is to be found in the quinone structure of the dyestuffs, which will form the subject of a later communication. The present paper describes some derivatives of anhydrobisdiketohydrindene, to the study of which the author was led by a certain similarity between the formula for cœrulignone and the tautomeric form of a condensation product obtained by W. Wislicenus and Kötzle (Ber. **20**, 598) from diketohydrindene, to which they ascribed the formula—


$$C_6H_4{:}\genfrac{}{}{0pt}{}{CO}{CO}{:}C{:}C{:}\genfrac{}{}{0pt}{}{C_6H_4}{CH_2}{:}CO.$$

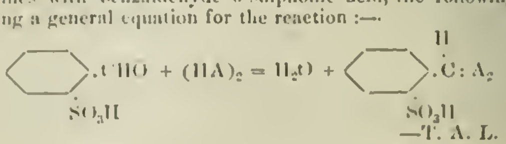
When this compound is boiled for a short time in glacial acetic acid (25 times its weight) with an aromatic amine—for instance, $p$-toluidine ($1\frac{1}{2}$ mols. in 5 parts of acetic acid)—equimolecular proportions condense, with elimination of water, forming with a yield of 80—90 per cent. of the theoretical, a blue dyestuff which can be dissolved in caustic potash, and dyes wool from a faintly alkaline bath, the colour being subsequently developed by passing through weak acid. On boiling an alcoholic solution of the dyestuff with hydrochloric acid, it is decomposed into its components, and in the case of the ψ-cumidine derivative, this was determined quantitatively. The dyestuffs have a less basic character than those of the cœrulignone series, and are not soluble in cold alcoholic hydrochloric acid, but are very easily soluble in cold alcoholic potash with a reddish-yellow colour, and are separated as blue precipitates again by acids. The same dyestuffs are also obtained from diketohydrindene, $C_9H_6O_2$, but the yields are not so good. Presumably the diketohydrindene is first converted into the anhydro compound, which then reacts. Experiments with other members of the diketohydrindene group only gave negative results. Secondary bases, such as diphenylamine, and tertiary, such as quinaldine (free from aniline), do not give the blue reaction with anhydrobisdiketohydrindene, so that the presence of a free amido group is indispensable.—T. A. L.

*Malachite Green Orthosulphonic Acid.* Suais. Bull. Soc. Chim. 1898, [1], 25—27.

The author claims to have embodied in a sealed communication certain reactions which were subsequently patented, and some theoretical considerations which have also been confirmed. By acting with $m$-sulphanilic acid on tetramethyldiamidodiphenylcarbinol, an orthosulphonated leuco base is formed according to the following equation, where A represents $C_6H_4N(CH_3)_2$:—



After removing the amido group, the leuco-base on oxidation gives a dyestuff possessing considerable fastness to caustic alkalis. This, however, is not a property of the hydroxyl derivative, which is reddened by alkalis, although it is orthosulphonated. Another fact in connection with orthosulphonated Malachite Green is its solubility in water in presence of alkalis without decolorising and the constitution of these dyestuffs is unknown, although there is no doubt as to that of the leuco bases, which can also be obtained, as Sandmeyer has shown, by condensing tertiary aromatic amines with benzaldehyde $o$-sulphonic acid, the following being a general equation for the reaction:—



—T. A. L.

*Podophyllum, A Chemical Examination of the Constituents of Indian and American.* Wyndham R. Dunstan and T. A. Henry. Proc. Chem. Soc. 1898, [189], 42—43.

*See under* XX., *page* 268.

## PATENTS.

*Polyazo Dyes* [*Cotton Blues, Blacks, and Browns*], *Production of New.* W. H. Claus, A. Rée, and L. Marchlewski, Manchester. Eng. Pat. 4369, Feb. 18, 1897.

The dyestuffs described are derivatives of amido-azo compounds obtained from γ-amidonaphthol sulphonic acid or from amidonaphthol disulphonic acid H. The following intermediate compounds are employed :—The amido-azo compounds obtained by combining the diazo compound of γ-amidonaphthol sulphonic acid with the same acid or with amidonaphthol disulphonic acid H, (*a*) in alkaline, (*b*) in acid solution, and (*c*) that obtained by combining diazotised amidonaphthol disulphonic acid H with the same acid or with γ-amidonaphthol sulphonic acid in acid solution. The dyestuffs produced have, amongst others, the following general formulæ :—

H.G.P.G.H
R.P.G.H.H
R.P.G.H.N

where P represents a tetrazotised *p*-diamine; G, γ-amidonaphthol sulphonic acid; H, the disulphonic acid above alluded to; R, an amine or a *m*-diamine; and N, a naphthylamine or naphthol sulphonic acid. The following is an example of the method for obtaining a blackish-blue dye for cotton :—21·2 kilos. of tolidine are converted into the tetrazo compound and combined in an alkaline solution with 60·8 kilos. of γ-naphthol sulphonic acid azo-γ-naphthol sulphonic acid azo-phenylene diamine. The intermediate product is formed at once, and 26 kilos. of γ-amidonaphthol sodium sulphonate are added to the mixture, which, after stirring for some time, is heated to 50° C., the dyestuff being ultimately separated in the usual manner.—T. A. L.

*Amido- or Hydroxytriphenylmethanes* [*Aurin Carboxylates, &c.*], *New Carboxylated Derivatives of.* H. R. Vidal, Paris, France. Eng. Pat. 5535, March 2, 1897.

The patentee claims processes for preparing the mono-, di-, and tricarboxylic acids of trihydroxy- or triamidotriphenylmethane, which consist in heating phenol with hydroxy- or amidobenzoic (ortho and meta) or naphthoic acids in the presence of sulphuric and oxalic acids, or zinc chloride and formic acid. For example, aurin monocarboxylic acid is obtained by employing 1 mol. of salicylic acid and 2 mols. of phenol, the dicarboxylic acid from 2 mols. of salicylic acid and 1 mol. of phenol, and the tricarboxylic acid from 3 mols. of salicylic acid. The purification of the products by dissolving in a solution of sodium metabisulphite and precipitating by an acid is also claimed. The mono- and dicarboxylic acids are bright yellow and furnish bright scarlet dyestuffs; the tricarboxylic acids tend towards a dark red.—A. C. W.

*Acetyl-Anthranilic Acid, Impts. in the Manufacture and Production of.* J. Y. Johnson, London. From The Badische Fabrik, Ludwigshafen, Germany. Eng. Pat. 6475, March 11, 1897.

Acetyl-anthranilic acid can be obtained by oxidising *o*-acettoluide with potassium permanganate in a weak acetic acid solution, but little more than a 30 per cent. yield is obtained. The addition of magnesium sulphate, which removes the alkali formed during the reaction, gives a yield of 75—85 per cent. of the theoretical. About 50 kilos. of *o*-acet-toluide, 103 kilos. of crystallised magnesium sulphate, and 6,000 litres of water are heated on a water-bath in an enamelled vessel provided with an agitator and thermometer, to 75°—80° until a clear solution is obtained, when 146 kilos. of crystallised potassium permanganate are added at once. The temperature rises to 85° C., and is maintained thereat for 1½ hour until the permanganate has completely disappeared. The hot solution is then filtered, and acidified with dilute sulphuric acid, the acetyl-anthranilic acid separating as a colourless precipitate. After filtering off, it is washed with a little water, pressed, and dried at about 70° C.—T. A. L.

*Colouring Matters* [*Blues, Blacks*], *Impts. in the Manufacture of.* H. R. Vidal, Paris, France. Eng. Pat. 6694, March 13, 1897. (Date under Internat. Convent., March 10, 1897.)

The process described consists in heating *p*-sulphanilic acid with *o*-, *m*-, and *p*-amidophenols, with 1.4-amidonaphthol, or with *p* phenylene diamine, in this latter case glycerin being used as an incorporating medium. The *p*-sulphanilic acid may also be replaced by naphthionic acid or *m*-sulphanilic acid, and the *p*-phenylene diamine by 1.4 naphthylene diamine. A mixture of 17·3 kilos. of *p* sulphanilic acid and 11 kilos. of *p*-amidophenol is heated in an oil-bath in an iron pot provided with an agitator to 170°—250° C. for about 2—3 hours, until the melt becomes too stiff to stir, which indicates the completion of the reaction. The dyestuff obtained gives blue shades on cotton from an alkaline bath. Treated in the same way, a mixture of *p*-sulphanilic acid and *o*-amidophenol gives a brown mass soluble to a black in alkalis and dyeing cotton black. A similar colour is obtained from *m*-amidophenol, whilst *p*-phenylene diamine gives a violet.—T. A. L.

*Sulpho Acids of the Naphthalene Series and of Colouring Matters derived therefrom, Impts. in the Manufacture of.* H. H. Lake, London. From Farbwerk Mühlheim, vorm. A. Leonhardt and Co., Mühlheim-on-the-Maine, Germany. Eng. Pat. 7213, March 19, 1897.

According to Eng. Pat. 19,253 of 1895 (this Journal, 1896, 708), the introduction of a further sulphonic acid group in the position 4′ into 1.1′.3-amidonaphthol sulphonic acid leads to the production of a valuable disulphonic acid. The present specification deals with the alkylation of this disulphonic acid. A solution containing 3 kilos. of amidonaphthol disulphonic acid and 0·5 kilo. of sodium carbonate in 15 litres of water, is heated with 10 litres of spirit and 1·5 litre of ethylbromide in an autoclave, for 12 hours, to about 90° C. When cold, the melt is made acid with hydrochloric acid, and the spirit and ethylbromide are distilled off. The alkylated acid crystallises from the concentrated solution. It is more easily soluble than the non-alkylated acid, and is only salted out with difficulty. The aqueous solution, after treatment with nitrous acid, gives a yellow solution, which is not turned violet by adding sodium carbonate. On heating the acid with dilute sulphuric acid to 130° C., the 4′-sulphonic acid group is split off. When combined with various diazo compounds in an alkaline solution, it yields a variety of shades. For instance, diazobenzene gives a deep claret; α-diazonaphthalene, a dark blue; tetrazodiphenyl, a greenish-blue; diazo-azobenzene, a greenish-black; and 2 mols. of *p*-nitro-diazobenzene, a deep black.—T. A. L.

*Safranine Derivatives* [*Blues, Greys*], *Manufacture of New.* O. Imray, London. From the "Farbwerke Höchst," Hoechst-on-the-Maine, Germany. Eng. Pat. 7329, March 20, 1897.

By condensing safranines alkylated in the one nucleus with unsaturated aldehydes or ketones, or their generators, and subsequent oxidation, or by combining the two operations, new dyestuffs are obtained which give blue and grey shades on tannined cotton, fast to alkalis. The non-alkylated safranines under similar treatment do not yield any valuable products. The following examples illustrate the process :—70 kilos. of dimethyl-safranine (from 1 mol. of dimethyl-*p*-phenylene diamine and 2 mols. of aniline), 20 kilos. of nitrobenzene, 60 kilos. of glycerin, and 50 kilos. of concentrated sulphuric acid are heated under an inverted condenser. The melt, which changes during the reaction from green to dark brown, is dissolved in water, and the dyestuff is precipitated with zinc chloride. It dissolves easily in water, with a reddish-grey colour, and in concentrated sulphuric acid with an olive-green colour, whilst it is insoluble in alcohol or ether. The shade produced on tannined cotton is a pure grey, fast to soap and alkali. 50 kilos. of safranine (from 1 mol. of diethyl-*p*-phenylene diamine and 2 mols. of aniline), 100 kilos. of concentrated hydrochloric acid, and 80 kilos. of acetone are heated for several hours. A green solution results, which turns blue on dilution with water. The whole is raised to the boil and oxidised at that temperature with bichromate, the dyestuff being precipitated with zinc chloride. The safranine from dimethyl-*p*-phenylene diamine gives a similar but redder dyestuff. Both products are readily soluble in water and alcohol, and the latter solution exhibits a strong red fluorescence. The aqueous solution of the dyestuff is not precipitated by sodium acetate or ammonia, but by soda lye. Cotton mordanted with tannin is dyed a violet-blue, fast to soap and alkalis.—T. A. L.

*Azosafranine Derivatives, Impts. in the Production of.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 7337, March 20, 1897.

The action of diazosafranines on amines has already been described in Monit. Scient. 1886, 984, and it is mentioned that a claret dyestuff is obtained by combining diazosafranine with solutions of the salts of the dialkylated anilines. The patentees now discover that if the reaction be carried out as in Eng. Pat. 3488 of 1895 (this Journal, 1896, 110) in the presence of acetic acid, intense greenish-blue to dark green dyestuffs result. For example, 7·4 kilos. of tolusafranine are diazotised at 0° C. with 7 kilos. of hydrochloric acid at 20° B. and 1 kilo. of nitrite. To this is then added 20 kilos. of sodium acetate, and immediately afterwards a solution of 3 kilos. of diethylaniline in 300 litres of water and 2·3 kilos. of hydrochloric acid is allowed to run in, with constant agitation. The reaction takes about 24 hours to complete, and after heating to 90° C., the dyestuff is salted out, pressed, and dried. Other safranines may be employed, and the dyestuffs obtained, which are easily soluble in water, dye tannined cotton fast to washing and milling, and also to light. They resist high temperatures, and are said to surpass in this respect all the azo-safranine derivatives hitherto known—a fact which is important in steaming printed goods.—T. A. L.

*Colouring Matters [Anisolines], Impts. in the Manufacture of.* G. B. Ellis, London. From "La Société Chimique des Usines du Rhône anc. G. P. Monnet et Cartier, Lyons, France. Eng. Pat. 8175, March 30, 1897.

The improvement consists in treating the rhodamine with one molecular proportion of caustic potash or other alkali in an alcoholic solution, and then heating this salt with an alkyl halogen at or below the temperature of the water-bath. A solution of 0·7 kilo. of caustic potash in 50 kilos. of alcohol has suspended in it 5 kilos. of diethylrhodamine base; alter adding 5 kilos. of ethyl chloride, the whole is heated in an autoclave at the temperature of the water-bath for two hours or more, the product being finally worked up in the usual way. The ethyl chloride may be replaced by an equivalent amount of ethyl bromide or iodide, and the operation may be conducted in a vessel provided with a reflux condenser.—T. A. L.

*Colouring Matters [Blue], Impts. in the Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken, Elberfeld, Germany. Eng. Pat. 8188, March 30, 1897.

Blue dyestuffs for wool fast to acids, alkalis, and light are obtained, according to this specification, by combining one molecular proportion of the diazo derivative of a 1.1′- or peri-amidonaphthol sulphonic acid with one molecular proportion of a 1.1′-naphthylamine sulphonic acid, in which one hydrogen of the amido group is replaced by a methyl, ethyl, phenyl, tolyl, or xylyl radicle. The diazo compound from 31·9 kilos. of 1.1′.3.3′-amidonaphthol disulphonic acid and 6·9 kilos. of sodium nitrite is filtered off, stirred to a paste with water, and then added with constant agitation to a solution of 30 kilos. of 1.1′-phenylamidonaphthylene sulphonic acid and 40 kilos. of sodium acetate in 1,500 litres of water. The reaction is complete in about four hours, when the dyestuff separates as a dark precipitate with a metallic lustre. It is filter-pressed, dried, and ground, and yields a blue powder which dyes wool from an acid bath, having the valuable properties above mentioned.—T. A. L.

*Safranine Derivatives, Manufacture of New.* O. Imray, London. From The Farbwerke Hoechst, Hoechst-on-the-Maine, Germany. Eng. Pat. 8383, April 1, 1897.

By condensing formaldehyde with asymmetric dimethyl- or diethylsafranine in presence of a strong mineral acid and subsequently oxidising the product, valuable grey dyestuffs are produced very fast to soap and alkali. A mixture of 100 kilos. of asymmetric diethylsafranine, 200 kilos. of concentrated hydrochloric acid, and 100 kilos. of formaldehyde (40 per cent.) is heated on the water-bath until the safranine reaction has disappeared. The greenish-black solution is largely diluted with boiling water and oxidised with 28 kilos. of bichromate dissolved in water. The filtered solution is then precipitated with zinc chloride. The product dissolves in water with a bluish-green colour and dyes tannined cotton a pure neutral grey. The aqueous solution of the dyestuff is not precipitated by sodium acetate, but it is by ammonia, alkalis, or mineral acids.

—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*Threads or Fibres from Silk Worms and other Insects of a Similar Kind, Impts. in Obtaining.* A. Millar, Glasgow. Eng. Pat. 2713, Feb. 2, 1897.

The large intestine is removed from worms when they have obtained their maximum size. These organs are placed in a vertical cylinder, fitted with a piston and having a stop-cock, fitted with a very fine nozzle, at the bottom. The stop-cock extends a short way into the cylinder, the top end is closed, and the circumference is perforated with holes and covered with fine gauze. This forms a filter. The whole is surrounded with a water jacket heated to a suitable temperature. The piston is weighted, and on opening the stop-cock, the gelatinous substance contained in the large intestine, is delivered from the fine nozzle, and falls on to a travelling band, which is about 100 yards long. During its passage along the band, the thread dries, and at the end it can be taken off and wound on to a small drum. The upper surface of the band is covered with a non-adhesive substance, and the whole machine should be worked in a room at 80° F.

The worms may, if desired, be poured first of all into a hydro-extractor; on revolving this, the whole of the gelatin is separated. The gummy substance on the threads is removed by soaking or boiling the hanks in soap and water.—C. M.

*Jute Fibre, Raw: Method of Improving the Colour of.* J. Shearer, Maryfield, Dundee, Scotland. Eng. Pat. 2736, Feb. 2, 1897.

Jute, while being passed through the softening machine or the carding machine, is sprayed or sprinkled with diluted commercial phosphoric acid.

The most convenient strength to use is a mixture of 1 part of acid—that is, an acid containing 50 per cent. of phosphoric acid and 50 per cent. carrying agent, such as mineral shale oil—to 250 parts of water. A 400-lb. bale of raw jute is sprayed with about 64 lb. of this mixture.

—C. M.

*Jute Fibre, Raw: Process for Improving the Colour of.* C. O'Brien and J. Shearer, Maryfield, Dundee. Eng. Pat. 2960, Feb. 4, 1897.

This invention is substantially the same as that described in the preceding specification. Instead of phosphoric acid, one-third per cent. solutions (by weight) of oxalic, sulphuric, or hydrochloric acids, or mixtures of these, may be used.—C. M.

*Cotton, Mercerising, in the Form of Hanks; Process and Apparatus for.* C. D. Abel, London, and J. Kleinewefers Söhne, Crefeld, Prussia. Eng. Pat. 7093, March 18, 1897.

Non-stretched cotton hanks are placed loosely round the circumference of a centrifugal machine made of perforated sheet metal, or in grid form. Two centrifugators are coupled, one to each end of a shaft, the other ends being supported by hollow spindles passing through the centres. The spindles have holes perforated along their length inside the drums. Soda-lye is delivered from an outside reservoir through the spindles, and thence, on rotating the drums, it is forced through the hanks into a metal casing which surrounds the centrifugator.

By employing a three-way cock at the entrance of the spindles into the machine, the hanks may be washed with soda-lye, or water, at will, and, when thoroughly mercerised and washed, they can be dried whilst still on the centrifugator.

It is claimed that by using this process, there is no appreciable shrinkage in the hanks, which may be loosely removed from the drums.—C. M.

*Ramie and other like Fibrous Materials, Improved Process for Decorticating and Degumming.* A. L. C. Nodon and L. A. Bretonneau, Paris. Eng. Pat. 18,728, Aug. 12, 1897.

Stems of ramie, having been split longitudinally, are arranged in layers in a wooden frame so as to represent a hurdle. The stems are slightly separated from one another, and are firmly secured in their places by bolts, which should be of bronze or other suitable metal, iron being avoided.

The frames are then arranged in an enamelled iron vat, a 30—40 per cent. solution of soap is run in at about 90° C., and the solution is gradually raised to boiling by a steam coil. This treatment is continued for 48 hours, when the hurdles are removed, drained, and the fibres then separated by a scraper having a rubber edge. The hurdles are now placed in hot water, then in a 6 per cent. solution either of potash alum, sulphate of alumina, or calcium chloride, and finally washed.

In this manner the ramie is entirely freed from gummy matters, and is also slightly bleached. The yield, after drying, is about 30 per cent. of textile fibres, 55 per cent. of wood, and 15 per cent. of bark.—C. M.

*Cloth, An Improved Fireproof.* W. H. Bancroft and G. Bancroft, King Cross, Halifax, Yorkshire. Eng. Pat. 24,978, Oct. 28, 1897.

Cloth, manufactured preferably with a mule-spun woollen weft and a fine English cross-bred worsted warp, is passed through a bath containing 20 per cent. of tungstate of soda, 10 per cent. of phosphate of soda, and 70 per cent. of water.

It is then calendered in the usual way, and forms, by the above method of manufacture, a cloth which is practically fireproof, has a glazed outer and a soft inner surface.
—C. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Basic Dyestuffs, Developing and Combining, in Conjunction with Diamine Colours.* A. Wöscher. Chem. Zeit. 1898, **22**, 78.

The author applies the method of diazotisation and development on the fibre to basic dyestuffs on a tannin-antimony mordant, and has obtained the following results with a 1½ per cent. magenta dyeing, after treatment with 4 per cent. of nitrite and 10 per cent. of hydrochloric acid (of 20° B.) and subsequent washing. On development, β- and α-naphthol give a brick-red; *m*-tolylene diamine, a fiery yellowish-brown; resorcinol, a golden brown; γ-amido-naphthol sulphonic acid (Fast Blue developer A N), α- and β-naphthylamine, and amido-azo-benzene, a cachou shade; whilst phenol and amidodiphenylamine (Fast Blue developer A D) give a drab. The new colours are said to be very fast to light and washing. The affinity for basic dyestuffs still remains, and the shades obtained are much faster than those produced on substantive or developed colours. Very fine fast brown shades are obtained by dyeing the fabric, previously dyed with a diamine colour, with a basic dyestuff, diazotising, and then developing. Basic dyestuffs can also serve as end components by dyeing first with a diamine colour, then with a basic dyestuff, and subsequently combining with nitrazol or diazotised *p*-nitraniline. Magenta, Chrysoidine, Bismarck Brown, and Phosphine give the best results. The first produces a dark Bordeaux, Bismarck Brown and Chrysoidine a dull brown, and Phosphine a leather-yellow shade. Methylene Blue and New Methylene Blue become more greenish after the treatment, but no actual combination appears to take place.
—T. A. L.

*Dyeing Properties of some Dyestuffs, Influence of the Constitution on.* Albert Liebmann. Chem. Zeit. 1898, **22**, [6], 35—36.

The author prepared a number of azo dyestuffs by combining the diazo compound of *p*-amidosalicylic acid ($NH_2 : OH : CO_2H = 1 : 4 : 5$) with α- and β-naphthol, 1.4′-, 1.1′-, 1.2′-, 1.2-, 2.2′-, 2.3′-, and 2.3-dihydroxynaphthalene, and also substituted *m*-amidobenzoic acid and *p*-amidophenol for the *p*-amidosalicylic acid. The results are embodied in the following table:—

| — | | *p*-Amidosalicylic Acid. | | *m*-Amidobenzoic Acid. | | *p*-Amidophenol. | |
|---|---|---|---|---|---|---|---|
| | | Unmordanted 2 per Cent. | Chromed 2 per Cent. | Unmordanted 2 per Cent. | Chromed 2 per Cent. | Unmordanted 2 per Cent. | Chromed 2 per Cent. |
| α-Naphthol ... | | Brownish-orange | Dark brown | Bright reddish-orange. | Bright reddish-orange. | Dark brown-orange | Dark brown-orange |
| Dihydroxy-naphthalene. | 1.4′..... | Dirty brown | Deep dark brown | Dull brownish-red | Dull brownish-red | Brownish Bordeaux | Brownish Bordeaux |
| | 1.1′..... | Brown | Blue-black | Reddish-brown | Dark brown | Dirty lilac | Dark greyish-violet |
| | 1.2′..... | Reddish-brown | Reddish dark brown. | Orange | Orange | Dull yellowish-brown. | Dull yellowish-brown. |
| | 1.2...... | Reddish-brown | Brownish-black | Dull yellowish-red | Violet | Dirty light brown | Dark grey |
| | 2.2′..... | Orange-red | Brownish-red | Yellowish-brown | Yellowish-brown | Terra-cotta | Terra-cotta |
| | 2.3′..... | Dull cherry-red | Brownish Bordeaux | .. .. | .. .. | .. .. | .. .. |
| | 2.3...... | Bordeaux | Violet-black | Light brown | Dull reddish-brown | Dull brownish-red | Blackish-brown |
| β-Naphthol ... | | Reddish-orange | Dark reddish-brown. | Yellowish-orange | Yellowish-orange | Scarlet | Scarlet |

The comparative dyeings were made on unmordanted wool and on wool mordanted with bichromate and tartaric acid, as well as on chrome-mordanted cotton. Nietzki's statement that the dyestuffs from the dihydroxynaphthalenes are deeper in shade than the corresponding naphthol dyestuffs is confirmed. To a certain extent the shades obtained from *p*-amidosalicylic acid are a mixture of those from *m*-amidobenzoic acid and *p*-amidophenol. The property of forming lakes due to the presence of the carboxyl and hydroxyl groups in the *o*-position or in the *o*- or peri (1.1′) position in the naphthalene ring is well marked in all the colours examined, and it is noticeable that whilst there is only a slight difference in depth of shade between the colours on mordanted and unmordanted wool from *p*-amidosalicylic acid, so long as only one lake-forming group is present, the shade becomes considerably bluer, besides being much darker, when the naphthalene ring also contains a mordant group.—T. A. L.

*Dianisidine Blue, New Applications of.* J. Rhodes. J. Soc. Dyers and Colourists, 1898, **14**, 22—25.

Dianisidine-blue, obtained by preparing the fabric with β-naphthol in alkaline solution and Turkey-red oil, or ricinoleate of ammonia, or the so-called "para-soap," drying, and developing in tetrazodianisol together with a copper salt, gives, on "mercerising" with caustic soda at 60° Tw. to

80° Tw., a dull violet. This corresponds in shade to the violet obtained without using a copper salt, or mercerising; it is, however, more fugitive to light, although analysis of the fibre showed no trace of copper. If, however, the blue, after washing free from soda, be treated with dilute hydrochloric acid, and then passed into a boiling solution of copper sulphate, very fine indigo shades are produced, which are superior to indigo in their fastness to light. If the mercerised blue is again treated with caustic soda of 60° T., a dull violet shade is obtained, which cannot be restored to its original brightness by boiling with copper sulphate. This method of using Dianisidine-blue lends itself to the production of varying indigo shades. The following are examples of the process. Mixture for preparing the fabric:—250 grms. of β-naphthol, 590 grms. of caustic soda solution of 36° T., 1,000 grms. of ammonium ricinoleate or Turkey-red oil, 150 grms. of sodium acetate. This is made up to 10 litres of water, and the fabric, after immersion, is dried and then printed with the following:—12·8 grms. of Dianisidine-base are dissolved in 200 grms. of water and 20 c.c. of hydrochloric acid of 36° T. Then 50 c.c. of nitrite of soda solution, $\frac{145}{1000}$, and 50 grms. of ice are slowly added, and the mixture allowed to stand 20 minutes, tested with starch iodide paper, and then 36 grms. of cupric chloride solution of 77° T. and 574 grms. of gum tragacanth are finally added. This gives a dark indigo shade, which may be reduced for medium or light shades by mixing 1 part of the dark colour with 3 parts of gum tragacanth, $\frac{50}{1000}$, for medium, or 8 parts of gum tragacanth and starch thickening for the light shade.

The printed fabric is dried, washed, soaped at 160° F., washed, and again dried. Having mercerised with the necessary strength of caustic soda, it is dried, washed till free from soda, acidulated with hydrochloric acid, and then placed in a bath of boiling copper sulphate solution containing 2 oz. of copper sulphate per gallon of water. Finally, the fabric is washed, dried, and finished. By this process, blues of a pure indigo shade are obtained.

A process is also given for plain dyeing, and the author points out that, by mercerising after the colour is developed, a faster blue is obtained than by mercerising first and then dyeing. It is interesting also to note that, in working with tetrazodianisol and cupric chloride, if a steel doctor be used, there is no deposit of copper on it, as there is when printing other colours developed with copper salts.—C. M.

### *Two-coloured Effects Produced by Printing Caustic Soda on Wool.* A. Kertész. Färber Zeit. 9, [3], 35—36.

It is well known that cotton printed with caustic soda dyes more intensely than those parts which are not printed.

The author finds that it is not difficult to obtain similar effects on wool. If glycerin be added to the printing-colour so as to moderate the action of the caustic soda, and care be taken to print without heating, the fibre is not affected, even when the goods remain unwashed for a long time. The printing-colour is composed of a mixture of caustic soda, gum-tragacanth, and glycerin.

After printing, wash without drying and then dye.

Or, instead of washing in water, it is best to pass the pieces, at 50° C., through a bath containing 4—5 kilos. of ammonium chloride per 100 litres. The pieces are then washed in water.

It is preferable not to dye at the boiling point, since if so dyed, the colours of the printed and unprinted parts tend to become uniform.

The difference in shade of the printed and unprinted parts is more marked by adding colouring matters to the printing colour; or by adding lead acetate to the ammonium chloride bath, the lead salt combining with the alkaline sulphides formed in the printed parts in the wool, and so producing a deeper shade.

The process seems capable of being carried out with the lightest fabrics, *e.g.*, ladies' dress goods, cashmeres and felts, as well as yarns.

Sliver is somewhat affected by the caustic soda; hence in this case the process is not applicable.—J. E. II.

### *Azo-Puce for Grounds Enriched by Discharge Colours.* P. Dosne. With Report by H. Schmid. Bull. Soc. Ind. de Mulhouse, Dec. 1897, 408—410.

It is difficult to get a level and dark chocolate by using insoluble azo-colours developed on the fibre, prepared with β-naphthol. By adding amidonaphthol, *e.g.* amidonaphthol B D (Cassella), to an alkaline solution of β-naphthol, an excellent level chocolate is obtained on being developed with *p*-nitraniline, and working in a boiling alkaline solution of copper chloride ($CuCl_2$).

The author used the white-azo-discharge of M.L.B.; for coloured-discharges, Rhodamine, Auramine, Brilliant Green, Victoria Blue (40 grms. of colour + 60 grms. of tannin, per litre of white-azo-discharge, thickened with 30 grms. of wheat starch and 30 grms. of dextrin per litre).

The fact that this chocolate can be directly discharged by the azo discharge (M.L.B.) is extremely interesting.

Schmid confirms Dosne's results, but prefers to use Persian berries, Phenocyanine, and a mixture of these two, for yellow, blue, and green discharges.—J. E. H.

### *Azo-Puce for Grounds, by the Saddening of p-Nitraniline Red by means of Aniline Black; Note on.* H. Schmid. Bull. Soc. Ind. de Mulhouse, Dec. 1897, 411—413.

The chocolates now obtained by diazotising, and enriched with colours by the resist process, are not altogether satisfactory. A simple means of producing a solid chocolate is to take a finished *p*-Nitraniline Red, pad in a solution of steam-aniline-Black (prussiate), and pass through the small Mather and Platt steaming-chamber. The value of this chocolate is that it will allow of discharging direct, by simply printing on the *p*-Nitraniline Red, the white or coloured-azo-discharge (M.L.B.) previous to passing through the Aniline Black solution.

The *p*-Nitraniline Red is destroyed by the reducing action of the discharge, which, however, acts as a resist to the Aniline Black.

By printing with sodium acetate (which resists the Aniline Black, but has no action on the *p*-Nitraniline Red) a red is produced. Thus we get white and red on a chocolate ground. The cloth is prepared in the usual way with 25 grms. of β-naphthol R per litre, dried, and dyed with 14 grms. of *p*-Nitraniline per litre. After drying, pad in steam-Aniline-Black bath, dry in the hot flue, and print the discharge colours.

Steam 10 minutes without pressure. Pass through warm dilute hydrochloric acid and finally soap.—J. E. H.

### *Dyed Discharge Colours obtained on Tannin Mordant discharged with Sodium Aluminate, Note on.* Aug. Romann. Bull. Soc. Ind. de Mulhouse, Dec. 1897, 406—407.

By operating as described, variously coloured discharges can be produced on tannin mordant.

Pad the piece in tannin mordant (10—20 grms. per litre), pass through tartar emetic, wash, dry, and print on sodium aluminate. Pass through the Mather and Platt steamer at 80° C. for three minutes.

Wash out the thickening with ammonium chloride and again wash.

Dye at 40° C. with such basic-colours as Methylene Blue, Auramine, Safranine, Methylene Green, Aniline Violet, &c.

After dyeing, pass through boiling water, if the white be not good.

Then dye again with mordant colours, *e.g.* Alizarin (red, yellow, orange), Persian-berries, Quercitron-bark, &c.

After this dyeing operation, a light soaping follows.

For certain shades it is better to prepare the cloth with sulphated oil and citric acid, steaming, washing, and soaping.
—J. E. H.

### *Half Woollen Fabrics, with Dark-coloured Shoddy; Dyeing of.* [*Shoddy Unions, Dyeing of.*] A. Kessler. Färber Zeit. 9, [4], 49—50.

The weft of the carded goods in question consists of differently coloured shoddies along with more or less white wool, or even white cotton. The warp is cotton.

These mixed class unions, on being dyed with substantive colours, remain unaltered in appearance, and therefore the colours always appear mixed, even in the darker shades.

To avoid too mixed an appearance, the dark ground must be reduced to a paler one, and the fabric must be of a uniform shade. This might be effected by stripping the goods with potassium bichromate and sulphuric acid, which would make the dyeing still more expensive. But, even without stripping, the cost of the substantive colours is an obstacle to their introduction.

Now with these goods, cheapness is the chief stipulation, and to make them appear of a richer quality they must be made to feel somewhat stiff. This stiffness is imparted by means of tannate of iron.

On the other hand, goods dyed with substantive colours require to be thickened with dextrin, &c.—a further item of expense which cannot be disregarded.

The author proceeds as follows :—After stripping the goods with sulphuric acid (in this case the shoddy is not of such a yellow-brown colour as when stripped with potassium bichromate and sulphuric acid), the wool is at once dyed with acid colours. The goods are then slightly rinsed in the same vat (after running off the dye-liquor) with cold water, taken out, and washed in the chain form. The cotton is dyed cold in a fresh bath, with the requisite amount of substantive colour and Glauber's salt.

However, it is preferable to use the jigger, since less dye-liquor is needed, and consequently less dyestuff. This means a reduction in expense.

The jigger is prepared with dye solution and 4 kilos. of Glauber's salts per 100 litres. The goods are run through the cold liquor until the cotton is dark enough, *e.g.*, 1 hour for 200 m. of material. The dye-liquors are preserved.

If for the first bath 3 kilos. of dyestuff are necessary, $2\frac{1}{2}$ kilos. are required for the second bath, 2 kilos. for the third, $1\frac{1}{4}$ kilos. for the fourth, and half a kilo. only for the fifth and each succeeding bath.—J. E. H.

### *Felt Hats, The Use of Brilliant Black in the Dyeing of.*

C. A. Otto. Färber Zeit. 1898, **9**, [4], 52.

The hat bodies, without previous wetting-out, are entered into the boiling dye-bath, which is made up for 25 kilos. of goods with $2\frac{1}{2}$ kilos. of Glauber's salt, $1\frac{1}{2}$ litres of acetic acid, 900 grms. each of Brilliant Black B and E (B.A.S.F.), 100 grms. each of Victoria Yellow C and Patent Green W. (M. L. and B.).

Boil three-quarters of an hour, then add 1 litre of acetic acid (66° B.), well diluted, and continue boiling for half an hour.

The black is fast to wear, and gives the hat bodies a soft handle and greater lustre ; it is well dyed through, and stands shrinking and pressing without being changed ; it is, however, very sensitive to alkalis.

Hats which are first dyed and then stiffened must be rinsed with carbonate of soda and ammonium chloride to remove excess of size.

By this treatment, however, not only the stiffening, but also the dye, partly disappear, so that the hats require to be dyed again.

Hats which are first stiffened and then dyed are not so stiff, and become soft after wearing for some time. These hats cannot be so effectively hot-pressed, or the size strains out, and must then be removed with sodium carbonate or ammonium chloride.—J. E. H.

## PATENTS.

### *Fibre, Raw Jute ; Method of Improving the Colour of.*

J. Shearer, Maryfield, Dundee, Scotland. Eng. Pat. 2736, Feb. 2, 1897.

*See under* V., *page* 240.

### *Fibre, Raw Jute ; Process for Improving the Colour of.*

C. O'Brien and J. Shearer, Maryfield, Dundee. Eng. Pat. 2960, Feb. 4, 1897.

*See under* V., *page* 240.

### *Dyeing Apparatus for Carded Wool.*

A. Schmidt, Lanificio di Gavardo, Italy. Eng. Pat. 4853, Feb. 23, 1897.

This apparatus for dyeing carded wool wound upon bobbins (wool-tops), consists of a vessel in two parts, the upper part containing the bobbin-holder, and the lower part, which is merely a continuation of the upper, acting as receiver for the dye-liquor. In the lower vessel works a turbine with pairs of blades inclined slantwise downwards, and in the walls of both parts of the vessel are a number of passages communicating below with the receiver. Within these passages, steam pipes rise to about one-third of the height of the apparatus, each pipe being provided at the top with a nozzle. When steam is caused to pass into these pipes, the combined action of the turbine and the steam, forces the liquid in the receiver into the passages, at the top of which it emerges and is showered down on the material to be dyed. The liquid thus passes downwards through the wool into the receiver, and the circulation is repeated. The vessel is provided at the top with a marginal ring, which prevents the contents of the bath from overflowing.—R. B. B.

### *Mordanting and Dyeing Animal Fibre, Process of.*

O. P. Amend, New York, U.S.A. Eng. Pat. 30,038, Dec. 18, 1897.

According to this invention, animal fibres are mordanted with chromium at a low temperature. This is effected by successive treatment with chromic acid and a reducing agent, or, in cases where the dyestuff itself acts as a reducing agent, by treatment with chromic acid alone. The fibre is immersed for half an hour at the ordinary temperature in a bath containing free chromic acid. The strength of this bath may vary from $\frac{1}{30}$ per cent., when the subsequent reduction is performed in the mordanting bath, to 5 per cent., when the reduction takes place in a separate bath. At the low temperature employed, all oxidation of the fibre is avoided. The reducing agent—*e.g.*, 0·2—0·5 per cent. of sulphurous acid on the weight of liquid—is in practice added to the chromic acid bath, and the fibre further worked in this bath for $\frac{1}{2}$—1 hour, after which it is ready for dyeing.

When the dyebath contains a substance capable of reducing the chromic acid, it is not necessary to employ a separate reducing agent. For instance, a black may be dyed by treating the fibre with chromic acid as above, and then steeping for three hours in a 1 per cent. solution of aniline salt, the colour being afterwards developed in the usual way at any suitable temperature, preferably at 80° C. —R. B. B.

### *Mordanting Animal Fibre, Process of.*

O. P. Amend, New York, U.S.A. Eng. Pat. 30,039, Dec. 18, 1897.

The mordanting process described consists of four operations, as follows :—(1.) The fibre is treated with a dilute acid which will not reduce chromic acid, such as oxalic, acetic, or sulphuric acid. By preference, a 0·1 per cent. solution of oxalic acid is employed at a temperature of about 125° F., and in this bath the fibre remains for about half an hour. (2.) The fibre is then steeped for half an hour in a solution of chromic acid of about $\frac{1}{30}$ per cent. strength. In practice, the chromic acid may be added to the first bath, and the temperature allowed to fall naturally. (3.) An addition is made to the same bath of 0·2 per cent. (on the weight of liquid) of bisulphite of soda and the fibre further treated for half an hour ; the temperature during this operation may vary within wide limits, but must not be raised to such a point that the fibre is oxidised. (4.) The acid remaining in the fibre from the above treatment is neutralised by washing in a sodium carbonate solution of about $\frac{2}{15}$ per cent. strength for half an hour, the temperature being gradually raised to 140° F. The fibre is then ready for the dyebath.

It is claimed that the process as described is applicable to animal fibre both in the scoured and unscoured conditions.

If the fibre has been previously scoured it is possible to combine the first two operations, adding the oxalic acid and chromic acid simultaneously, and in certain cases it may also be possible to dispense with the neutralising process. — R. B. B.

*Tannin-Antimony Lakes of Polyazo Dyestuffs and Basic Safranine Azo Dyestuffs on the Vegetable Fibre, A Process for Dyeing Fast.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 5259, Feb. 26, 1897.

Two methods are described of dyeing with the basic-azo-dyestuffs referred to in the title, and both these methods are simpler and more rapid than the ordinary method of applying basic colours in three operations, *viz.*. mordant with tannin, fix, and dye. The new methods are:—

(*a.*) The dyebath contains, in addition to the colouring matter, tartar emetic or other antimony salt, with or without neutral salts—*e.g.*, salt and sodium sulphate—which favour the attraction of colour by the fibre. After boiling for one hour, an addition of tannin is made to the same bath and boiling continued for half an hour.

(*b.*) The cotton is dyed with the colouring matter, with or without the addition of a neutral salt, and is subsequently treated in a separate bath containing tannin and an antimony salt. Although a precipitate of antimony tannate will, of course, be formed in this second bath, it is stated that the affinity to the fibre is evidently so great that the tannate of antimony not only does not withdraw the colouring matters from the fibre, but, on the contrary, is itself withdrawn from the bath by the dyestuffs in the fibre which are not yet fast, thus forming the fast tannin-antimony colour-lake on the fibre.—R. B. B.

*Washing and Boiling off, Impts. in the Method of, and in the Preparation of Fabrics to be Dyed or Printed.* J. Waugh, Bradford, Yorks. Eng. Pat. 6498, March 12, 1897.

It is claimed that phenols or amines of the aromatic series, especially phenol and cresol, may with advantage be employed as partial or entire substitutes for soap in washing or boiling-off vegetable fibres. It is stated that by using 1 kilo. of raw carbolic acid in 400 litres of water, a better result is obtained than with 16 kilos. of soap in the same quantity of water, and that the relative cost is threepence in the former case to five shillings and sixpence in the latter. A higher temperature can also be employed with phenol than with soap, without injuring the colour in the case of dyed goods.

Other applications of phenol are to replace dung in clearing and fixing operations, and as a substitute for, or as an addition to, oil mordants, previous to dyeing with alizarin, &c.—R. B. B.

*Textile Staples, Filaments, or Fibres on Tubes and Bobbins; Improved Appliance for Dyeing and Bleaching.* L. Koenig, jun., St. Petersburg, Russia. Eng. Pat. 9011, April 8, 1897.

This patent describes an improvement in the bleaching and dyeing machines which contain perforated bobbins carrying the fibre and fitted on perforated supply tubes. The improvement consists in leaving an annular space between the bobbins and the supply tube, in order to afford a freer passage for the liquid. This space is enclosed and rendered liquor-tight, below, by a shoulder of the bobbin fitting on to a shoulder of the tube, and above by a nut attached to the bobbin, screwing on a threaded extension of the supply-tube.—R. B. B.

*Colouring Yarns, Impts. in Method of and Apparatus for.* C. L. Horack, Brooklyn, U.S.A. Eng. Pat. 24,533, Oct. 22, 1897. (Under Internat. Convention.)

In the colouring of carpet yarns by means of printing stripes of thickened colour upon the yarn and subsequently steaming, it is extremely difficult to prevent irregular "running" of one stripe into the next. According to this invention, by means of a spreader mounted on the carriage in the rear of the colour-wheel, the entire width of the colour stripe in course of application is subjected to a rubbing action, and simultaneously one-half of the adjoining streaks on either side are also subjected to the action of the spreader, so that the colour of the printed stripe is thus spread over half of each adjoining stripe, *viz.*, the one printed previously and the one to be printed next. It is claimed that uniform shading effects are thus produced.—R. B. B.

*Marbling, Mottling, or Colouring Paper or Cardboard on one Side in the Paper Machine; An Improved Process for.* E. Lehmann, Fockendorf, Germany. Eng. Pat. 25,117, Oct. 29, 1897.

The process consists in conveying on to the paper web at a suitable place in the machine, by means of a roller, spray, or other device, a mixture of coloured albuminates, resin soap, or fat soap, with chemicals, *e.g.*, acids or alum, which are capable of precipitating the former substances.

—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Cryolite: Its Mining, Preparation, and Utilisation.* W. C. Henderson. Journ. Franklin Inst. **145**, [1], 47—54.

Cryolite, the double fluoride of sodium and aluminium, $6NaF, Al_2F_6$, has been found in Greenland, at Miask in the Ural Mountains, and more recently in Colorado and Wyoming. The only deposit of commercial importance is that discovered in 1811 by Giesecke, at Ivigtut in South-West Greenland. Ivigtut lies on the Arksuk Fiord at a spot 20 miles from the S.W. coast-line and $8\frac{1}{2}$ miles from Isua, the nearest Esquimaux settlement. The mineral is easily accessible for removal, no portion being at a greater distance than 150 feet from low-water line and its general elevation not exceeding 10 feet from high water. The greatest length of the deposit extends in a direction parallel to the sea, and covers a distance of about 400 ft., 100 ft. of which is washed by the sea.

The deposit is mined by a Copenhagen company, who hold it under a lease from the Danish Government. The only inhabitants of Ivigtut are the miners from Copenhagen, and during the summer season (April to November), about 140 men are employed in quarrying, mining, sorting, and piling the cryolite. The drilling is done by hand and the mineral dislodged by blasting. It is carefully sorted into two grades, Nos. 1 and 2, of about 99 per cent. and 92 per cent. respectively, which are taken to the top of the mine in 2-ton cars, running on an incline railway operated by steam. Here it is formed into piles 100 ft long, 20 ft. wide, and 4 ft. high (about 37 cubic fathoms). The cubic fathom constitutes the unit and weighs 13·6 tons. Last season's output, the largest known, was 13,000 tons, of which 10,500 tons were received at Philadelphia. The best grade goes to Copenhagen, the second to Philadelphia, and the value of $\frac{1}{5}$ of each cubic fathom mined and shipped goes to the Danish Government. All the waste is dumped into the sea for the purpose of building wharves and securing a more accessible anchorage. Just before winter sets in, the mines are flooded with sea-water to prevent the accumula- of ice and snow which would otherwise occur, and during winter, quarrying operations are considerably lessened. After the general thaw sets in, the mines are drained by steam pumps, three weeks being required for the purpose. Of the minerals usually associated with cryolite, some—such as quartz, galenite, pyrites, &c.—are separated by sorting; others, occurring only with cryolite, and rare, are removed in the manufacturing process, since they suffer no change under the treatment by which cryolite is decomposed.

This treatment is the well-known Thomsen method for the manufacture of soda and alumina by calcining an intimate mixture of limestone and cryolite. Other uses to which cryolite is put are in the manufacture of aluminium by electrolysis, wherein the cryolite is used as a solvent for the pure alumina, dissolving this substance to the extent of 21 per cent., and in the manufacture of cryolite glass or hot-cast porcelain.—J. T. C.

*Perchlorates, Production of, by Electrolysis.* F. Winteler. Chem. Zeit. **22**, [11], 89—90.

The formation of perchlorates during the electrolytic preparation of chlorates of the alkalis or alkaline earths, in presence of electrodes of platinum or peroxides, is most favourably influenced by the following conditions: the presence of an acid solution and prevalence of a low temperature at the anode; the employment of currents ranging between 4 and 12 ampères per sq. decimetre; and the use of highly concentrated electrolytes. Thus, for example, if a neutral solution of potassium chlorate (saturated at the ordinary temperature) be electrolysed ($da = 8$) without a diaphragm, and the electrodes cooled down to 10° C. by means of water, 73 per cent. of the oxygen otherwise eliminated is utilised in the formation of perchlorate. Under the same conditions a yield of 95 per cent. is obtained from a saturated (1:1) solution of sodium chlorate.

The direct cooling of the electrodes is of prime importance, and the apparatus should be so arranged that only one side of each comes in contact with the electrolyte, in order that the current may be accurately regulated. The best output is caused by a current of from 8—12 ampères, the yield being decreased when these limits are exceeded. Chlorates are converted into perchlorates even in presence of 4 per cent. of alkali, provided the agitation of the anode and cathode solutions is effected solely by the liberated gases.

The evolution of ozone is so considerable when concentrated solutions are employed, that the caoutchouc fittings of the apparatus are rapidly destroyed. On this account the process might possibly be suitable for the technical preparation of this gas. When carbon electrodes are used for electrolysing chlorates, rapid destruction occurs and no perchlorate is formed. No other metals than platinum or iridium are suitable for electrodes, the ion $ClO_3$ liberated at the anode forming, in such case, soluble salts.

The conditions under which the electrolysis of dilute hydrochloric acid into perchloric acid can be effected by platinum electrodes, as reported by Riche (Comptes Rend. 1858, 348), are identical with those already described, the best temperature being one at which no gaseous chlorine is liberated.—C. S.

*Chromium Sulphide and the Sulphochromites.* R. Schneider. J. Prakt. Chem. 1897, **56**, [22, 23, and 24], 401—424.

According to Berzelius, when chromium oxide is fused with potassium polysulphide and the melt extracted with water, chromium sulphide is left. The author finds that the principal product of such a reaction is not chromium sulphide, but an alkaline sulphochromite. The potassium compound, for example, is best obtained by fusing 1 part of potassium chromate with 24 parts of potassium carbonate and 24 parts of sulphur. On treatment with cold water, reddish bluish-grey crystalline leaves remain, which are not decomposed by the polysulphide solution. These crystals consist of potassium sulphodichromite, $K_2S.2Cr_2S_3$. By treatment with hydrochloric acid in entire absence of air, the corresponding acid, $H_2Cr_4S_7$, is obtained, the form of the crystals remaining unchanged. The acid is very unstable, and is at once oxidised by air; gently heated in carbonic acid gas it decomposes as follows:—

$$H_2Cr_4S_7 = H_2S + 2Cr_2S_3$$

If the mass resulting from the fusion of chromium oxide and potassium polysulphide be repeatedly boiled with water, the sulphodichromite is decomposed, and pure chromium sulphide, $Cr_2S_3$, remains, as stated by Berzelius.

If sodium carbonate be used instead of the potassium compound, or if a mixture of the two carbonates in equal quantities be employed, sodium sulphochromite, $Na_2Cr_2S_4$, is obtained. By the action of hydrochloric acid on this salt in the absence of air, the acid $H_2Cr_2S_4$ is formed. By admixture with aqueous solutions of silver nitrate, copper sulphate, and lead nitrate, sodium sulphochromite is converted into the sulphochromites of the heavy metals.

—A. C. W.

*Hydrofluoric Acid, Determination of.* J. Zellner. Monatsh. für Chem. 1897, **18**, [10], 749.

*See under* XXIII., *page* 275.

*Acetic Bacteria, Contributions to the Knowledge of the.* W. Henneberg. Wochenschr. für Brau. 1898, **15**, [2], 19.

*See under* XVII., *page* 258.

*Ammonia and Hydrochloric Acid, Reaction between Dry, and the Vapour Density of Dry Ammonium Chloride.* S. Gutmann. Annalen, 1898, **299**, [3], 267.

*See under* XXIV., *page* 283.

*Gas Liquor, Distillation of, for the Production of Liquid Ammonia.* O. Pfeiffer. J. für Gasbeleuchtung, **41**, [5], 69.

*See under* II., *page* 231.

*Perchlorate in Nitrate of Soda [Chili], Method of Estimation of.* R. Selckmann. Zeits. f. angew. Chem. 1898, [5], 101.

*See under* XXIII., *page* 275.

*Aluminium Fluoride as an Antiseptic in Distilleries.* H. Cluss and H. Feber. Zeits. für Spiritusind. **21**, [1], 2—3.

*See under* XVII., *page* 258.

*Tartrates, A New Ferment of: Bacillus tartricus.* L. Grimbert and L. Ficquet. Journ. Pharm. Chim. 1898, **7**, 97.

*See under* XXIV., *page* 283.

## PATENTS.

*Ammonia, An Improved Method of Producing.* C. H. Mehner, Charlottenburg, Germany. Eng. Pat. 2654, Feb. 1, 1897.

*See under* III., *page* 237.

*Freezing Mixture, Improved.* W. G. Gregory, senior, Salford, Manchester. Eng. Pat. 11,977, May 14, 1897.

The mixture consists of 3 parts of tincal (crude borax), 4 parts of bay salt, and 112 parts of ice. As the mixture slowly liquefies, the liquid is drawn off by a properly designed tray and channel, to cool and sweeten the air in refrigerating chambers, &c.—E. S.

*Nitric Acid Condensers, Impts. in.* J. V. Skoglund, Bayonne City, New Jersey, U.S.A. Eng. Pat. 22,816, Oct. 5, 1897.

The vapours from the still are admitted to the lower part of a tower or chamber, having a false bottom, and packed with pieces of glass, pottery, or the like, and kept at such a temperature that but little condensation can take place. From the top of the tower the vapours are conducted to the bottom of a second similarly packed and heated tower, whence they ascend into a condensing pipe, which is horizontal except at the bends, and is cooled by a water spray, and then pass into a cooled porcelain vessel having an exit for uncondensed gases, and a return pipe leading from the bottom to the top of the second tower. As strong nitric acid boils at a lower temperature than a weaker acid, the latter condenses in the first tower, and the former in the second. The acid that condenses in the cooled tube, together with the red gases and impurities, runs back upon the pieces of acid-proof material in the towers, flowing over them in "thin films" to the bottom, while the heat of the ascending gases and vapours acts to drive off the red gases, chlorine, &c., while colorless nitric acid condenses. It is stated that "chemically pure white nitric acid is produced in one operation and without loss of acid." Air is admitted to the towers, and the acid is drawn off from the bottom of these by trapped pipes. Certain parts of the apparatus are made of pure lead, which is stated to be but slightly acted upon by the acid vapours.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Glass, Fusible Colours for.* Sprechsaal, **30**, [40], 1160—1161.

These colours are prepared by fusing the materials (flux and pigment) in a Hessian crucible until the mass is clear, and, after stirring with a hard earthenware rod, pouring into water, the pulverised mass being finally ground.

*Fluxes.*—(1) (Rocaille flux) sand, 5 parts; minium, 15 parts. (2) Sand, 10 parts; calcined borax, 10; minium, 10 parts. (3) Sand, 3 parts; borax, 3; minium, 6; nitre, 1 part.

*Yellow Colours.*—*Chrome:* Flux, 15 parts; lead chromate, 2 parts or flux, 10 parts; barium chromate 2 parts.

*Brilliant Yellow:* Flux, 25 parts; lead chromate, 3 parts; barium chromate, 2 parts. *Greenish-yellow:* Flux, 10 parts; silver chromate, 1 part. *Naples Yellow:* Flux, 14 parts; potassium antimoniate, 2 parts. A similar citron yellow is obtained from: flux, 25 parts; potassium antimoniate, 3 parts; ferric oxide, 1 part.

*Blue Colours.*—The cobalt compounds employed must be pure. A special flux composed of: sand, 2 parts; minium, 2; borax, 2; and potassium bitartrate, 1, is used, and a blue frit of sand, 4 parts; borax, 3; potassium tartrate, 2; and cobaltous phosphate, 3 parts, gives good results; 1 part of frit mixed with 3 to 6 parts of flux giving shades ranging from dark to light blue. *Sky-blue:* Flux, 15 parts; blue frit, 2; zinc white, 1 part. *Deep Blue:* Flux, 8 parts; cobaltous carbonate, 1 part. *Light Blue:* Flux, 15 parts; cobaltous oxide, 1 part. *Azure Blue:* Flux, 7 parts; cobaltous oxide, 1 part; zinc white, 2 parts. *Turquoise Blue:* Cobaltous oxide, 4 parts; zinc oxide, 1; and calcined alum, 10 parts, fused with: sand, 30 parts; borax, 25; minium, 60 parts. *Greenish-blue:* Flux, 10 parts; zinc oxide, 1; cupric oxide, 0·5; antimony oxide, 0·2; cobaltous oxide, 1 part. *Royal blue:* Sand, 2 parts; potash, 4; cobaltous oxide, 2 parts; fused, mixed with Rocaille flux, 5 parts, and fused over again.

*Green Colours.*—*Chrome-green:* Flux, 6 parts: chromium oxide, 1. *Yellow-green:* Same proportions with 1 part of lead chromate. *Sap-green:* Sand, 10 parts; borax, 6; minium, 6; cupric oxide, 1; chromium oxide, 0·5. *Blue-green:* Flux, 12 parts; chromium oxide, 2; cobaltous oxide, 1; zinc oxide, 1. *Dark Green:* Flux, 30 parts; chromium oxide, 2; cobaltous oxide, 2; or 6, 2, and 1 parts respectively. *Copper-green:* Flux, 6 parts; cupric oxide, 1; chromium oxide, 0·5. *Black-green:* Flux, 12 parts; chromium oxide, 1; cobaltous oxide, 1. Green shades can also be prepared by mixing cobalt blue and antimony yellow fusible colours.

*Red and Violet Colours.*—Gold-purple is used for rose-reds and (precipitated) ferric oxide for ordinary reds. Since the shade produced by this oxide changes from orange-red to red, dark violet, and brown, as the intensity of the firing increases, it requires watching in order that the desired shade may not be exceeded. Occasionally zinc oxide or alumina is mixed with the ferric oxide, 3 parts of ferric sulphate being calcined with 2 parts of zinc sulphate or 3 of alum.

*Light Red.*—Flux, 8 parts; ferric-zinc oxide, 1 part. *Red:* Flux, 10 parts; ferric-zinc oxide, 2. *Orange-red:* Flux, 14 parts; yellow ferric oxide, 2; red ferric oxide, 2; antimony oxide, 1 part. *Yellowish-red:* Flux, 18 parts; red ferric oxide, 2; lead chromate, 1 part. *Dark Red:* Flux, 6 parts; red ferric oxide, 1.

The most beautiful rose-red tints are obtained with the so-called purple of Cassius. 3 grms. of gold are dissolved in 40 grms. of hydrochloric acid and 10 grms. of nitric acid, the gold chloride remaining after the evaporation of the solvent being re-dissolved in 2 litres of water and thrown down by the gradual addition of a solution of 10 grms. of tin in 40 grms. of hydrochloric acid and 15 grms. of nitric acid, diluted with 500 grms. of water. The carmine-coloured precipitate is thoroughly washed and stored in a flask. *Carmine-red:* Purple flux (composed of sand, 10 parts; borax, 4; potassium carbonate, 4; minium, 2 parts), 20 parts; gold-purple, 1 part. *Purple-red:* Purple flux, 15; gold-purple, 1 part.

*Violet Shades* are prepared from manganic oxide alone or mixed with violet ferric oxide; finer qualities from gold purple with manganic or cobaltic oxide.

*Manganese Violet.*—Sand, 10 parts; borax, 6; minium, 6; manganic oxide, 3; fused together. *Blue-violet:* Same flux with manganic oxide, 2·5 parts; cobaltic oxide, 0·25 part. *Purple-violet:* Purple flux, 20; gold-purple, 1; cobalt-blue, 1 part. *Flesh Tints:* Iron-red, 2; purple-red, 3; purple flux, 5 parts; or 1, 2, and 4 parts respectively.

*Brown and Black Colours.*—Ochre, brown ferric oxide, or ferric chromate is used for browns. *Red-brown:* Flux, 30; ferric chromate, 5 parts (from red ferric oxide). *Sepia-brown:* Flux, 30; brown ferric oxide, 4; lead chromate, 1 part. *Dark Brown:* Flux, 24; dark brown ferric oxide, 3; lead chromate, 1; or flux, 30; ferric sulphate, 4; manganic oxide, 1; cobaltic oxide, 0·5 part.

*Black.*—Sand, 10; borax, 6; minium, 10 parts; cobaltic oxide, 5; manganic oxide, 5 parts. *Deep Black:* Flux, 3; iridium oxide, 1 part.—C. S.

### *Stoneware, Cracking of, in the Muffle.* P. Hermann.

Sprechsaal, 30, [46], 1342—1343; [47], 1370—1371.

The tendency of stoneware to crack when fired in muffle furnaces for the purpose of fixing over-glaze pigments, is due to several causes, all the result of defective manipulation. In the first place, an excessive tension of the glaze, due to the injudicious adjustment of the relative co-efficients of expansion of glaze and body, will cause the ware to break. Secondly, an excessive admixture of quartz; the use of insufficiently ground quartz; insufficient firing in the first kiln; or an immoderate thickness of glaze, will produce the same result, the three first-named defects resulting in the production of insufficiently cohesive ware, to which the fissures developed in the glaze under the influence of the heat of the muffle will extend—particularly when the glaze is unduly thick—the contraction of the glaze then tearing the body of the ware asunder.

Finally, if the muffle be heated too quickly at the outset, the defects already existing in the ware will be intensified, with the result that the pieces will readily craze and crack from the irregular expansion produced. This is particularly the case with ware of irregular density, due to an excessive proportion of plastic clay, which necessitates a moderate temperature in the first kiln, the results of which are imperfect shrinkage and corresponding inequality in the density of the piece.—C. S.

### *Stoneware, Fired, Engobage Decoration on.* P. Hermann.

Sprechsaal, 30, [50], 1461—1463.

The chief drawback to the application of "engobage" to unbaked stoneware, *viz.*, the risk of spoiling the shape of the pieces in the soft state, is overcome by reserving the decorative work until the biscuit stage is reached. There is then no difficulty in getting the engobage to adhere, the porosity of the ware being sufficient for this purpose, provided the pieces are freed from dust. The question of the relative shrinkage of the biscuit ware and the overlay is one of slight importance, and is found to be altogether negligeable in practice. Greater cohesion between the two masses can be secured by incorporating with the engobage a proportion of flux or glaze, which acts as a cement when fired. Under-glaze pigments may be used for coloured engobage bodies; or, in the case of reds and browns, ochreous clays, without flux, are employed. For example, a *brick-red* engobage body is prepared from: fat, white-burning stoneware clay, 40 parts (by weight); yellow-burning crucible clay, 35 parts; white quartz sand, 100 parts; red-burning ochreous clay, 20 parts; whilst a *dark brown* is furnished by: quartz sand, 50 parts; red-burning ochreous clay, 25 parts; ferruginous manganese oxide, 7·5 parts. Fineness of division is essential to success. The firing temperature for the above mixtures is 900°—1,000° C., and where greater heat is to be applied, the tendency of the ochre to darken in colour is counteracted by an admixture of alumina.

For white engobage, white stoneware body mixed with white glaze is used, and for blues, greens, rose-reds, &c., the body is mixed with about 25—35 per cent. of under-glaze pigment and 10—15 per cent. of stoneware glaze, the exact proportions varying according to the shade desired. For *pale blue*, the pigment is prepared from ammonium alum, 2,500 parts; aluminium sulphate, 2,500; cobalt oxide 200, fused together, 1 part of the product being mixed with 3 of dry stoneware body and 10 — 15 per cent. of white glaze. For *rose-red*, 4 parts of pink-red under-glaze colour, 12 parts of dry body, and the above proportion of glaze, make a typical engobage.

The covering applied should be as thin as possible—as a rule not more than half the thickness of the engobage on unbaked ware; but where fluxes are used, the layer may be thicker—a condition which allows of the exercise of economy in the use of the pigments, darker shades being produced by applying a thicker coating of engobage.—C. S.

### PATENTS.

*Enamelling Glass Plates, and their Application to Walls and Ceilings for Decorating or Covering same; Impts. in.* N. E. Geille, Brussels. Eng. Pat. 23,807, Oct. 15, 1897.

Plates of glass are coated on one side with hot enamel and then gradually heated in a furnace at a temperature sufficient to cause the enamel to firmly combine with the glass. Clear glass can be thus rendered opaque or semi-opaque in any colours, or imitations of marble, or frosted or cracked surfaces, &c. can be produced. The enamel is put on with a rough surface to admit of its adhering to the surface of the wall to be covered. In lining walls, &c., the joints between the plates are concealed by edgings of enamelled glass.—H. H. B. S.

*China Clay, Improved Method of and Means for the Drying of.* A. C. Tulloch, Ealing, W. Eng. Pat. 24,505, Oct. 22, 1897.

The china clay, after its removal from the settling-tanks, is spread out upon trays in a chamber heated by hot air, and having a controllable exit orifice. The trays are removable, enabling them to be inserted and withdrawn as required.—H. H. B. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Blast-Furnace Slag after Treatment with Nitric Acid, as an Addition to Hydraulic Cement.* A. D. Elbers. Eng. and Mining J. 1897, **64**, [23], 664. (See also this Journal, 1897, 680, 800, and 915.)

The author, in previous articles, has shown why an admixture of prepared slag, though very beneficial in its ultimate results, is apt to have the effect of slightly lowering the initial strength of a cement, *i.e.*, the resistance to tension and compression which the cement acquires within, say, from one to seven days after being gauged. This defect, however, it is stated, can be overcome by treating the prepared slag with an aqueous solution of sodium carbonate, about 0·5 per cent. of $Na_2CO_3$ rendering the slag so efficient that a suitable admixture of it with Portland cement is apt to raise the tensile strength of the latter, 50 to 100 lb. per square inch at a seven days' test. The prepared slag is "alkalised" by moistening it with about as much of the sodium carbonate solution as it can absorb, the strength of the solution being regulated accordingly. The slag absorbs by capillary attraction, and can retain about 20 per cent. of water; hence a 2·5 per cent. solution applied in that proportion will impregnate the slag with an amount of hydrated carbonate equivalent to 0·5 per cent. of $Na_2CO_3$. The water of the solution that the slag has absorbed, evaporates readily at ordinary room temperature (70° F.); the slag treated with alkali does not clog much, and it falls easily apart after becoming completely dry. It appears to be more practical and certainly less expensive to treat the slag while it is still in the leaching vats, and after it has been reduced to a semi-dry state; but in this case, a stronger solution would be required, as, of course, much less of the solution would be absorbed. Dry-mixing of the alkali with both the prepared slag and the cement was tried, but did not answer, the cement, after setting, retaining a large proportion of the alkali in some spots and very little in others, and its working therefore being more or less irregular and unreliable.

The author considers that it is much more desirable to produce a high initial strength by the above method, which is in every respect harmless, than by the rather common practice of overliming Portland cement, which is certainly fatal to its durability.—A. S.

*Cement, Testing.* F. Schott. Chem. Zeit. Rep. **22** [4], 28.

The report (drawn up by the author) of the committee appointed by the German Association of Portland Cement Manufacturers, to establish uniform methods of testing, contains the results of the experiments made on the subject. It appears from the tensile and compression tests on 28 days' old blocks, that the variations were greater in the case of 1 : 1 cement and sand mixtures than when the proportion 1 : 3 was employed; and also that the personal equation is a considerable factor influencing these variations. Owing to the great influence of the proportion of water on the strength of the mortar, a more exact method of performing this determination is desirable.

It is proposed that in place of fixing the number of meshes and gauge of wire for the sieves, the dimensions of the actual apertures should be standardised. The Amsler-Lafont compression testers are said to have yielded concordant results at the various testing stations.—C. S.

*Cement Testing, Causes of Differences in the Results of.* M. Gary. Mitt. aus den Konigl. tech. Versuchsanst. zu Berlin, 1898, **16**, [1], 1—87.

Owing to complaints of certain firms in the cement trade of the unfavourable results of the tests made at the Royal Technical Institute at Berlin, an exhaustive series of experiments were carried out with the view of drawing up better regulations for testing samples. The principal results and recommendations are as follows:—

Normal cement mortar (1 : 3) suffers deterioration if the operation of mixing is prolonged beyond three minutes. Mixing by means of a machine, such as Steinbrück's, which mixes the mortar without grinding the sand, is to be preferred to mixing by hand.

The proportion of water used in mixing greatly influences the hardness of the cement, and it is desirable that more specific directions should be issued on this point.

The normal sand should be carefully washed before use, and should not contain more than 0·09 per cent. of loamy or earthy matter. Some fixed relation between the proportions of the finer and coarser particles should be agreed to. For oiling the moulds, a mixture of three parts of rape oil and one part of petroleum has been used advantageously.

As the hardness of the mortar is greatly influenced by the degree to which it is pressed into the moulds, the quantity put into the moulds should in every case be weighed.

The stamping out of cubes of neat cement is beset with difficulties, and special directions should be issued for carrying out this test.

If the mixing is made by hand with a trowel, the greatest solidity will be obtained by consolidating by 150 blows with a 2-kilo. hammer. An average rapidity of 50 blows per minute is recommended.

Briquettes for testing tensile strength and resistance to crushing should, as far as possible, be of uniform solidity, and should be faced up smooth on the upper surfaces.

Briquettes for tensile strength should not remain longer than half an hour in the moulds, but those intended for the crushing test should be allowed to remain 24 hours.

Cements which set slowly must be suffered to remain quite undisturbed during the setting. Freshly made briquettes must be carefully protected from draughts of air or from drying.

The water used for mixing Portland cement briquettes should be of uniform temperature. Warm water gives more favourable results than cold. The influence of temperature, however, varies with the characteristics of the cement, though it is about the same upon tensile strength tests as upon crushing tests.—H. H. B. S.

*Timber, American Forest.* Papier Zeit. **22**, [101], 3616.

*See under* XIX., *page* 263.

### PATENTS.

*Cement, Impt. in the Manufacture of.* Petrifite, Ltd., London. From J. Steiger, Athens. Eng. Pat. 29,212, Dec. 19, 1896.

The claim is for a process for manufacturing a cement consisting of specially prepared magnesium chloride, magnesia, and a salt of lead.

For instance: 35 parts of magnesium chloride are boiled with a little water for 8 to 10 hours, with continual agitation, until the mass becomes of a pasty consistence. The mass, after cooling, is then ground to a fine powder, and mixed with 60 parts of finely powdered magnesia, and 5 parts of a powdered lead salt, such as the acetate.—A. S.

*Refuse, Solid, of Breweries, Distilleries, Starch, Sugar, Poudrette Factories, and the like; A Process for Utilising the.* A. Weickmann, Munich. Eng. Pat. 3900, Feb. 13, 1897.

The refuse is treated with soda lye, and the pulp so produced mixed with different kinds of finely powdered resins. The lye is then washed out with water, the water remaining pressed out, and the residual mass dried in a vacuum drying apparatus, and finally pressed, while warm, into moulds. The material is then used for laths, panels, wall-coverings, &c.—H. H. B. S.

*Artificial Stone, Impts. in the Method of and Means for Making.* G. Christie, Hull. Eng. Pat. 6525, March 12, 1897.

Cement and sand, or any of the ingredients usually employed in the manufacture of artificial stone, are used, but instead of the full quantity of water being added at once, only just sufficient is used to allow of the mass being moulded. The moulded mass is then immediately turned out, and sprayed with water or steamed. The result is a rough surface almost undistinguishable from natural stone.
—H. H. B. S.

*Artificial Stone, Preservative Paint, and other analogous Materials; Impts. in the Manufacture of.* L. Grote, Landberg-on-Lech, Germany. Eng. Pat. 17,142. Sept. 12, 1893. 3rd Edit. (See This Journal 1894, 255.)

The patentee uses various combinations (according to the purposes intended) of chloride of magnesium, acetate of lead, plumbate of lime, baryta or strontia, magnesite waste from the manufacture of carbonic acid, and water. To these are added filling materials, such as "slag wool," sand, gravel, clay, or other mineral matter, wood, wood-pulp, wood shavings, paper, or similar materials.—H. H. B. S.

## X.—METALLURGY.

*Coke [Metallurgical], The Sulphur Content of.* O. Simmersbach. Stahl u. Eisen, **18**, 18; Chem. Zeit. Rep. **22**, [4], 29.

The main source of the sulphur in coke is the iron pyrites in the coal, sulphates being present only in small amount. The pyrites is reduced to $Fe_2S_3$, and 25 per cent. of the sulphur is liberated, so that when the yield of coke attains 75 per cent., the percentage sulphur content is the same as that of the coal. If $Fe_7S_8$ or FeS is formed, the proportion of sulphur in the product is correspondingly reduced. The liberated sulphur may, however, be fixed by the ash-constituents, iron, lime, and magnesia, in which event a larger proportion of this element may exist in the coke than in the original coal. When the coke is quenched, some of the sulphur—though merely a trifle—is expelled.

The injury effected in the blast-furnace by the sulphur in the coke consists in reducing part of the iron to sulphide. To combat this and to remove the sulphur, greater heat is required, as is also the presence of silica and manganese or an excess of lime—the latter to maintain the basic character of the slag. The influence of sulphur on the yield of iron in a blast-furnace turning out 80 tons per diem, is such that the use of coke with 1·5 per cent. instead of 1 per cent. of sulphur will entail an annual loss of about 390*l*. The average sulphur content of German coke is 1 per cent.
—C. S.

*Steel, Siemens-Martin Process, Use of Imperatori's Method in.* P. Klein. Rigaer Ind. Zeit. 1897, **23**, 237; Chem. Zeit. Rep. 1898, **22**, [3], 16.

Experiments on the utilisation of finely-divided iron ore were tried by the author at the Bogoslawski Works in the Ural. Treatment in the blast furnace gave results that were commercially unsuccessful, but application in the form of briquettes in the open hearth was more satisfactory. Originally, pig iron with about 67 per cent. of low carbon forge scrap had been charged; but, owing to difficulties, iron ore had been substituted for the scrap. This ore had added greatly to the quantity of slag produced, and had thus interfered with the action of the flame. The ore contained 86·25 per cent. of $Fe_2O_3$, and required an addition of 9·6 or 19·3 per cent. of carbon to effect its reduction according as $CO_2$ or CO was to be formed. In applying the Imperatori process, ore and charcoal were mixed dry, and then made up to a paste with slaked lime, and formed into briquettes weighing 15—16 lb., which were air-dried and heated. On cooling, these retained their original shape, but consisted of a porous mass resembling spongy iron, containing about 0·18 per cent. C. When the percentage of carbon, which should correspond to the formation of CO, was reduced, more slag was formed, and with a certain minimum proportion of carbon, the slag did not separate from the spongy iron even at a continued white heat. The trials on a large scale showed that, whereas the original pig and scrap, or pig and ore, processes gave a loss of at least 15 per cent. by weight, the Imperatori process showed a gain of 10 per cent.
—W. G. M.

*Iron Plates and Tubes that have Failed in Use, Composition of.* Finkener. Mitt. aus d. k. tech. Versuchsanst. zu Berlin, 1897, **15**, [6], 277—278.

Analyses of 12 samples of boiler plates or tubes are given, all of which samples had failed by corrosion when in use, the pitting being generally quite local. The percentage of carbon in these ranged from 0·03 to 0·31 (average, 0·08), that of silicon from 0 to 0·89 (average, 0·01), of phosphorus from 0·03 to 0·309 (average, 0·102), of sulphur from 0·01 to 0·10 (average, 0·04), of copper from 0 to 0·27 (average, 0·12), of nickel from 0 to 0·13 (average, 0·05), and of manganese from 0·05 to 0·82 (average, 0·33) per cent. Of all these specimens, which exhibited but slight power of resistance to chemical attack, there was not one that was poor in manganese and phosphorus; and of two new tubes from the same works, one contained 0·51 per cent. of Mn and was strongly corroded in spots by sulphuric acid, whilst the other contained only 0·09 per cent. of Mn and resisted attack. Hence, it would seem that these two elements are responsible, directly or indirectly, for the want of resistance to oxygen and water at comparatively elevated temperatures. —W. G. M.

*Matte Smelting at the Hall Mines, British Columbia.* R. R. Hedley. Eng. and Mining J. 1897, **64**, [24], 695.

The matte-smelting furnace in use at the works connected with the Hall mines in British Columbia, is probably the largest furnace of its kind in the world, and from the time it was started, on Sept. 4th, up to Nov. 5th, 11,676 tons of charge have been treated, of which 1,587 tons consisted of barren flux, chiefly limestone. To every ton of charge is added 200 lb. of slag. The matte produced amounted to 1,029 tons, averaging about 49 per cent. of copper by wet assay. The percentage of coke used varies between 14·5 and 16 per cent., according to the quality of the coke and the amount of sulphur in the charge. The concentration effected is about 14·25 parts of charge into 1 of matte, but the furnace has run successfully with a concentration of over 20 to 1. It is stated that the furnace will admit of a very large proportion of fine ore. The character of the ore is chalcopyrite, bornite, tetrahedrite, and kindred minerals in variable gangue, having an average composition of silica, 33 per cent.; ferrous oxide, 9·5 per cent.; manganese oxide, 8 per cent.; lime, 7·5 per cent.; magnesia, 4 per cent.; alumina, 15 per cent.; copper, 4 per cent.; and sulphur, 3·2 per cent. Limestone, the flux generally used, contains about 10 per cent. of silica, and the resulting slag has an average composition of silica, 43 per cent.; lime, 95 per cent.; ferrous oxide, 12 per cent.; manganese oxide, 1 per cent.; alumina, 18 per cent.; copper, 0·315 per cent.; and silver, 1·15 oz. per ton. The dimensions of the furnace are:—Area at tuyères, 144 ins. by 44 ins.; at top of jackets, which are 5 ft. 6 ins. high, 144 ins. by 61 ins.; and at feed floor, 160 ins. by 72 ins. The total height of columns is 12 ft. 6 ins., but the charge is generally maintained 4 ft. lower, varying, however, with conditions of charge, &c. The tuyères, eight on each side, are reduced from 6 ins. to 4¾ ins. The centre of the tuyères is 24 ins.

above the bottom as originally constructed. The bottom is supported by a cast-iron plate resting on six jack screws carried by a truck, and consists of a coil of 2-in. water piping embedded in steep (brasque), and a course of fire-brick on end. On this the furnace makes it own bottom. Provision is made for tapping on the side, but the end tap only is used. The flow is practically continuous, and separation is effected first in a large conical pot and then in a second settling vessel of similar form, but smaller, from which the slag flows into a powerful stream of water. Drawings of the furnace, end- and side elevation, are given. —A. S.

*Gold and Platinum Nuggets and Gold Ingots, Crystalline Structure of.* A. Liversidge. Proc. Roy. Soc. of New South Wales, Oct. 3, 1894, 70—79. (See also this Journal, 1894, 398).

It is stated that the investigation, so far as it goes, tends to prove that gold nuggets do not show that they have been built up of concentric coatings round a nucleus, but that they possess a well-marked internal crystalline structure, and that they usually enclose foreign substances; also that a similar crystalline structure is shown by gold which has been fused. The author, however, does not consider that native gold has necessarily been in a fused condition, but that it has been deposited from solution and usually within veins or pockets in rocks, although if it had been deposited round nuclei, it might still have possessed the crystalline structure which has been observed. A series of 12 photographic plates and etchings is given in illustration. —A. S.

*Gold, The so-called "Selective Action" of Cyanide of Potassium for.* W. A. Dixon. Inst. of Mining and Metallurgy, Nov. 17, 1897.

The author maintains that there is no such thing as the "selective action" of potassium cyanide for gold in weak solutions, and for the baser metallic oxides in strong solutions. Gold in the presence of oxygen has at all times a greater affinity for the cyanide than the baser metallic compounds have, and in the very weak solutions containing dissolved oxygen, the former action chiefly takes place, there being not enough cyanide present to extract the other bodies. In the stronger solutions the same action takes place whilst there is any oxygen present, but the cyanide also dissolves the metallic oxides without the aid of oxygen. Consequently the strong solutions appear to have a greater affinity for the base metals. To thoroughly wet a ton of ore, about 105 galls. or 7,350,000 grains are required. Dividing the co-efficient of solubility of oxygen by 5, to reduce it to that due to the partial pressure of oxygen in air, it can be found that the above quantity of water may contain as a maximum 43,383 grain measures, or 62 grains weight, of oxygen in solution. This quantity would be sufficient to aid cyanide in dissolving 3 oz. 3 dwt. 15 grs. of gold. The cyanide required would make with the above water a solution containing only 0·0047 per cent. of cyanogen.

In practice the above saturation point is never reached, but, on the other hand, very few ores contain 3 oz. of gold per ton, and the ore is re-treated with fresh solutions until the gold is extracted. Furthermore, a greater quantity of cyanide is necessary than that theoretically required by the oxygen present, because the small quantity of gold is spread over a large mass of ore, and the rates of diffusion of the oxygen and cyanide, which should act together, in solution are very different. Finally, the author takes exception to the use of the word "cyanicide" as applied to those substances, such as metallic oxides, which use up and form compounds with the cyanide, but which do not actually destroy it.—A. W.

*Gold from its Cyanide Solution, Influence of the Anodes in Depositing.* E. Andreoli. Eng. and Mining J. 1898, **65**, [4], 100.

It is stated that the Rand Central Ore Reduction Company (Transvaal) is probably the only company in the world which recovers gold electrolytically from the cyanide solutions. This company has developed and improved the process invented by Siemens and Halske for the electrolytic treatment of weak gold cyanide solutions, containing only a few pennyweights of the precious metal per ton, and it is stated that not only tailings, but thousands of tons of old acid slimes are now treated electrolytically, and yield the gold, which cannot be precipitated by the zinc process. At the present time, iron anodes are used in the electrolytic process, but since all base metals are soluble in the cyanide solution, the latter becomes contaminated, and at the most, only 70 or 80 per cent. of the gold is deposited. In a really good electrolytic installation, the cyanide solution should remain clear, and the anodes should be perfectly insoluble, so that they cannot cause contamination of the liquor from which the gold is to be separated. The author recommends the use of peroxidised lead anodes, and states that they will last for several years, will not contaminate the cyanide solution, and can be easily and cheaply manufactured. In the Siemens process, iron anodes are used, which are soluble, and lead cathodes, which have to be melted and cupelled. The author recommends the use, in conjunction with peroxidised lead anodes, of cathodes of iron, or other metal for which gold has a great affinity, and to which it strongly adheres. When the iron plates are sufficiently coated with gold, they are withdrawn from the solution, rapidly dried, and dipped in molten lead, which immediately absorbs the gold, with which it forms an alloy. The stripping of gold from the iron cathodes is complete, and the iron plates may be replaced in the tanks, when they again become coated with gold. In this way, instead of a monthly clean up, the gold can be stripped from the iron cathodes every week or every fortnight, without interrupting the work, since the gilded plates can be withdrawn one after another from the tanks, dipped in the molten lead, and then washed and again put into the solution.—A. S.

*Alloys: Fourth Report to the Alloys Research Committee of the Inst. Mech. Engineers.* W. C. Roberts-Austen. J. Inst. Mech. Eng. 1897, 31—69.

Reference is made to Charpy's work on brasses (this Journal, 1896, **15**, 117). In noting the fact that the annealing of brass may begin at 430° C., and may be very efficacious at 500° C., while the "burning," or rapid diminution in extensibility, of the metal will occur with pure alloys at 900°, or with commercial alloys (which contain 0·15 per cent. of Sn and 0·2 per cent. of Pb) at temperatures below 800° C., the author points out that he and Osmond have shown that a gold-antimony alloy, revealing large crystalline planes after slow cooling, becomes minutely granular at 250° C. The effect is compared with that of annealing cast steel at 800° C.

Experiments have now been made in the eutectics of copper-zinc alloys, and they show that in the case of industrial brasses (those containing less than 45 per cent. of Zn), only three freezing points need be considered. Thus, with the alloy containing 71 per cent. of Cu, there are two freezing points, one of copper at 952° C., and one of the eutectic alloy of copper with the compound CuZn at 896° C. Again, in the alloy with 68·6 per cent. of Cu there are three freezing points, 935°, 883°, and 450° C., whilst in that with 62·6 per cent. of Cu, the upper two of the points merge into one at 892° C., whilst the lower point is constant at 450° C. Attention is drawn to the want of definiteness in the usual statements as to the fusing points of alloys, owing to the failure to grasp the fact that the metal is in a more or less pasty condition through a considerable range of temperature. The exact composition of any eutectic mixture may be found by slowly heating a sample of the original alloy in a stout steel cylinder with a central bore about ½ in. in diameter, provided at each end with a loosely fitting plunger; the whole is subjected to a pressure of about 0·5 ton per sq. in. by means of a hydraulic press bearing upon the plungers. A thermo-couple is inserted in a cavity drilled in the cylinder from outside, and, heat being applied, the temperature at which a portion of the metal is squeezed out in the fluid state is noted; the composition of the eutectic alloy and its temperature of fusion are thus readily found. The temperature is again raised until a further portion is extruded, and so on until finally a comparatively infusible residue is left. In some cases the extruded portions are again fractionated.

The connection between the eutectics and the mechanical properties of the alloys may be traced. The alloys CuZn and $CuZn_2$ are very brittle, and the latter has been shown by Laurie to be a chemical compound. The eutectic, freezing at about 890° C., formed from the alloys with 73 to 60 per cent. of Cu, contained 63 per cent. of Cu, which does not represent any definite atomic proportion, but probably consisted, when solidified, of an intimate mixture of Cu with CuZn. The mixture of these hard and soft substances produces great strength, but the presence of any considerable proportion of the eutectic diminishes the extensibility of the material. Hence the most extensible alloy is that which shows just the first appearance of the eutectic, *viz.*, that containing 71 per cent. of Cu, and the strongest of the whole series is that which has the composition of the eutectic itself. The alloy with 63 per cent. of Cu has naturally but one freezing point; those with less copper than this show a great diminution in the tensile strength and extensibility, probably because the compound CuZn is unaccompanied in them by free copper. It is probable, however, that this compound is soluble in one or more of the other compounds richer in zinc that are known to exist. With the alloys containing more than 70 per cent. of Zn, there are three eutectics, freezing respectively at about 680°, 580°, and 420° C., but they are apparently formed by compounds less brittle than CuZn and $CuZn_2$, so that the material is not quite so weak as those containing 60 to 70 per cent. of Zn.

It is well known that the addition of 1½ per cent. of iron (as in Aich's metal) to a brass with 61 per cent. of Cu greatly increases the strength of the metal. The study of the cooling curves of such metals shows that the iron combines with an eutectic fusing at 450° C. in the non-ferrous alloy, and forms a less fusible compound, so that the halt in the cooling curve is then absent. A source of weakness is thus removed, and at the same time the fusing point of the mass is raised. The obliteration of the low eutectic causes the Aich's metal to retain its strength at temperatures at which brass becomes very brittle, as is shown in the following table:—

| Temperature of Test. | Tensile Strength per Sq. In. | |
|---|---|---|
| | Brass.* | Aich's Metal.* |
| ° C. | Tons. | Tons. |
| 20 | 20·7 | 25·6 |
| 100 | 13·6 | 22·2 |
| 200 | 12·8 | 17·9 |
| 250 | 10·4 | 15·8 |
| 300 | 7·3 | 12·4 |
| 350 | 6·8 | 11·2 |
| 400 | 7·1† | 9·7 |
| 450 | 3·6 | 5·1 |
| 500 | 2·8 | 4·1 |

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* Brass = Cu, 61·2; Zn, 38·8 per cent. Aich's metal = Cu, 59·8; Zn, 37·9; Fe, 1·5; Sn, 0·8 per cent.

† Increase due to experimental irregularities.

It may be affirmed as a general principle that the obliteration of any eutectic which is much more fusible than the mass as a whole (but not otherwise) will lead to an increase in strength. Great stress is laid on the necessity, so seldom recognised by experimenters, of determining all the eutectic points of a series of alloys, instead of only one. Thus Gautier, from his cooling curve of Ag.Zn alloys deduced the inference that zinc is isomorphous with a certain compound of the two metals, containing about 60 per cent. of Ag, and that the curve of cooling from the temperature of fusion of the compound to that of zinc is therefore a straight line. But the existence of another eutectic with a fusing point but little higher than that of zinc negatives this conclusion. Again, in examining the effects of impurities, it is desirable to ascertain in what condition a small quantity of added metal exists in the mass before undertaking elaborate tests of the mixture.

The question of the elastic limit requires much study. It has been shown by Bauschinger that even the smallest load must produce permanent set. Hartmann considers that a stressed material is "polarised" in the direction in which distortion occurs, and that it tends to become "depolarised" by returning to its original form. Such polarisation is caused by an intermolecular flow or slip, usually at an angle of about 45° with the direction of the stress, causing the hardness produced by "cold-working," and evidenced by the lines of Luders which are shown to run spirally along a stressed bar of well-polished metal. The addition of impurities to the metal, unlike cold-working, alters the atomic arrangement of the molecules.

The author's work on solid diffusion of metals (this Journal, 1896, **15**, 596) is reviewed and it is pointed out that the rapidity with which the diffusion occurs supports the "gaseous theory" of metallic solutions, which supposes that in very dilute solutions the atoms of the foreign element are so widely separated that they exert pressure and act in the same way as would the atoms of a gas. It also indicates the possibility of structural changes in metals used in machinery and in engineering construction even at the ordinary temperature.

Pictet, in 1879, showed that there is a simple relation between the atomic weights, the co-efficients of expansion, and the melting points of all metals. Since the rate of increase of amplitude at any given temperature is obtained by multiplying the thermal co-efficient of linear expansion by the cube root of the atomic volume, it follows that the absolute temperature of fusion of a metal must be intimately connected with the atomic volume. Many pure metals and alloys have now been examined in this connection. The test-bars were rods 6 ins. long by $\frac{3}{16}$ in. in diameter, and were strengthened as far as possible by careful hand-hammering. Only in this way could constant results be obtained. It was found that many alloys fall on a curve with melting point and tenacity as co-ordinates. Pure metals and some alloys, however, agree fairly well with another curve; but in this case the metals or alloys considered in relation to their melting points are weaker than those in the former curve. It is, however, difficult to reduce the pure metals to a standard condition for comparison, and for this reason they are not so well suited as alloys for studying the relation in question. It is remarkable that when two alloying metals have approximately the same atomic volume, the resulting alloy is weaker than could have been anticipated from its melting point, when judged as an alloy. And when the atomic volumes are identical, the alloy falls upon the curve of the pure metals. Alloys which are chemical compounds usually do not fall upon either curve; but as they are brittle, the irregularity is probably due to the inability of the test-piece to yield when under stress. The curve for alloys, if produced, would pass through a hardened steel having a melting point of 1,400° C. and a tenacity of 90 tons per sq. in. Both curves are shown.

*Improvements in the Recording Pyrometer.*—In previous readings with the pyrometer, great delicacy was obtained, but at the expense of the constancy of the zero, owing to the strain put upon the suspending wire of the mirror by the large angular deflections obtained. This difficulty is now met by opposing the current from the couple (which otherwise would cause a large deflection of, say, 50°) by that of a large Clark cell, the amount of this current being controlled and accurately measured by a special potentiometer. In this way, at a temperature, for example, of 231° C., the current produced by 204° C. would be neutralised by that of the Clark cell and measured by the potentiometer, whilst that caused by the remaining 27° C. deflects the galvanometer mirror. The zero is thus more constant, but the sensitiveness of the galvanometer is not impaired, for its resistance is not increased. This delicate method of recording requires special arrangements for actuating the sensitised plate.

*Comparison of the Thermo-junction with the Air-Thermometer.*—Several comparisons have been made between the two methods of pyrometry, with results favourable to the accuracy of the air-thermometer; but there is still an uncertainty, amounting to about 10° C., as to the true melting point of gold. Heycock and Neville gave it as 1,061°·7, and Holborn and Wien as 1,072° C.; but Holman, Lawrence, and Barr, accepting the latter number as correct, found the melting point of copper to be 1,095° C., or 13° higher than Holborn and Wien made it. The author is experimenting in this field with a new air thermometer.

—W. G. M.

*Pigments (Zinc and Blanc Fixe), Preparation of, from Zinciferous Residues.* W. Stahl. Berg- u. Hütt. Zeit. **71**, 1; Chem. Zeit. Rep. **22**, [4], 28.

*See under* XIII. A., *page* 255.

## PATENTS.

*Amalgams containing Aluminium, Magnesium, or Zinc; Impts. in or in connection with the Preparation of.* W. G. H. Wolff, Berlin. Eng. Pat. 2350, Jan. 28, 1897.

Ternary alloys having the formula $Al_{111}ZnHg$, or $Mg_{125}ZnHg$, or $Mg_{125}AlHg$, are prepared by precipitating mercury and zinc on cut aluminium or magnesium, or by precipitating mercury alone on the zinc alloy $Al_{111}Mg_{125}Zn$, whereby the zinc in the magnesium alloy may be replaced by aluminium. In the provisional specification, four claims are made, instead of the one quoted above from the complete specification, *viz.*, the manner of preparing alcoholates of aluminium and magnesium by means of zinc and mercury; manner of generating hydrogen by the action of zinc chloride and corrosive sublimate on sheet aluminium in aqueous alcoholic solution; the preparation of the ternary alloy first named; and the method of reducing organic bodies by such alloys.—W. G. M.

*Copper, Pure; Process and Apparatus for obtaining, in a Single Operation.* La Société des Cuivres de France, Paris. Eng. Pat. 7021, March 17, 1897. (Under Internat. Convention.)

A converter lined with refractory material has its mouth and the tuyères opposite to one another, and a side chamber or pocket centrally between them. The converter may be turned completely over, and may be so placed that the entrance to the pocket is either above or below the level of the fluid material resting in the belly of the converter when the latter is "turned down." The pocket has two openings (at the top and bottom respectively), which can be closed at will, and is so shaped that any portion of the charge, which enters it when it is beneath the fluid level, will be retained after it has been turned up again. The tuyères are so arranged that they project upwards beyond the bottom lining, and there is in consequence an annular depression around them. White-metal (copper matte) is blown in this converter until the foreign metals present, have separated and sunk into the annular depression, where they collect, unaffected by the blast, until the vessel is turned into such a position that they run into the pocket, after which the converter is again turned up, and the blow is continued until the copper is completely reduced. The metallic impurities in the side chamber are, meanwhile, run out through the lower of the two vents provided for the purpose. The converter may also be used as a tilting reverberatory furnace, in which case its mouth is brought up against a lateral opening connected with a chimney. —W. G. M.

*Staining or Colouring Metals and other Materials, A New Process for.* F. Moss and S. J. Davis, Edgbaston. Eng. Pat. 8626, April 5, 1897.

The metals, wood, stone, &c., to be stained are coated with or dipped into a solution of the salts of one or more metals, *e.g.*, copper and iron, then dried at 100° F. for 20—30 minutes, steamed for a like period, and again dried. Finally, they are boiled in water for the same length of time, and mechanically polished, if desired.—R. B. B.

*Ores, Treatment of, Impts. in, and Apparatus therefor; the Invention being also Adapted for the Treatment of Sewage.* J. Poole, Coolgardie, W. Australia. Eng. Pat. 14,575, June 16, 1897.

The ore pulp is conveyed by an elevator into a series of long, shallow tanks, with an overflow at the opposite end of the system, through which it passes to a series of shallow tanks placed each at a slightly lower level than the last, until it is discharged into a series of deep settling tanks. While passing through the shallow vessels, it is constantly stirred by rakes connected to an eccentric, so that they have a reciprocating motion imparted to them. Each rake is suspended at any desired height from the bottom of the vessel by flexible cord passing over a pulley to a winch. The rakes may be made hollow, so that air or gases may be passed through the liquid while they are in operation. Cyanide solutions may be employed instead of water in these tanks, or, if free milling ores are being treated, mercury may be placed on the bottom of the tanks. The tailings are allowed to settle in the first pair of depositing tanks, and, passing into a sump, are elevated to a second pair, and thence into a third pair of similar depositing tanks. Whilst in the last-named vessels, the tailings may be subjected to the further action of cyanide, if desired. The apparatus is also adapted for the treatment of sewage with air or chemicals, and for the settling and decantation of same.—W. G. M.

*Iron Castings, Annealing; A New or Improved Process of.* C. M. White, London. From the King Annealing Process Company, Belleville, Ill., U.S.A. Eng. Pat. 28,568, Dec. 3, 1897.

The [white-iron] castings are embedded in a mixture of manganese alloy, such as ferro-manganese, and refractory material, such as sand or mill-scale, with or without all or any of the following in admixture:—A readily combustible substance (*e.g.*, sawdust), a volatile body (*e.g.*, turpentine), an acid (*e.g.*, vinegar), and a greasy material (*e.g.*, suet). Or, they may be painted over with an adhesive substance (*e.g.*, suet), and then coated with the manganese alloy, after which they are to be packed in the refractory material, with or without the aforesaid additions. After being thus packed they are annealed at a suitable temperature. A mixture especially recommended consists of: Sufficient silicious sand to enclose the castings, with 5 per cent. of an 80 per cent. ferro-manganese, 10 per cent. of sawdust, 5 per cent. of tallow or beef suet, and sufficient vinegar and turpentine to render the mixture thoroughly moist throughout. There are 14 claims.—W. G. M.

*Ores or Compounds, Pulverised or Finely-divided; An Improved Method or Process of Extracting Metals from.* J. Rudolphs and J. Landin, both of Stockholm. Eng. Pat. 28,896, Dec. 7, 1897.

The ore is mixed with finely-divided carbon and with materials containing nitrogen and hydrocarbons (*e.g.*, dried blood and coal-tar), such as will produce caking of the mass on moderate heating. After compression, the blocks are heated to about 400° C., and are then ready for charging into the smelting furnace. The fluxes necessary to the subsequent smelting may be balled with the rest of the charge. The products of distillation obtained during the heating stage may be condensed for use again. Instances of the application of this process to bog-iron ore, to alumina (for the electrolytic smelting process), and zinc oxide, are quoted.—W. G. M.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (A.)—ELECTRO-CHEMISTRY.

*Lead Accumulators, Impts. in.* F. Foerster. Chem. Industrie, 1898, **21**, [3], 47—51.

An accumulator is in theory a reversible voltaic cell, which shall store without loss, electrical energy supplied to it, and give that energy out again without loss, when required. Many cells, Daniell's for example, are theoretically accumulators, but are not reversible in practice, for mechanical or physical reasons; and the only cell to be considered in practice is the lead accumulator. This, too, is only capable of restoring about 80 per cent. of the energy supplied to it—a defect inherent in its nature, and arising from the fact that the chemical reactions during charging are not exactly the reverse of those during discharge. But the lead accumulator will give out practically the whole of the energy which it actually takes in from the charging current; hence improvements are to be looked for, not in the direction of increased efficiency, but, on the one hand, in the increase of the capability of the cell to retain its charge for a long while without diminution, and on the other, in the increase of its capacity, and of its power to retain that capacity unimpaired under widely varying conditions of charge and discharge.

By the use of pure acid, and the avoidance of great differences of concentration in different layers, together with care in manipulation, so as to avoid short-circuiting in the cell, very satisfactory results have been attained as far as the retention of charge is concerned; though in cells consisting of a paste of red lead supported by a framework of lead, the continual changes of volume during the conversion of that into lead-sponge or peroxide, &c., tend to disintegrate the plates and form a source of danger from short-circuiting. Many of the various devices (specially shaped framework, porous covers or cases for the plates, canals to facilitate the escape of gas) invented to overcome this difficulty, are very successful.

With insufficiently porous material, the inner portions are never reached, which not only means diminished capacity, but a diminution of the available current that can be used, both in charge and discharge. It means also an increase of the liability to disintegration if this current be exceeded—a disadvantage especially where accumulators are used for such purposes as street traction. Attempts at improvement in this direction have mostly gone on the lines of increasing the fineness of division of the spongy lead, either chemically or mechanically; the Gelnhausen Electric Company, for example, grind the spongy lead with pumice-stone powder, whilst Pollak forces molten lead into the interstices of a powdered substance, such as common salt, which is afterwards dissolved out of the solidified mass. The addition of manganese dioxide to the active mass, is not, on the whole, to be recommended, as slow discharge is apt to take place in an open circuit. Experience has shown that the addition of the various organic substances which have been suggested from time to time, does not prolong the life of a cell, though the complete oxidation and disappearance of such organic substances, should leave the lead in a state of favourable porosity.

For securing good contact between the support and the active mass in the positive plate, no plan is so good as "formation" by the old Planté process: the time needed for this, however, is prohibitory. The Hagen manufactory carry this process on for about ten weeks, and then cover the partially "formed" plates with the ordinary paste; whilst, according to Luckow, a few days suffice to produce a "formed" plate, if the electrolysis of a very dilute, neutral, or faintly acid or alkaline solution of a salt of the alkali- or alkaline earth metals be carried on by a moderately strong current in one direction, between lead plates.

The lessening of the weight of an accumulator of given capacity is also a problem to be worked at. Gülcher has made an important advance in this direction. He makes his plates of a sort of fabric, the warp being of lead wire and the weft of glass wool. The finely-divided active mass adheres firmly to this, and the finished plates are cased in glass wool, which insulates them, and lessens the risk of disintegration from mechanical causes, without impeding too much the circulation of the liquid. This accumulator has not more than one-third to one-half the weight of an ordinary cell of equal capacity.—J. T. D.

*Perchlorates, Production of, by Electrolysis.* F. Winteler. Chem. Zeit. **22**, [11], 89.

*See under* VII., *page* 244.

## PATENTS.

*Calcium Carbide, Impts. in Electric Furnaces for Use in the Manufacture of; applicable also for obtaining Metals from their Salts.* [*Reversible Furnace.*] L. Bresson and P. Pacotte, Lyons, France. Eng. Pat. 2790, Feb. 2, 1897.

The furnace consists of a graphite crucible surrounded by magnesia or other non-conducting substance, and provided with journals or axle ends, enabling the same to be reversed to remove the product. The anode is formed by a metallic breech, which is in perfect contact with the base of the crucible, and the cathode by several carbons equally distributed over the surface of the furnace, or by a single carbon circularly displaceable around the axis of the furnace. The cathode can be quickly raised or lowered by means of a rack, and the arc is kept constant by connecting a motor as a shunt on the furnace, the motor raising or lowering the carbons as required. To diminish the undue excess of carbon required in the manufacture of calcium carbide, the carbon and lime are agglomerated into small balls, and fed into the furnace in this form instead of in a powdered state. – G. H. R.

*Secondary Batteries, Elements for; Impts. in and in the Manufacture of.* D. G. Fitz-Gerald, London. Eng. Pat. 5805, March 4, 1897.

The object is to construct a cheap, light, and durable secondary battery more particularly adapted for electrical locomotion.

The claims relate (1) to a "method of coating lead sheets with electrolytically deposited and mechanically consolidated crystalline spongy lead by subjecting the sheet, whilst still moistened by the electrolyte, to the pressure of rollers"; and (2) . . . . "causing the endless sheet [of lead] to travel continuously between anodes in the electrolytic bath, and at the same time subjecting it, whilst still moistened with the electrolyte, to pressure of rollers so as to simultaneously deposit and consolidate the spongy lead upon the sheet . . ." Apparatus for doing this is shown.—J. C. R.

*Electric Furnaces ["Continuous"], Impts. in.* R. C. Contardo, Sèvres, Seine, France. Eng. Pat. 3288, Feb. 8, 1897.

"The object is a continuous electric furnace, so contrived that the whole of the heat generated by one or more voltaic arcs, as well as that arising from the rich combustible gases produced by the reduction of the oxidised mineral under treatment, can be utilised." The claims refer to the drawings, and it is stated by the patentee that "a simple furnace constructed according to my invention comprises essentially a reduction chamber for the mineral, an electric heating chamber, and a collecting chamber for the metal." For this furnace, materials are to be used of "ordinary fire-resisting or refractory" nature, or of materials cooled by a circulating cooling fluid.—J. C. R.

*Cell or Battery, Dry Primary: An Improved.* W. Shaw, Edinburgh. Eng. Pat. 6674, March 13, 1897.

"A dry cell, having a zinc casing, constituting the negative element, a carbon rod built round about with plumbago and manganese dioxide constituting the positive element, and hydrochloric acid killed with zinc, ammonium chloride, and acetic acid the electrolyte" [absorbed by plaster of Paris, paper pulp, sawdust, &c.].—J. C. R.

*Galvanic Batteries, Carbon Plates for: Impts. in.* R. W. Atkinson and S. F. Walker, Cardiff. Eng. Pat. 12,028, May 15, 1897.

It is proposed to render carbon plates more durable under the action of corrosive liquids used in batteries, by saturating them with sulphur, "filling up the pores." The plates are saturated in a bath of molten sulphur direct, or they are first saturated with a volatile liquid (chloride of sulphur, carbon tetrachloride, water, &c.) to remove the air, and then immersed in the sulphur bath until all the liquid is driven out. These sulphur-saturated plates may have a metallic connection made to them electrolytically, using them first as anodes in copper sulphate or other solution "until the sulphur is removed from that portion of the plate upon which it is wished to deposit the metal," and then as "cathodes in a solution of the salt of the metal to be deposited."—J. C. R.

*Ozone, Impts. in and in Means for the Production of.* E. Bohm, Barnsbury. Eng. Pat. 7177, March 19, 1897.

The apparatus consists of a series of rows of open-ended glass tubes with internally and externally disposed electrodes enclosed in a chamber, and connected up respectively to the opposite terminals of an electric circuit. These tubes are contained in a suitable frame of some non-conducting material, and the chamber is provided with inlet and outlet pipes. The electrodes may consist of loops of wire, or be formed of wire gauze as described in Eng. Pat. 615, Jan. 1897 (this Journal, 1898, 159).—G. H. R.

*Waterproofing and other Purposes, An Improved Compound for; applicable also as an Acid-resisting and Insulating Material for Electric Cells.* C. Baswitz, Berlin. Eng. Pat. 14,469, June 15, 1897.

The typical composition of this material is as follows:—Asphalt, 150 parts; paraffin, 50; vaselin, 30; and petroleum spirit, 770 parts. The value of the vaselin lies in its forming a homogeneous mixture with the other ingredients, and in its power to "sterilise what is mixed with it." The compound can be stained with any suitable dye; and it is adapted for waterproofing cotton or linen garments, awnings, tents, asbestos for insulating batteries, and wool or silk fabrics.—F. H. L.

*Electric Accumulators [Planté], Plates or Electrodes for; Impts. in.* H. H. Lake, London. From C. Francke, Berlin. Eng. Pat. 29,273, Dec. 10, 1897.

Three claims. 1. "An electrode, the characteristic feature of which consists in the arrangement of ribs [bent upward], the cross sections of which are limited by curved lines, the object being to increase the active surface of the electrode as compared with electrodes having straight ribs of equal thickness."—J. C. R.

## (B.)—ELECTRO-METALLURGY.

### PATENTS.

*Copper and other Metals, Impts. in and connected with Apparatus employed in the Electro-Deposition of.* W. E. Heys, Manchester. From E. Dumoulin, Paris. Eng. Pat. 2711, Feb. 2, 1897.

Improvements are claimed in apparatus for the electro-deposition of copper and other metals on rotary cathodes, moving in contact with "membranes" (brushes, burnishers, and the like), and consist of means for imparting to the membranes a longitudinal reciprocation; and also of means and appliances whereby the mandrel (cathode) is suspended between two adjustable spindles, one or both of which is or are pressed against the mandrel by springs, and one or both of which may be withdrawn longitudinally for the removal of the mandrel.—A. S.

*Copper and other Metals, Impts. in and connected with Apparatus for the Electro-Deposition of.* W. E. Heys, Manchester. From E. Dumoulin, Paris. Eng. Pat. 2712, Feb. 2, 1897.

The patentee claims, in apparatus for the electro-deposition of copper and other metals, improvements consisting in:—(1.) A switch arranged in connection with each vat, by the operation of which, and of resistance in combination therewith, the whole or any part of the current may be conducted through the vat with which it is connected, or the whole or any part of the current may be diverted to the next vat. (2.) Means for circulating and controlling the temperature and condition of the electrolyte. (3.) A partitioned cistern having the top edge of its partition level with the normal electrolyte level, and with one side in communication with the bottom of the depositing vat, and its other side in communication with the circulation pipe, and having or not having a bye-pass connection between the up-flow and down flow pipes; and (4) a series of filtering tanks for the purification of the electrolyte.

—A. S.

*Calcium Carbide, Impts. in Electric Furnaces for Use in the Manufacture of; applicable also for Obtaining Metals from their Salts.* L. Bresson and P. Pacotte, Lyons, France. Eng. Pat. 2790, Feb. 2, 1897.

*See under* XI. A., *page* 252.

*Metals, Impts. in Apparatus for the Electro Deposition of. [Rotatory Drum.]* J. E. Hartley, Birmingham. From F. W. Zingsem, New York, U.S.A. Eng. Pat. 3526, Feb. 10, 1897.

The drum or cage which contains the articles to be plated, is given an oval form, so that the movement of the articles may be accelerated periodically, and, to further insure this, the shaft of the drum carries cams resting on rollers, and the bearings allow of its rise and fall. The anodes are carried on a removable frame supported between the two ends of the drum, and the current is conveyed through a loose non-rotatory collar which bears on a fixed collar carried by the shaft.—G. H. R.

*Zinc-bearing Ores and Zinc-bearing Products, Impts. in the Combined Electrolytic and Leaching Treatment of. [Carbon Anode.]* E. A. Ashcroft, Newcastle, New South Wales. Eng. Pat. 7532, March 23, 1897.

This process, which is an improvement on those described in Eng. Pat. 13,850, July 1894 (this Journal, 1894, 957), and Eng. Pat. 13,534, July 1895 (this Journal, 1896, 658), consists in the employment of carbon instead of soluble iron anodes, and the solution of ferric sulphate or chloride employed in leaching the zinc ores, is not prepared electrically, but is only regenerated in the anode compartments. The zinc-bearing solution, free, or nearly free from iron, is first circulated through the cathode compartments of the tank, and then it is mixed with ferrous chloride or sulphate solution, and is passed into the anode compartments, where the iron salt is raised to the ferric state. Thence a suitable quantity of the solution is led into vats containing scrap iron, and, when a sufficient quantity has been dissolved, it is returned to the anode compartment, where it is again raised to the ferric state. The operation is thus cyclic.

—G. H. R.

# XII.—FATS, OILS, AND SOAP.

*Wool-Fat, The Composition of.* L. Darmstaedter and J. Lifschütz. Ber. **31**, 97—103.

In this paper, the authors continue their researches on the composition of wool-fat. (This Journal, 1896, 206, 460, 548; and 1897, 150.) They find that the softer portion of the wool-fat contains neither lanoceric nor lanopalmic acids, thus differing from the harder portion or "wax." It contains, however, a considerable quantity of an unsaturated acid, which constitutes about 40 per cent. of the acids present.

By shaking out the saponified soft fat with petroleum spirit, and evaporating the latter, a yellow, amber-like body is obtained, which is readily soluble in hot and cold alcohol, although it contains carnäubylic and cerylic alcohols. A characteristic constituent of the soft fat is the so-called isocholesterol, which is not found in wool-fat "wax." To obtain this body, the above-mentioned crude yellow mass is heated for some time with 5—10 times its volume of methyl alcohol, and then allowed to stand in the cold for about 10 hours. A portion remains insoluble and is filtered off, the filtrate containing a body having a lower melting point, and which the authors intend to investigate further. The insoluble portion contains the "isocholesterol." After further purification, this body is obtained as a white crystalline powder. It is a neutral substance, insoluble in water, but readily soluble in hot alcohol, from which it is not precipitated on cooling. The air-dried substance melts at 120°—121°, but when dried at 80°, it loses 2·7 per cent. of water, and its melting point is raised to 137°—138°. An analysis of isocholesterol indicated the formula $[(C_{26}H_{45}O)_2O]$.

From wool-fat "wax," the authors obtained about 2 per cent. of solid alcohols. About half of these consisted of cerylic alcohol. An acid was also obtained which closely resembled cerotic acid, although its identity with the cerotic acid of beeswax is somewhat doubtful.—W. P. S.

*Fats, The Cause of the Rancidity of.* J. A. Mjöen. Forschungs Ber. 1897, **4**, 195—203.

The author's experiments confirm the conclusions of other observers as to the rancidity of fats being due to an oxidation process caused by the action of air and light, and accompanied by the formation of free acids and a decrease in the iodine value. But, contrary to Ritsert's experience (Natur. Wochenschrift, **5**, 136), he finds that light, although playing a considerable part, is not an essential factor of the decomposition process, oxidation being caused in fats subjected to a current of air and protected from the light. Further, there is a distinct difference between the chemical changes produced by the action of air alone and that caused by the combined action of air and sunlight. For instance, a sample of butter with an acid value 1·2 and iodine value 32·7, after exposure to sunlight for three days had the acid value 10·7 and iodine value 30·9; whereas, when the same butter, kept at about 60° C., was subjected to the action of a current of air alone, about five weeks were required before a similar acid value was reached, and the iodine value had then fallen to 19·8. There was also a difference in the appearance of the butter; that exposed to the sun becoming deep yellow, whilst that subjected to the current of air maintained its original colour for two or three weeks, and then suddenly became snow-white. It is suggested that this change was possibly due to enzymes, similar to those which cause the alteration in freshly-cut apples, &c., and which have been termed "oxydases" by Weigert.

Cod-liver oils behaved in manner analogous to butter, turning rancid after being subjected to a current of air for several weeks, and in some cases becoming so thick that they could not be poured from the vessel. Olive oil, however, when treated in the same way, showed no alteration in composition after 28 weeks.

The author considers that he has proved positively that bacteria and moulds have no action upon fresh fats, but he is not prepared to state that they play no part in subsequent changes when once a decomposition process has commenced.
—C. A. M.

*"Table Oil": Is the Term to be Limited to Pure Olive Oil?* Chem. Rev. Fett- u. Harz-Ind. **4**, [23], 318—319.

This question has recently been eagerly discussed in Austria-Hungary. Formerly the municipal authorities at Buda-Pesth restricted the term to pure olive oil, but extended it at the request of the Local Chamber of Commerce, and now their example has been followed by the commission appointed to draw up a *Codex alimentarius Austriacus*. The Zeits. für Nahrungsmittel-Untersuchg. supports this decision, and holds that to confine the term "table oil" to any single oil is unjustifiable, there being a number of other oils equally as well suited as olive oil for edible purposes—some, in fact, superior, owing to lesser liability to rancidity. Besides, mixtures of sesamé, earth-nut, cotton-seed, and other oils with olive oil, have all along been imported into Austria as pure "table oil," the Customs tests not having been able to detect admixtures of 10 to 15 per cent. of such oils.—C. S.

*Glycerin and Dioxyacetone, A New Total Synthesis of.* O. Piloty. Ber. **30**, [19], 3161.

*See under* XXIV., *page* 283.

*Olive Oil, Portuguese: Coloration with Baudouin's Reagent.* A. J. F. de Silva. Bull. Soc. Chim. 1898, **19**, [2], 88.

*See under* XXIII., *page* 275.

*Fat Analysis, Uniform Methods of.* F. Ulzer. Chem. Rev. Fett- u. Harz Ind. **4**, [22], 299.

*See under* XXIII., *page* 276.

*Soap, Analysis and Valuation of. Methods fixed by the Société Suisse des Chimistes Analystes.* J. Suisse de Chim et Pharm. **35**, 491; Bull. Assoc. Belge des Chim. **11**, [7], 191.

*See under* XXIII., *page* 276.

### PATENTS.

*Oils for Pharmaceutical, Veterinary, Technical, and other Purposes; Impts. in and connected with the Preparation of Mineral and Vegetable.* W. E. Pearson, Hamburg. Eng. Pat. 2662, Feb. 1, 1897.

In this process, vegetable oils, petroleum residues, resinous oils, &c., are mixed with 10—20 per cent. of oleic acid, and purified by washing with sulphuric acid (1 per cent. solution) and afterwards with dilute soda ($\frac{1}{3}$ per cent.). The oils are then dried, preferably by heating and stirring. Air may be blown through the oil to hasten the treatment. The oils thus prepared can, it is said, be used as solvents for substances otherwise insoluble. They are simply "shaken in the prepared oils, without pressure or heat." A 10 per cent. alcoholic solution of "strong basis" is added at a temperature not exceeding 80° C., and the mixture is allowed to cool. The resulting compound may be used as a liniment, with the addition of any drug or chemical easily dissolved by the oils.—W. P. S.

*Linseed Oil, An Improved Method of and Apparatus for Bleaching.* R. W. English, Buffalo, U.S.A. Eng. Pat. 9799, April 17, 1897.

The method claimed, consists in subjecting the oil to a temperature sufficient to bleach it, an arrangement of tanks and heating apparatus for effecting this purpose being also claimed.

The oil is first heated in a tank by means of steam pipes to a temperature of 300° F., and then passes down into the hot chamber through a pipe which branches out into several arms near the bottom of the chamber. These arms are perforated with small holes, and allow the oil to be equally distributed over the bottom, where it is heated to 600° F. The oil rises in this chamber, and is drawn off into the cooling or collecting tank.—W. P. S.

*Oil, Impts. in Apparatus for Treating Flax and other Oleaginous Seeds for the Extraction of.* A. Euston, St. Louis, Missouri, U.S.A. Eng. Pat. 18,550, Aug. 10, 1897.

The apparatus claimed consists of a steam-jacketed cylindrical vessel which has a height of 16 ft. and an interior diameter of 2 ft. A vertical rotating shaft extends through the vessel, having attached to it at certain distances, a series of rakes or agitators. Below each set of rakes is an arm or cross-piece, serving as a brace to the shell of the vessel, and having upright arms to aid in mixing and dividing the mass of crushed seed. Steam pipes project into the interior of the cylinder at suitable intervals. Tubes are also provided for the insertion of thermometers. The top of the vessel may be left open; the lower end is closed, and is fitted with a sliding door or delivery valve. The lower bearing of the revolving shaft is in the centre of the plate closing the lower end of the cylinder.—W. P. S.

*Oil, Impts. in the Treatment of Oleaginous Seeds for the Extraction of.* A. Euston, St. Louis, Missouri, U.S.A. Eng. Pat. 28,206, Nov. 30, 1897.

According to this invention, the seeds are crushed without injuring or bursting the oil cells. This crushing is effected between rollers carefully adjusted so that the seeds are merely "fractured, but not crushed so as to injure the integrity of the oils cells." The crushed seed is then heated in a suitable vessel with water, and finally pressed, to extract the oil.—W. P. S.

*Soap, Scented, Antiseptic, Medicinal, or Multi-coloured or Variegated: Impts. in the Manufacture of, and in Machinery or Apparatus therefor.* P. Villain, Warrington. Eng. Pat. 7228, March 19, 1897.

The claims of this patent are for the insertion at the centre of the soap tablet of a container of wood or other material filled with perfume, or of a block of odoriferous wood, and for a process for manufacturing fancy soaps containing coloured soap, perfumes, &c., consisting in the production of a hollow bar of soap, and filling the same with soap of another kind.

The apparatus for producing this hollow soap is also claimed. In this apparatus the soap is moved through a conical vessel by a revolving helix, and passes through a perforated plate at the smaller end of this vessel into a second conical chamber in which is placed a central rod. The soap issues from this chamber by an annular opening, the upper end of the central rod terminating in the centre of this opening.—W. P. S.

*Soap, Bleaching.* L. Grote, London. Eng. Pat. 11,241, June 11, 1897.

The claims are for a process of manufacturing soft and hard bleaching soaps. The soft soap is prepared by mixing solutions of caustic soda or potash, calcined soda, salt, soluble glass, chloride of magnesium, and cocoanut oil, and boiling,—stirring well during the operation.

The hard soap is obtained by mixing together in the cold, solutions of caustic soda or potash, chloride of magnesium, soluble glass, and cocoanut oil.—W. P. S.

*Saponification, A New or Improved Electrolytic Method of and Apparatus for.* A. L. C. Nodon, L. A. Bretton-neau, and P. D'Alton Shee, Paris. Eng. Pat. 22,129, Nov. 19, 1897.

The apparatus claimed consists of a truncated conical trough, which may be made of masonry, wood, or enamelled sheet iron. A vertical spindle passes through the centre of this vessel, having attached to it perforated blades, which act as stirrers. Vessels made of porous material are placed above the trough, and contain a solution of soda lye. They are provided with plates of cast iron, serving as positive poles, whilst the spindle is connected with the negative pole of a source of electric energy. The upper blades of the stirring device are fitted with wire brushes, which rub on the bottoms of the porous vessels. The temperature of the contents of the trough is kept at 90°—100° C. by an arrangement of steam- or hot-water pipes. A blow-off cock at the bottom of the trough serves to empty the latter. The oil to be saponified is placed in the trough with water, and a current of electricity passed through the apparatus, the stirrers being also set in motion. A neutral soap is formed in about seven hours. The difference of potential necessary for the working, varies between 2—4 volts, the current required being about 100 ampères per cubic metre of the substance to be made into soap. Some fats require an excess of soda; this excess may be removed by reversing the current.—W. P. S.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

*Pigments (Zinc and Blanc Fixe), Preparation of, from Zinciferous Residues.* W. Stahl. Berg- u. Hütt. Zeit. **71**, 1; Chem. Zeit. Rep. **22**, [4], 28.

When zinciferous pyrites residues (with 7—10 per cent. of Zn) are treated by chlorination roasting, the resulting lyes may be profitably worked up for zinc pigments. After precipitating gold, silver, copper, lead, &c., by metallic zinc, and throwing down the residual metals by sodium sulphide, the clarified lye is ready for treatment in any of the following ways:—*Zinc-white* may be thrown down by sodium carbonate, the precipitate being calcined in a muffle. *Zinc sulphide* can be precipitated by sodium sulphide from the boiling liquor. It is ground and lightly calcined after being filtered. To prepare *Lithopone I.*, an amount of barium sulphide equivalent to the zinc present is added to the lye, whereupon the following reaction occurs:—

$$Ba(HO)_2 + Ba(HS)_2 + 2ZnCl_2 + 2Na_2SO_4 = \\ 2ZnSBaSO_4 + 4NaCl + 2H_2O.$$

An inferior lithopone (zinc 19·71, barium sulphate 70·59) can be recovered from the residual lye from the above zinc sulphide process by adding zinc chloride lye thereto, and precipitating with barium sulphide, 70·59. *Blanc fixe* is obtained by treating with the product obtained by roasting an inferior variety of heavy spar with carbon, all the filtrates, obtained from the above products and chiefly consisting of sodium sulphate. The following reaction occurs:—

$$Ba(HO)_2, Ba(HS)_2 + 2Na_2SO_4\,aq. = \\ BaSO_4 + 2NaHO + Ba(HS)_2 + Na_2SO_4\,aq.$$

On acidification with hydrochloric acid, the undecomposed barium sulphide and sodium sulphate react according to the equation—

$$Ba(HS)_2 + 2NaHO + Na_2SO_4 + 4HCl = \\ BaSO_4 + 2H_2S + 4NaCl + 2H_2O.$$

The $H_2S$ is removed by the aid of steam, and the *blanc fixe* filtered and calcined, the residual brine being employed to moisten a fresh parcel of ore for chlorination.—C. S.

*Oil Paints, The Thickening of.* W. Lippert. Zeits. angew. Chem. 1897, 779.

It is well known to varnish makers that certain oxide pigments, more especially zinc white, when ground into oil or varnish, cause the paint after a short time to become so thick [*vulg.* "livery"] as to prevent its being applied with a brush. In the text-books this phenomenon is said to be due to the combination of the metallic oxides with rosin acids; but as a matter of fact it is determined simply by the presence of any free acid in the vehicle, whether it be the fatty acid of an oil or the acid of any resin. The intensity of the thickening, however, is not necessarily proportionate to the amount of oxide and the acidity of the oil or varnish, but it depends rather on the particular nature of the two substances taking part in the reaction. Indeed, drying oils and varnishes, even when they are fairly old, should contain but little free acid; and the same remark applies to [refined] rosin oils; while cotton-seed oil, after purification with alkali, has none at all. The acids of a solid rosin do not produce so marked a thickening effect in paint as the fatty acids of an oil, and these in their turn are less active than the acids of some (Australian) copals. It is also worthy of notice that, in the majority of cases, paints which thicken on exposure to air, yield no skin or only an imperfect one, and, contrariwise, paints that skin do not thicken; so that the formation of a dried film on the surface may generally be taken as a sign that no change in consistency has occurred in the body of the material.

In order to investigate this problem somewhat more closely, a normal paint composed of 10 grms. of commercial zinc white and 15 grms. of "manganised" oil was made up. Other mixtures containing the pigment were next prepared from different vehicles, the above proportions being retained except when it was necessary to increase the liquid portion to 20 grms. to allow for extra viscosity. All the samples were exposed to the air, and were compared with the standard after 24 hours and also after 8 days. The standard itself skinned strongly, but preserved its original consistency. Neither the presence of 50 per cent. of dark or clear ("window-glass") rosin, nor of 66 per cent. of pale or dark rosin oil, nor that of 66 per cent. of cotton-seed oil, caused any thickening to take place; but the excessive amounts of foreign matter in some cases hindered or prevented the formation of skin. The addition of 7 per cent. of free linseed acids to the manganese varnish induced no visible change, but on increasing the proportion to 50 per cent., the paint thickened almost instantaneously. When either pale or dark rosin oil, cotton-seed oil, linseed oil containing flocculent matter, or Baltic hemp-seed oil was used alone to suspend the zinc oxide, no alteration in consistency occurred, but in the three latter instances, the pigment partly settled to the bottom. Both pale and dark Japanese wood (or tung) oil (this Journal, 1896, 661, and 1897, 684), which has been recommended as a substitute for linseed, caused the paint to appear "livery" in 24 hours; so that this product is not suitable for the manufacture of paint.

These experiments show that Amsel's process for distinguishing between rosin oil and mineral oil in linseed varnish, and for the detection of cotton oil therein (this Journal, 1897, 924), is not trustworthy.—F. H. L.

*Whiting, White Lead, and Ochre; The Behaviour of, with various Oils.* C. Böttinger. Chem. Zeit. 1898, **22**, [12], 102.

If the liquid matter in putty which has been made for a few weeks be extracted with ether, and its iodine number determined, it will be found to be smaller than that of the original oil. If, again, a certain proportion of a fatty oil, over and above the linseed ordinarily employed, be added during the manufacture of putty, the quality of the material will be greatly improved: it will be softer and dry less quickly.

To investigate these points, the following experiments were carried out:—A linseed oil having an iodine number of 183·3 was mixed with white-lead, and the mass exposed to light; after 17 days the iodine number had fallen to 131·0; in two months to 122·7. When ochre was used as the pigment, the corresponding values were 145·6 and 138·9. Whiting gave 172·0 at 8 days; 139·2 after 35 days. The oil itself contained no appreciable amount (under 0·05 per cent.) of ash. A similar product was next made up from pure calcium carbonate, and was kept for about six weeks, being kneaded together at frequent intervals. After a final mixing to incorporate the thick, brittle, greyish-yellow skin with the rest of the putty, it was thoroughly extracted with ether; and the residue was treated with water, alcohol, and again with ether, to remove the last traces of oil. During the process, a number of rotten skins were isolated, which gave, on gentle ignition, 51·91 per cent. of ash, containing 78·4 per cent. of lime. Heated on the water-bath with dilute hydrochloric acid, they evolved much carbonic acid; and at length, after losing about 76 per cent. in weight, they were converted into a caoutchouc-like body, and retained only 2·87 per cent. of ash. Some of the matter dissolved by the acid being also soluble in ether and alcohol, it should seem probable that these skins might consist in part of organic calcium salts.

In order to study the action of different oils, eight samples of putty were prepared from one specimen of whiting, of which they each contained 10 grms. (1) was made up with 10 c.c. of linseed oil (iodine number, 183·3); (2), 10 c.c. of linseed and 2 c.c. of ordinary olive oil (iodine number, 89·9); (3), 10 c.c. of linseed and 5 c.c. of olive oil; (4), 10 c.c. of linseed and 2 c.c. of petroleum: (5), 10 c.c. of linseed and 5 c.c. of arachis (earthnut) oil (iodine number, 117·8); (6), 10 c.c. of linseed and 5 c.c. of "putty oil" (Kittöl) (iodine number, 47·1): (7), 10 c.c. of olive oil alone; (8), 10 c.c. of arachis or earthnut oil alone. After standing four weeks, (1) formed a tough mass which adhered so strongly to the vessel holding it that it could only be removed with a knife; (2) was less adherent and still soft; (3) was unchanged in consistency and covered with clear oil; (4) resembled (1), but was not quite so hard—the petroleum had evaporated; (5) had separated into oil and chalk; (6) was covered with skin, and was not specially adhesive; (7) and (8) had separated. On extracting with ether, the oils from the first six samples smelt strongly of acrolein; the amount recovered, as well as that of the solid matter, and other details being recorded in the table annexed. It will be seen that the iodine numbers were smaller in every case than the original oils; and only those putties which contained linseed oil gave any considerable quantity of the brownish-yellow, insoluble, organic residue shown on the last line of the table; or, in other words, that only linseed oil is capable of entering into combination with lime under the conditions now referred to. The experiments also prove that any glyceridic oil may be employed to make putty dry more slowly and to prevent it from becoming too hard; but that a petroleum oil (no clue is given as to the boiling points of the sample used) evaporates too completely to produce any permanent effect on the material.

| — | | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. |
|---|---|---|---|---|---|---|---|---|---|
| Weight of oil recovered ........ | Grms. | 8·0395 | 9·9601 | 13·1791 | 8·2243 | 12·6750 | 12·1508 | 8·8578 | 8·4736 |
| Iodine number of recovered oil. | .. | 103·1 | 99·8 | 115·5 | 98·3 | 111·6 | 86·6 | 68·9 | 83·8 |
| Weight of pigment recovered .. | Grms. | 11·02 | 10·65 | 9·93 | 10·91 | 9·86 | 10·84 | 10·12 | 9·85 |
| Pigment insoluble in dilute acid | „ | 1·1380 | 0·8425 | 0·2277 | 1·1608 | 0·2525 | 0·9323 | 0·2120 | 0·1435 |
| Pigment insoluble in acid, alcohol, and ether. | „ | 1·0895 | 0·8205 | 0·1667 | 1·1106 | 0·2153 | 0·8924 | 0·1348 | 0·0860 |
| Ash of insoluble matter .... | Per Cent. | 14·19 | 9·99 | 37·64 | 17·28 | 26·25 | 8·9 | 31·76 | 56·12 |
| Organic matter in pigment recovered. | Grms. | 0·9349 | 0·7985 | 0·1040 | 0·9187 | 0·1588 | 0·8210 | 0·0879 | 0·0378 |

—F. H. L.

## PATENTS.

*Preservative Paints, Pigments, and the like, Impts. in.* A. J. Boult, London. From F. Fritsche and Co., Hamburg-Uhlenhorst, Germany. Eng. Pat. 2667, Feb. 1, 1897.

A COMMON paint, varnish, or lacquer is rendered specially "preservative" by the addition of 2 to 5 per cent. of metanitroparacresol ($CH_3.NO_2.OH$, 1:3:4). This body possesses the advantages of being solid at ordinary temperatures, of having such a low melting point that it can be mixed with the other ingredients during grinding, of producing a quick-drying paint "due to a conversion of the resins and metallic salts," of being cheap and easily prepared, and of not being acid, so that it cannot injure ships' bottoms.—F. H. L.

*White Lead [Precipitated], Impts. in and relating to the Manufacture of.* T. C. Sanderson, London. Eng. Pat. 6321, March 10, 1897.

ALTHOUGH precipitated white lead may have the same chemical composition and (true) specific gravity as the "Dutch" product, yet its apparent density (weight per bushel, &c.) is much lower. By the inventor's process a pigment is recovered, which, it is alleged, is equal, as to its physical qualities, to the older material. A solution of basic lead acetate is treated with carbonic acid until the liquid is converted into the normal salt. The precipitate is allowed to subside, the liquor run off, fresh basic acetate introduced and agitated with the solid matter, and the gas admitted again. The character of the final product may be varied by increasing the number of precipitations; or by altering the proportions between the white lead and basic liquor operated upon each time. The claim, therefore, is essentially for depositing upon a previously obtained precipitate of basic lead carbonate, a fresh layer or layers of the same substance in order to increase its apparent density.—F. H. L.

### (*B.*)—RESINS, VARNISHES.

*Shellac, The Bleaching of.* J. Fels. Papier Zeit. 1898, 23, [5], 152.

A COPPER pan holding 80 or 100 litres, having a draw-off cock at its lowest point and a perforated copper false bottom, is filled with 50 or 70 litres of water, 1·6 kilos. of caustic soda or 4 kilos. of sodium carbonate are added, and the whole heated to the boil. 10 kilos. of shellac are then introduced gradually and with constant agitation in order to prevent the mass sticking together in lumps; for should this occur, solution would be greatly retarded, and the bleached resin would prove very soft. When all is dissolved, the liquid is run into a second vessel capable of holding some [illegible]00 litres. A solution of sodium hypochlorite is next prepared by extracting 10 kilos. of bleaching powder with 200 litres of water, decomposing the calcium salt with sodium carbonate in the usual way. The hot shellac solution is mixed with the cold bleaching liquid, the last additions being made at intervals of five minutes, until the colour is only a pale yellow, when the vessel is covered up to keep it warm, and allowed to rest for several hours. Finally the clear liquor is drawn off into a third vessel holding 1,000 litres, raised (if necessary) to a temperature of 30° C. by the addition of hot water, and then precipitated with 5 per cent. sulphuric or preferably hydrochloric acid. The resin is thrown into cold water, filtered off and dried; there is no occasion to cast it into sticks, but if it must be kept for any length of time, this should be done under water. The yield is 110 or 115 per cent.—F. H. L.

*Driers and Varnishes.* M. Weger. Chem. Rev. Fett- u. Harz-Ind. **4**, [23], 313—316.

For testing the drying property of varnishes, the author recommends a comparative examination with a normal varnish, spread on glass in a thin layer and kept at a uniform temperature of about 15° C. without exposure to direct sunlight, the tackiness of the drying varnish being tested by finger pressure. The organoleptic tests, coupled with the drying test, are, however, the simplest and best. On the other hand, Amsel's "water reaction" is characterised as altogether incapable of fulfilling the advantages claimed for it.

With regard to the oxygen absorption test as an indication of drying qualities, the author considers that too much reliance should not be placed upon it, since, although the original method can be improved upon by spreading the varnish out in thinner strata (*e.g.*, about 0·0012 grm. to cover a surface of 1 sq. cm.), the relatively high weight of the plates employed and the hygroscopicity of the exposed surface, constitute sources of serious error.—C. S.

### (*C.*)—INDIA-RUBBER, &c.

*Rubber Goods, Estimation of Mineral Matter in.* L. de Koningh. J. Amer. Chem. Soc. 1997, **19**, [12], 952.

*See under* XXIII., *page* 277.

### PATENT.

*Waterproof Coating and Impregnating Substance, and a Process for the Manufacture of the same.* S. Hansel and E. Hornung, Vienna. Eng. Pat. 29,635, Dec. 14, 1897.

Twenty-five parts of albumin or gluten are dissolved in an equal weight of cold water, 1 per cent. of salicylic acid, phenol, or other antiseptic added, then 3 to 10 parts of india-rubber, gutta-percha, or their substitutes previously dissolved in any suitable solvent, and the whole agitated together. If a very flexible coating be required, 5 to 13 parts of glycerin, syrup, fat, oil, or oil-rubber may also be introduced. The composition is employed for waterproofing paper or other fibrous material, and the albumin is coagulated by passing the fabric between hot rollers. If preferred, however, the albumin may be rendered insoluble before the rubber, &c. is added; or it can be left soluble, sufficient sulphur incorporated to vulcanise the rubber, and the two operations of vulcanising the latter and of coagulating the former carried out by heat after the composition has been applied to the surface.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Leather, Chrome-Tanned, Finishing of.* F. Veselý. Časopis pro Průmysl Chemický, 1897, **7**, 354; Chem. Zeit. Rep. 1898, **22**, 15.

As soon as the leather exhibits an uniform grey-green colour in section, it is washed with luke-warm water in the tumbling drum, pressed, and blacked or coloured with coal-tar dyes, in which operation, a higher temperature is permissible than is the case with other leathers. The blacked material is then treated with a very carefully determined mixture of fat and soap at 30° C. The excess of fat is removed, and the skin is twice staked, and pumiced on the flesh side, and treated with a finishing mixture on the grain. —W. G. M.

Erratum.—This Journal, 1898, page 165. In table heading, for "Organic Extraneous Matter," read "Organic Extractive Matter."

## XV.—MANURES, Etc.

### PATENT.

*Cereals, New Process for Increasing the Growth of, and their Yield by Means of Bacteria, and a Method for Developing and Conserving the latter on a Large Scale.* H. E. Newton, London. From A. Caron, Germany. Eng. Pat. 5574, March 2, 1897.

It was found that certain aerobic bacteria present in the soil, have the power of increasing that portion of the combined nitrogen of the soil which is available for plants. The bacteria are quite distinct from the leguminous root-nodule bacteria, which assimilate free atmospheric nitrogen. One bacterium, termed *Bacillus Ellenbachensis Alpha*, is especially active, and, when present in the soil in large numbers, is said to render the application of natural or artificial manure unnecessary for growing cereals. It is best cultivated in a porous substance. Bread is saturated with broth, crumbled, put into suitable sterilised vessels, and inoculated with the Bacillus. After a short time the substance is thoroughly infected with the bacteria, which afterwards change into spores. For preservation the whole is dried at a low temperature, the spores remaining effective for years.

The bacteria may be developed on gelatin or broth suspended in a little water, which is then poured over the seed-grain. They may also be directly applied to the soil, in a dry state, after being mixed with some soil, gypsum, or other indifferent substance.—N. H. J. M.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Gentianose, Preparation of.* Ém. Bourquelot and L. Nardin. Comptes Rend. 1898, **126**, [3], 280—281.

Gentianose, a sugar analogous to cane sugar, was discovered in 1881 by Mayer (Zeits. physiol. Chem. **6**, 135). It is prepared as follows:—The fresh roots of yellow gentian are cut into thin slices and put into a flask containing boiling alcohol. An inverted condenser is then fitted to the flask, and the boiling continued for 20 to 25 minutes. When cold, the contents of the flask are expressed, filtered, and the filtrate distilled. The residue in the retort is neutralised with precipitated chalk, filtered, and evaporated on the water-bath; it is then dissolved in the smallest possible amount of water, and, whilst hot, treated with four or five parts of 95 per cent. alcohol. After 15 hours a viscous layer is deposited on the sides of the flask, and the liquid is poured off into a second flask. Crystallisation begins at once, and is complete in about 15 days. The substance is then recrystallised from 95 per cent. alcohol.

Gentianose melts at 207—209° C. (corr.) and is dextrorotatory, $\alpha_D = + 31°·25$. It does not reduce Fehling's solution. When heated with dilute sulphuric acid it becomes lævo-rotatory, and readily reduces Fehling's solution.—N. H. J. M.

*Starch, Process for the Treatment with Chlorine of.* Siemens and Halske. Zeits. für Spiritusind. 1898, **21**, 59—60. (Ger. Pat. 95,765.)

Raw or bleached commercial potato starch possesses a peculiar ethereal odour which also finds its way into the various products obtained by its hydrolysis. According to the invention, the starch is made into a sludge and introduced into a vessel provided with stirring machinery, in which, after raising the temperature to 45° C., it is treated with chlorine or chlorine water. The product is then washed with water and dried in the usual manner, or if glucose is to be prepared, the treatment with acid immediately follows the washing. It is stated that the treatment with chlorine leads to the partial disruption of the starch cellulose, the halogen entering into the starch granule, and there attacking the bodies giving rise to the ethereal odour, which are subsequently removed by washing with water.—J. L. B.

*Glucose, The Action of Benzhydrazide on.* G. Pinkus. Ber. 1898, **31**, [1], 31.

*See under* XXIV., *page* 283.

### PATENTS.

*Molasses, Impts. in and connected with the Extraction of Sugar from, by the Use of Barium Compounds.* L. Sternberg, New York, U.S.A. Eng. Pat. 18,429, Aug. 7, 1897.

In the process of extracting sugar from molasses by barium hydroxysulphide, the residual solution or waste lye contains the 5 to 10 per cent excess of barium hydroxysulphide employed, together with barium hydrosulphide and all the impurities originally present in the sugar solution.

The object of the present invention is to recover the barium and sulphur compounds present in the lye by transforming them into insoluble compounds. The lye is treated with excess of sulphurous acid, and the excess removed by barium carbonate, whereby barium sulphite and sulphur are precipitated. The precipitates are separated by filter-presses and dried and calcined in the presence of air, thereby forming barium sulphate and gaseous sulphurous acid, adapted for repeated use.

The drawings show a suitable saturation apparatus consisting of a horizontal cylinder containing a rotating stirrer and surmounted by a condensing tower.—L. J. de W.

*Sugar, Processes for Separating Impure, at one Operation, into Crystallised Sugar and Molasses.* M. Sachs, Kiew, Russia. Eng. Pat. 7861, March 26, 1897.

The discharge liquors of the first product of sugar refineries, molasses desaccharising works and affination processes, which have hitherto only been evaporated down to string proof, are brought to grain or crystals by first boiling down a certain quantity of the liquor in the vacuum apparatus in the usual manner, and then adding consecutively, further portions of the liquor previously heated. The completely boiled filling-mass, whilst still in the vacuum apparatus, is cooled by cutting off the steam and maintaining the vacuum for some time before it is discharged into the crystallisers.

Or, the ordinary process of boiling down to string is applied to the portions of discharge liquor, previously heated, in order to effect, by subsequent cooling and subjecting to centrifugal action, a separation at once into sugar and molasses. For the crystallisation, mashing vessels can be used with single walls and without arrangement for cooling or heating with steam. The cooling of the crystallising room may be effected by means of draught flues or by fans, which draw off the air warmed by the cooling of the filling mass, such air being conveniently discharged below the furnace grates of the steam boilers. The filling mass may also be cooled by a partial vacuum in vessels hermetically sealed and provided with stirring apparatus. When the temperature has sunk to 30° C., the treatment in the centrifugal machine can begin.

Where the liquor is boiled to grain, it is advantageous to add molasses, about 20 per cent. of warmed molasses being added at the end of the boiling, and the mass again thickened as far as is possible, so that it may be discharged from the pan. When this has been done, the entire charge is run off into the crystallising vessels, and is mixed with 10 to 20 per cent. of molasses, preferably heated to the temperature of the filling-mass. The second addition of molasses may be made in the vacuum apparatus, the mixture being facilitated by a stirring device, such stirring device being, however, only put in action after the heating devices have been turned out of action.

The processes are suitable for working sorghum juices. Where the purity falls below 72, there is added sugar, purer saccharine juices, or filling mass, until a purity of at least 72 is obtained, before boiling to grain or crystals.

The concentration of the filling mass must be carried so far that it shall contain a proportion of water and non-sugar such as is necessary in order that the discharge from the filling-mass, after cooling and crystallisation, shall constitute real molasses.

The warmed discharge liquors to be boiled, are to be introduced at the middle of the bottom of the vacuum vessel, instead of from above and at the side, as heretofore. —L. J. de W.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Aluminium Fluoride as an Antiseptic in Distilleries.* H. Cluss and H. Feber. Zeits fur Spiritusind, **21**, [1], 1898, 2—3.

As aluminium fluoride is a commercial product and easily obtained in quantity, the authors have compared its antiseptic power with that of other fluorides, with a view to substituting it for them.

The salt had a composition corresponding to the formula $Al_2F_6 + 18H_2O$; it crystallises in fine colourless crystals, which dissolve easily in hot water and slowly in cold. It is not so corrosive as the alkali fluorides.

The experiments were made with worts prepared in the usual manner from potatoes and maize; some of the experiments were carried out in the laboratory and some in the distillery.

It was found that aluminium fluoride was a more satisfactory antiseptic than the other fluorides when equivalent amounts of these were compared; the fermentations were free from acid and the action of the yeast or diastase was not interfered with.

About 33 grms. of aluminium fluoride per hectolitre were found to give the best results.—A. L. S.

*Acetic Bacteria. Contributions to the Knowledge of.* W. Henneberg. Wochenschr. für Brau. 1898, **15**, [2], 19—21.

The author has isolated two bacteria which he has named *Bacterium oxydans* and *Bacterium acetosum* and has compared them with Hansen's vinegar bacteria, *i.e.*, *B. Pasteurianum*, *B. Kützingianum*, and *B. aceti*.

*B. oxydans* was obtained from a low fermentation beer and is a motile one; *B. acetosum* was obtained from a high fermentation beer and is a non-motile form.

Colonies of *B. oxydans* on dextrose gelatin are at first round but soon become irregular.

In streak cultures the bacteria usually spread from the streak in opposition to the behaviour of Hansen's three varieties. *B. acetosum* in streak cultures gives a fine well defined mark.

*B. oxydans* is distinguished from other varieties by the thick, slimy, dirty-white masses of its colonies.

The skin formation of *B. oxydans* is not well marked, although a fine skin has been observed when growing on sterile beer. On the other hand, *B. acetosum* yields a thick, smooth white, and strong skin; in time, ridges are formed in it. The bacteria hang together in threads which greatly resemble *B. Pasteurianum*.

Like Hansen's bacteria, especially *B. aceti*, both of those isolated by the author give under certain conditions long thread-like forms.

A particular characteristic of these two bacteria are their abnornal forms; *B. oxydans* grown in wort to which a little alcohol has been added, forms thread-like cells with curious club-shaped swollen branches in three days at 30° C., and the same appear in sterile beer in two days at 26° C. *B. acetosum* growing in Grose beer forms at 30° C. in two days, swollen spindle-shaped shells. Both *B. acetosum* and *oxydans* grow best at ordinary room temperatures although the former also grows well at 30° C.

Comparing the acidifying powers of the acetic bacteria, the author finds that *B. Pasteurianum* produces acid only at 27°, *B. Kützingianum* between 27° and 36°, *B. aceti* best at 36°, *B. oxydans* best between 20° and 27°, and *B. acetosum* best at 27° C. The acetic bacteria can obtain their nitrogen from either organic or inorganic bodies; they cannot, however, obtain their carbon from ammonium tartrate or the lower (methylic, ethylic, propylic) alcohols.

The acetic bacteria easily oxidise dextrose and maltose; but galactose and dextrin are only oxidised by *B. oxydans*, which can also oxidise erythritol, mannitol, methylic, ethylic, and propylic alcohol.

Experiments made with sterile beer to which alcohol was added, gave the following amounts of acetic acid when the different bacteria were grown in it: *B. Kützingianum*, 5·4 per cent.; *B. acetosum*, 5·4 per cent.; *B. aceti*, 6·8 per cent.; *B. Pasteurianum*, 1·5 per cent.; *B. oxydans*, 1·6 per cent.—A. L. S.

*Yeast Manufacture: The Addition of the Lactic Acid Ferment.* G. Heinzelmann. Zeits. für Spiritusind. 1898, **21**, [1], 1.

The author points out that lactic acid destroys the action of diastase in the cold, and that very small quantities produce this effect at warm temperatures. He therefore recommends that much less than the usual quantity of the lactic acid ferment be added.—A. L. S.

*Yeast Manufacture: The Addition of the Lactic Acid Ferment.* G. T. Lövö. Zeits. für Spiritusind. 1898, **21**, [4], 31.

The author does not agree with Heinzelmann (see previous abstract); he finds that a larger quantity of lactic acid ferment gives a cleaner fermentation. He would use lactic acid itself, rather than add lactic bacteria to the wort.

—A. L. S.

*Glucase, the Enzyme of Maltose; Detection and Dissemination of.* Beijerinck. Centralbl. für Bacteriol. 1897, 221.

The author calls the ferments which hydrolyse starch, amylases, and those amylases which convert starch into maltose and achrodextrin he calls granulases. He investigates these ferments by a method of diffusion on gelatin plates. In order to discriminate between achrodextrin and erythrodextrin, iodine solution is used, and in order to recognise maltose and glucose, special yeasts are introduced into the gelatin.

In applying the first method, a 10 per cent. gelatin solution with ½ per cent. of gelatinised starch or soluble starch is made into a thin sheet, and the barley corn to be studied is cut longitudinally into two parts, and laid on the prepared gelatin sheet. The sheet is then kept in a moist atmosphere impregnated with chloroform vapour, for two days. The ferments, maltase and granulase, diffuse into the gelatin and hydrolyse the starch. If the gelatin sheet be then acted on by iodine solution, the bulk of the surface will be turned blue, but round the corn there will be seen two zones, the outside one (violet) showing the presence of erythrodextrin, and the interior, colourless. The maltase diffused more than the granulase, and the products of the former, erythrodextrin and maltose, are thus differentiated from those of the latter, achrodextrin and maltose.

The maltose cannot of course be recognised by this method. In order to differentiate maltose and glucose, the gelatin sheet is made from a 10 per cent. gelatin solution, 0·5 per cent. of potassium phosphate, 0·25 per cent. of asparagin, and a little of a pure culture of *S. fragrans* or *S. apiculatus* or *S. kefyr*. There must not be present a sufficient quantity to render the gelatin sheet at all opaque; these yeasts will not ferment maltose solutions, but will ferment glucose. When glucose is produced in the above manner on one of these sheets, its presence will be rendered visible by the growth of colonies of yeast.

Glucase was prepared from maize endosperms; they were extracted by alcohol of various strengths, and three preparations ultimately obtained, containing respectively 4·8, 1·1, and 2·3 per cent. of nitrogen, and of these the one containing the least nitrogen was the most active. Glucase converts soluble or boiled starch, dextrin, and maltose into glucose; but it cannot act on raw starch. Maltose is not an intermediate product when glucase acts on dextrin or soluble starch.

Glucase is not very plentiful in nature. The author has found it in rice, oats, maize leaves and roots, sorghum, millet, wheat, rye, and barley. It was not found in the germinating seeds of leguminous plants or in dates. The author states that seeds without endosperms, or with fleshy or horny endosperms, are free from glucase or diastase, whilst seeds with mealy endosperms contain glucase or maltase in the endosperm, and produce amylase when they germinate.

—A. L. S.

*Fermentation, Vacuum.* L. Aubry. Zeits. für d. ges. Brauwesen, **20**, [49], 631--633.

As carried on at the Helsingborgs (Sweden) Bryggerie-Actiebolag, the vacuum process consists in allowing the primary fermentation to proceed (in enamelled steel tanks) under a partial vacuum (450 mm. pressure), the carbon dioxide being withdrawn and replaced by a suitable quantity of air. After the primary fermentation is completed and secondary fermentation is in progress, the beer is run into the storage vat, and is "kräusened" with 10 per cent. of fermenting wort (Kräusen), and eventually fined. It is tapped at the end of 3½—4 weeks, and is said to be then superior to the ordinary beer. In some instances the carbon dioxide collected is liquefied, and returned to the matured beer.

The advantages claimed for the process comprise: economy of space occupied by the fermenting-tuns, of time in maturing the beer, ease of manipulation, and increased cleanliness. The author points out, however, that the plant is costly to erect and maintain; that the matured beer tastes somewhat young; that clarification is difficult, and necessitates the use of gelatin finings, and that the removal of the protective layer of carbon dioxide may expose the beer to infection unless the air supply be carefully watched, and may also facilitate the growth of organisms antagonistic to the yeast. Moreover, the flavour of carbonated beer is never so smooth and fine as that of beer in which the nascent gas has been retained; consequently, maturing in cask—or a similar process—is unavoidable. It is also stated that some difficulty is experienced in selecting suitable yeast for the vacuum process, since some yeasts do not thrive properly under the conditions prevailing therein.—C. S.

*Alcohol, Formation of, by "Intermolecular Respiration" of the Higher Plants.* E. Godlewski. Apoth. Zeit. **12**, 717—718; Chem. Centr. 1898, **1**, [1], 65.

It is stated that alcohol is formed during the changes which are the cause of "intermolecular respiration," *i.e.*, the evolution of carbon dioxide from plants, when the latter are placed in an atmosphero free from oxygen, or in a vacuum chamber. The author, in conjunction with Polženniusz, carried out some experiments with sterilised peas in aqueous solution *in vacuo*, and found that the proportion of alcohol formed, corresponds in general with the carbon dioxide evolved, according to the well-known equation of alcoholic fermentation, $C_6H_{12}O_6 = 2C_2H_5OH + 2CO_2$.

If sugar be added to the solutions, it is first inverted and then decomposed into carbon dioxide and alcohol; thus, there is no important difference between "intermolecular respiration" and the ordinary yeast fermentation. The total quantity of carbon dioxide evolved from the peas, amounted to more than 20 per cent. of the dry material.

—A. S.

*Slack Malt, Defective Flavour and Brilliancy of Beer Brewed with. A Cure proposed in a Curtailed Mashing System.* W. Windisch. Wochenschr. für Brauerei, 1898, **15**, 53—54.

In a brewery where the conditions of malt storage were very bad, the malt being heaped in a damp situation and rapidly becoming very slack, it was observed that the successive brews yielded increasingly unsatisfactory results as regards flavour and brilliancy of the beers, tendency to gluten turbidity, &c., in proportion to the age of the malt. The trouble, primarily due to the increasing and, at last, excessive slackness of the malt, was partly overcome, and especially the flavour and brightness of the beers much improved, by shortening the mashing process, a single mash being substituted for the triple-mash system at first in use. The inferior flavour of beers produced from slack malt, the author ascribes to the presence in such beer of furfurol in abnormally large amount. Furfurol is always produced during the mashing and boiling processes by the action of the acids of malt on the pentosans and pentoses likewise present. The author proves by experiment that the proportion of furfurol thus formed, increases with the duration of the mashing and boiling processes, being greater on the triple- than on the single-mash system; and, apart from this, is decidedly greater in the case of slack as compared with dry malt, no doubt on account of the high percentage of acid usually contained in the former.—H. T. P.

*Barley Wine.* Foreign Office Annual Series, 1898, No. 2823, p. 19.

In a consular report (1896, Annual Series No. 1868) a paragraph was inserted stating that a liquor having the character of port wine had been made from barley in Germany (see also this Journal, 1897, 196). In its issue of November 7, 1897, the *Rivista Agraria* compares the revolution these barley wines are likely to cause to agriculture as similar to that caused by beetroot sugar. It is stated that a vast establishment is at work at Wandsbek, near Hamburg, to produce these so-called "Malton-Weine," which have the characteristics of the wines of Southern Europe, owing to the entirely new process of fermentation to which they are subjected. The factory is said to produce 10,000 hectolitres, and its products were very highly appreciated at the Agricultural Society's exhibition at Hamburg. Many classes of wine have been closely imitated—sherry, port, Tokay, and Malaga being named amongst them. When the fermentation is completed, the new wine is put into huge vats of 200 hectolitres, and thence into casks of 70 hectolitres, where it is subjected to a high temperature. The transformation of the substances which give the liquor its taste and bouquet is thus accomplished in a few weeks, instead of requiring as many years, and at the same time all extraneous matter derived from the ferments is sterilised and rendered innocuous. The liquor can then be drawn off into small casks or bottles, and in three or four months it is ready for consumption. These artificial wines are notable for the large percentage of alcohol they contain. They run to 19 and even more per cent., derived entirely from the fermentation, and not due, as would be naturally supposed, to added alcohol. To barley growers the discovery may be of vast practical utility, as forming a new market for an article which agricultural depression has sorely depreciated.

The product is not sold under any false description, but is called "highly fermented barley wine."

The *Berliner Klinische Wochenschrift*, November 1, 1897, states that, though these malt wines have been variously judged by the public, the medical reports of them have been always favourable. In the ward of the local hospital the following brands were used in the course of the last half year: Tokay, sherry, and port. These wines were taken, almost without exception, very willingly by the sick, and preferred even to Greek and other southern wines. The favourable influence of the malt wine on the general health of the patient and on an exhausted nervous system. In cases of disease of the stomach, it has been observed that the mild malt wines were easily digested when other sweet wines proved troublesome. The opinion that only wines made from grapes should be given to invalids is characterised as unfounded prejudice. All wines equal in alcohol to the malt wine are so dear that they can only be prescribed for a few patients, and very few other wines contain the degree of nourishment of these malt wines.

*White Wines from Red Grapes, Utilisation of Grape Oxydase for the Production of.* A. Bouffard and L. Semichon. Comptes Rend. 1898, **126**, 423—426.

It has been shown that the oxydase of grapes is identical in properties with that existing in wines affected with "*la casse*," and Martinand (Comptes Rend., June 1895) has indicated a process for the production of white wines from red grapes, on which the oxidising and precipitating action of oxydase in the presence of air, on the colouring matter of grape-must, is utilised. The process has been simplified recently by one of the authors (Rev. de Viticult., March 1897); and in the present paper are embodied suggestions as to its application to the production of wines of all types (in respect to depth of colour, *e.g.*, "vins roses," "paillets," &c.). Advantage is taken of the fact that at any stage the oxydase may be destroyed and the colour of the must rendered permanent by the addition of a trace of sulphurous acid (2—5 grms. of $SO_2$ in the form of potassium bisulphite per hectolitre). In the ex[illegible]ction of the grape-juice, a moderate degree of pulpi[illegible] will suffice to ensure the presence of a sufficiency [illegible] and [illegible]dase in the must. The subsequent oxidation of [illegible]ouring matter may be effected in a volume [illegible] hectolitres of must, by blowing through it a c[illegible] air until the desired effect has been produced (20—30 minutes). Excessive aëration in the production of white wines, however, should be avoided, since the liquid again assumes a brownish hue after the complete decolorisation stage has been passed. The addition of a trace of sulphurous acid will prevent any such change occurring during subsequent stages. After fermentation, the flocks of oxidised colouring matter settle out along with the lees, so that a filtration of the wine becomes unnecessary. White wines produced in this way, possess the following advantages: absence of sulphurous flavour, permanence of colour, and normal chemical composition—advantages which cannot be claimed for wines bleached by large doses of sulphurous acid.

The author remarks that in the manufacture of red wine, excessive disintegration of the grapes and excessive aëration of the must should be avoided in order to prevent destruction of the colouring matter. Here, as also in the production of wine from white grapes, a trace of $SO_2$ is an effective preventative against the disease "*la casse*."—H. T. P.

*Wines, The Bitterness of.* J. Bordas, Joulin, and Rackowski. Comptes Rend. 1898, **126**, [8], 598—599.

The authors describe an organism from the deposit of a bitter wine which was isolated in a state of purity by successive cultivations in concentrated yeast-water rendered slightly alkaline and containing glucose and plate cultures. In yeast-water containing glucose, the bacillus occurs either as filaments, contorted or simple, consisting of rods joined end to end; after some days these filaments reunite to form clusters of segmented rods. The organism grows readily in the Laurent medium, modified by the addition of 10 per cent. of Collaspeptone. The liquid becomes cloudy in 24 hours, and in 8 days a perceptible bitter taste is developed; there is also a slight liberation of gas. In this medium the bacillus grows in small rods, sometimes motile, 1 μ broad and 4 to 5 μ long.

After six months, sterile wine which had been infected with this organism had a pronounced bitter taste. The wine was cloudy, some of the colouring matter was precipitated, and the deposit contained the characteristic filaments. The content of alcohol was unaltered; the proportion of glycerin and glucose was, however, less. The acidity was markedly augmented, arising from volatile acids; the presence of small quantities of ammonia was also observed. In a sterile wine which had been freed from alcohol, the growth was very rapid.—J. L. B.

*Diastase of Barley, Observations on the Determination of.* P. Terrat. Journ. Pharm. Chim. 1897, **6**, [11], 494.

*See under* XXIII., *page* 277.

*Vinegar Analysis and some Characteristics of Pure Cider Vinegar.* A. E. Smith. (Contribution from the Chem. Lab. of Case School of Appl. Science, No. 30.) J. Amer. Chem. Soc. 1898, **20**, [1], 3.

*See under* XXIII., *page* 276.

*Ethyl Sulphuric Acid [for Alcohol] from Coke-Oven Gas, Production of.* P. Fritzsche. Chem. Ind. **21**, [2], 27.

*See under* III., *page* 236.

*Acidity and Alkalinity, Estimation of, in Coloured Products.* F. Jean. Ann. Chim. Analyt. Appl. **2**, 415; Chem. Zeit. **22**, [4], 27.

*See under* XXIII., *page* 276.

*Wine, Chemical Analysis of. Decree of Instructions issued June 25, 1896, by the German Federal Council.* Veröffentl. d. Kaiserl. Gesundheitsamtes, 1896, **20**, 557; Bull. Assoc. Belge des Chim. **11**, [7], 196; [8], 237.

*See under* XXIII., *page* 277.

*Beer and other Liquids containing Acid Phosphates, Determination of Acidity in.* A. Ott. Zeits. für d. ges. Brauwesen, **20**, [51], 667; [52], 682.

*See under* XXIII., *page* 281.

## PATENTS.

*Sterilising, Purifying, and Deodorising Casks and other Receptacles for Beverages and other Materials; Improved Method of.* A. Apps, London. Eng. Pat. 6316, March 10, 1897.

The process consists in placing the casks, &c. in an air-tight chamber, and exhausting or partially exhausting the air, or in exhausting the air within the receptacle itself, and subsequently admitting ozone, ozonized air, or oxygen, with or without moisture. In this way the ozone is made to enter the pores of the wood, and the admission of the gas to the joints of the casks is facilitated by loosening or removing the heads before placing the casks in the chamber. If necessary, the process is repeated, and finally the receptacle is washed with steam or hot water.—C. A. M.

*Mixing together Liquid and Gaseous Bodies, Improved Method of, and Apparatus therefor.* A. J. Boult, London. From H. H. Dikema, Brussels. Eng. Pat. 1843, Jan. 23, 1897.

*See under* I., *page* 229.

*Separating Beer from Yeast, Improved Apparatus for.* E. G. Major-Lucas, Northampton. Eng. Pat. 6343, March 10, 1897.

This consists of a "back" with a sloping bottom, across which is stretched a filtering cloth at a suitable distance from the top, supported by wooden bars or divisions, which may extend to the bottom of the 'back." It is suitably constructed in two parts, the upper part fitting tightly within the top of the lower, and keeping the filtering cloth tightly stretched by means of its weight. The yeast skimmed from the fermenting tun is conveyed to the filtering "back," and the filtered beer is drawn off through a cock near the bottom. It is stated that by the use of this apparatus, labour is saved, filtering bags are dispensed with, and that the beer loses its bitterness through being separated so soon from the yeast.—C. A. M.

*Beer, Impts. in the Manufacture of.* G. Delory, Paris. Eng. Pat. 26,520, Nov. 13, 1897.

The object of this invention is to enable farina and other starchy substances to be used as substitutes for malt without the usual drawbacks of their assuming a pasty consistence and interfering with the fermentation by reason of the essential oils they contain. This is effected by heating the substance, spread in trays, in a drying oven or malt kiln at a temperature of from 140° to 160° C., according to its nature, until it becomes yellow. It is usually advantageous, and necessary in the case of rice and Indian corn, to first sprinkle the starchy material with a 2 per cent. solution of malt. By this treatment a neutral product is obtained, yielding an extract of from 90 to 92 per cent., and no pastiness occurs during saccharification. The essences have been volatilised by the heat, and the beer consequently remains clear after fermentation. It is stated that beer of the first quality can be prepared from a mixture of 1,000 kilos. of ordinary malt and 400 kilos. of amylaceous material thus treated.
—C. A. M.

*Beers, An Improved Funnel to Facilitate the Fining of; applicable also for Filling Casks.* H. G. Owles, London. Eng. Pat. 28,398, Dec. 1, 1897.

The funnel is constructed with a nozzle surrounded by a cone-shaped elastic ring so as to enable it to fit tightly within the bung of a barrel. Within the nozzle is a valve, movable by means of a rod working within a tube connected with the top of the funnel by means of a bridge-piece. The valve can be kept open by pushing the rod down and locking it by turning a pin on the side of the rod into an opening in the tube, and can be kept closed by means of a spring arranged between the button or handle at the top of the rod and the bridge piece at the top of the tube. In use the finings are introduced into the beer in the barrel, and the valve left open. The froth and scum rise into the funnel, and are subsequently removed with it after closing the valve. The funnel with the valve kept open, can also be employed for filling barrels in the ordinary way.—C. A. M.

*Beer, Non-Alcoholic; A Process for the Manufacture of.* V. Lapp, Leipzig-Lindenau, Germany. Eng. Pat. 30,208, Dec. 21, 1897.

The process comprises the following stages, the first of which is claimed alone, the remainder in combination with one or more of the others:—(1.) The boiled and clarified wort is treated with diastase at 57°·5 C., so as to produce the maximum of maltose and leave the minimum of dextrin. (2.) After the addition of pure lupulin obtained from hops, the wort is boiled, and exposed to the atmosphere whilst hot in a fine state of division by means of a centrifugal machine, with the object of causing the separation of albumin and similar substances, which might subsequently produce turbidity or unpleasant taste. (3.) A further separation of undesirable constituents and the production of a pleasant flavour is brought about by again subjecting the hot beer to centrifugal action, but this time in an atmosphere of carbon dioxide. (4.) The hot beer is rapidly cooled to 0° C. by means of freezing coils, which causes the deposition of other detrimental substances. (5.) The beer is fined and saturated with carbon dioxide at a temperature of 0° C. It is claimed that the non-alcoholic beer prepared is free from germs, and has great stability and digestibility.
—C. A. M.

*Malt-Flour, An Improved Process and Apparatus for Producing Beer-Wort from.* C. Schmitz. Boppard am Rhein, Germany. Eng. Pat. 28,579, Dec. 3, 1897.

By means of this invention it is stated that a clear wort can be obtained from malt-flour, nearly all the starch (to within 1·69 per cent.) being converted into sugar, whilst by other processes, from 5 to 6 per cent. of undissolved starch remains in the residue. The mash is made at 35° C., and the temperature gradually raised to 65° or 66° C. A portion is then withdrawn and reserved in a separate vessel, after which the heating is gradually continued to the boiling point. After being boiled for some time, the mash is cooled to from 69° to 65°, and the reserved portion of wort added, which, being rich in diastase, saccharifies the starch rendered soluble by boiling.

Claim is also made for a special apparatus in which this process may be carried out, comprising a heating copper, mash tun, cooling trough, cooling receiver, pumps, float, and deposit vessel, together with the corresponding pipes, taps, and valves. The mash tun is constructed with double walls, between which circulates steam or hot water drawn from the heating copper by means of a pump, and flowing back into the copper to be heated again. The wort to be reserved is drawn off from the surface of the mash through the float, which contains an air chamber and a series of concentric perforated chambers to retain impurities, and flows thence through a pipe into the depositing vessel. The special cooling trough is made with corrugated double walls, with partitions connecting the opposite points of the corrugations, so as to form a series of longitudinal chambers opening into one another at opposite sides. The cooling water enters the upper space and passes in a zigzag manner to the lowest. The hot wort runs from a tap at the end of a pipe coming from the bottom of the mash tun, runs over the interior of the cooling trough to the brewing pan, and is there mixed with the portion of wort previously reserved in the deposit vessel. It is stated that the whole of this process occupies about eight hours.—C. A. M.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (A.)—FOODS.

*Eggs, Preservation of.* J. Soc. Arts, 1898, **46**, [2363], 370.

This article is from a report of the United States Commercial Agent at Freiburg, on the results of experiments made for the purpose of ascertaining the most rational method of preserving eggs. The specific gravity is stated to be the best indication of the age of the eggs; with fresh eggs it is from 1·0784 to 1·0942. A solution of 120 grms. of common salt in 1 litre of water was prepared; this solution has a sp. gr. of 1·073, and only those eggs which would sink in it, were used in the experiments. 20 methods of preservation were examined, and 20 eggs were used for each method, the eggs being preserved for 8 months and then re-examined. The following results were obtained:—(1) Eggs placed for preservation in salt water all became bad (not rotten, but uneatable, the salt having penetrated through the shells). (2) and (3) Eggs wrapped in paper, and eggs preserved in a solution of salicylic acid and glycerin, 80 per cent. bad. (4), (5), (6), and (7) Eggs rubbed with salt, preserved in bran, provided with a cover of paraffin or varnished with a solution of glycerin and salicylic acid, 70 per cent. bad. (8), (9), and (10) Eggs placed in boiling water for 12—15 seconds, treated with a solution of alum, or placed in a solution of salicylic acid, 50 per cent. bad. (11), (12), and (13). Eggs varnished with water glass, varnished with collodion, or covered with lac, 10 per cent. bad. (14), (15), (16), and (17). Eggs varnished with various mixtures, preserved in ashes of wood, treated with boric acid and water glass, or treated with potassium manganate, 20 per cent. bad. (18), (19), and (20). Eggs varnished with vaselin, preserved in lime-water, or in a solution of water glass, none bad. The preservation in a solution of water glass is to be preferred, as varnishing the eggs with vaseline takes too much time, and the treatment with lime water sometimes communicates to the eggs a disagreeable odour and taste. The only drawback to the method of preserving in a solution of water glass, is that the shells, after this treatment, easily burst in boiling water, but this may be avoided by carefully piercing the shell with a strong needle.—A. S.

*"Table Oil": Is the Term to be Limited to Pure Olive Oil?* Chem. Rev. Fett- u. Harz-Ind. **4**, [23], 318.

*See under* XII., *page* 254.

*Butter, Practical Use of the Refractometer in the Examination of.* A. J. Swaving. Landw. Vers.-Stat. **49**, 341; Chem. Centr. 1898, **1**, [5], 352.

*See under* XXIII., *page* 276.

### PATENTS.

*Lupins for Yeast Manufacture, Cattle Food, and Similar Purposes; Impts. in or relating to the Preparation of.* H. Schowell, Magdeburg-Sudenburg, Germany. Eng. Pat. 3810, Feb. 12, 1897.

Process for utilising lupins by extracting the albuminous contents by the action of an acid solution, the peptonising liquid being renewed at given intervals so as to effect the peptonising of as much of the albuminous materials as possible. The lupines are treated, after maceration and removal of albuminous matters, with molasses or beet sugar residues without previous pressing.—N. H. J. M.

*Preservative Desiccation of Vegetable and Animal Substances; Impts. in and relating to Methods and Apparatus for.* B. L. Ryder, Chambersburg, Pennsylvania, Eng. Pat. 4730, Feb. 22, 1897.

The apparatus consists of a box divided lengthwise into three chambers one above the other, on the floors of which rails are fixed for resting trays on. The trays are of wire gauze in oblong wooden frames, so constructed that the air can pass underneath. The floor of the lowest chamber is inclined and has rails fixed to the sides as well as on the floor. These side rails terminate within the chamber, so that trays pushed along them to the end, fall to the bottom of the chamber; in this manner a number of trays may be automatically piled in groups one above another.

The heating apparatus is below the larger end of the lowest chamber. By means of doors at the end of each chamber, and sliding dampers or valves, the heated air can be directed through the chambers according to the amount of drying required; external air can also be admitted, if necessary.

At the end of the top chamber farthest from the heater, is an opening covered by a hinged hood, in which sulphur contained in a brazier may be burned; the opening of the hood is glazed. The brazier may, instead, be placed on the heater, so that the fumes may be carried upwards by the current of heated air.—N. H. J. M.

*Vegetable Extracts and Peptones of Commercial Utility, Process for the Production of New, by the Transformation of all Kinds of Yeast and these New Products of Commercial Utility Themselves.* J. Peeters, Schaerbeek, near Brussels. Eng. Pat. 26,985, Nov. 18, 1897.

Yeast is washed with 0·1 per cent. acetic acid four times, the last deposit pressed and treated with an equal weight of water containing 5 c.c. of strong hydrochloric acid per litre. It is then heated for about 20 hours at 60° C., and may be neutralised wholly or partly with soda. When cold, it is filtered and concentrated under diminished pressure at 60°—70° C. Salt is added in any desired quantity.

A new vegetable peptone may be prepared by continuing the evaporation of the above product until syrupy. When cold, the syrup is slowly dripped into alcohol (95° Tr.), and continually stirred. The amount of alcohol employed is 1 kilo. to the product of 1 kilo. of yeast containing 75 per cent. of water. The alcohol is poured away from the sediment, which is re-dissolved in 500 c.c. of water, boiled, allowed to cool, and filtered. After adding some common salt, the product is evaporated down at 60°—70° C. A sample of a new vegetable peptone thus prepared contained:—Water, 10; nitrogen, 12·5; ash, 12; potash, 4·61; and phosphoric acid, 5·99 per cent.—N. H. J. M.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Sulphite Liquor, Recovery of Waste from Wood-Cellulose Factories.* S. Ferenczi. Papier Zeit. 1897, **22**, [100], [102], [103], 3575—3679.

*See under* XIX., *page* 264.

### PATENTS.

*Purifying Water, Sewage, and other Foul Liquids; Impt. in the Method of, and Apparatus for.* W. M. Ducat, London. Eng. Pat. 654, Jan. 9, 1897.

This is an aerated filter bed designed for the continuous purification of raw untreated sewage, &c., by bacterial action, applicable also to the purification of drinking water.

The filter is constructed of suitable material, such as pebbles, clean washed gravel, pumice stone, stone chips, &c., arranged in superposed layers, diminishing in coarseness from the top to the bottom. For the thorough aeration of the filter, horizontal rows of open jointed pipes are laid in very coarse material between the adjacent strata; these pipes communicate with apertures in the containing walls, which are constructed of open brickwork, or, preferably, of superposed rows of earthenware pipes laid in cement. Pipes are provided for the escape of the effluent in a bottom drainage layer of coarse material.

For the maintenance of a uniform and sufficiently elevated temperature, the entire filter is surrounded by an air space enclosed within solid walls and a roof. Doors are provided in these outer walls for the admission of air in warm weather, whilst in very cold weather, air is admitted through a separate chamber in which a heating apparatus is placed.

The liquid to be filtered is distributed as uniformly as possible over the surface of the bed, and is supplied at such a rate that the surface is never submerged and the quantity of liquid in the mass is never sufficient to flow out through the pipes in the containing walls, which are inclined slightly upwards. By thus limiting the flow of liquid, an exhaustive action is set up, due to the absorption of oxygen and the motion of the fluid passing through the filter, whereby fresh air is continuously drawn into the mass.—L. A.

*Purification of Water, Sewage, and other Foul Liquids; Impts. in, or connected with the.* W. M. Ducat, London. Eng. Pat. 5803, March 4, 1897.

The essential feature of this invention is the construction of a filter or nitrification chamber for the purification of raw sewage, in which the stratification is the reverse of an ordinary filter, *viz.*:—very coarse at the upper part and gradually less coarse towards the lower part, the object being to permit of the sludge penetrating into the interstices of the filtering medium without choking them, so as to enable the solid matter in the sewage to be there broken down and liquefied by biological action. The filter is constructed, aerated, and used as already described (Eng. Pat. 654, 1897. See preceding abstract).—L. A.

*Sewage and Impure Waters; Impts. in Tanks and Apparatus for use in Connection with the same for the Purification and Treatment of.* F. W. Strong, Great Yarmouth, Norfolk (formerly of Bournemouth, Hants). Eng. Pat. 2228, Jan. 27, 1897.

This invention relates chiefly to arrangements for collecting and removing the sludge from precipitation tanks, also for filtering or straining the liquid.

The tank is constructed with a gutter at the lower end, for the reception of the sludge, at the bottom of which is a slotted plate communicating with a chamber below. By raising a lid which covers the slot, the sludge is forced by the head of water in the tank into the chamber and away through a pipe.

An improved scraper is next described, provided with flexible squeegees which may be worked backwards and forwards over the tank floor so as to sweep the sludge into the gutter, or when the tank is not provided with a gutter into a travelling slotted sludge pipe. The sewage is admitted to the tank behind a partition or wall, which conducts it to the bottom of the sludge gutter, from which it rises into the tank through a screen or strainer composed of sacking or other material.

An arrangement is also described in which the sludge is removed from a number of precipitation tanks by means of a single siphon pipe, which can travel by hand or power from tank to tank, and discharge the sludge from each into a trough or gutter at a higher level.—L. A.

*Ores, Treatment of; Impts. in, and Apparatus therefor, the Invention being also adapted for the Treatment of Sewage.* J. Poole, Coolgardie, W. Australia. Eng. Pat. 14,575, June 16, 1897.

*See under* X., *page* 251.

## (C.)—DISINFECTANTS.

*"Tfol" (Soapstone of the Arabs), Note on, and on a Method for Emulsifying Heavy Tar Oils.* Lahache. Journ. Pharm. Chim. 1898, **7**, 57—59.

The mineral soap used in Algeria and Tunis, "*tfol*," is somewhat waxy and unctuous to the touch, it varies in colour from red to grey and white, and in composition. It is composed of silicates of the alkalis and alkaline earths, calcium carbonate, free gelatinous silica, clay, and in small quantities, sulphates and chlorides of the alkalis and alkaline earths. This soapstone, when finely powdered, absorbs five times its weight of heavy tar oil, and from the paste obtained, water does not separate the oil, but on agitation with water an emulsion is formed. It is proposed to use the emulsion as a disinfectant.—A. C. W.

*Phenol, Estimation of, in Disinfectants in presence of Soaps.* W. Spalteholz. Chem. Zeit. 1898, **22**, [8], 58.

*See under* XXIII., *page* 281.

## PATENTS.

*Guaiacol, Impts. in the Manufacture of.* Kalle and Co., Biebrich-on-Rhine, Germany. Eng. Pat. 7233, March 19, 1897.

*See under* XX., *page* 269.

*Formic Aldehyde Compounds [Preservatives and Disinfectants], New, and Processes of Preparing the Same.* H. Oppermann and R. Goehde, Berlin, Germany. Eng. Pat. 6653, March 13, 1897.

Sugars of the cane sugar group form compounds with formaldehyde, $C_{12}H_{21}O_{10}.CH_2O$ + water; these are syrups which cannot be evaporated to dryness, are stable in dry air, but in moist air or above 50° C. decompose into their constituents. These compounds unite with the alkali haloids and so form substances stable below 80° C., thus:—

$$2(C_{12}H_{21}O_{10}.CH_2O)NaCl + 4 \text{ to } 6H_2O.$$

Compounds of both classes are claimed as new substances, and the processes for their preparation, which consist in the evaporation at a low temperature of a solution of the constituents in the proper proportion. The haloids may be replaced by potassium bitartrate or agar-agar containing that salt.—A. C. W.

## XIX.—PAPER, PASTEBOARD, Etc.

*Timber, American Forest.* Papier Zeit. **22**, [101], 3616.

Germany has nominated officials to study the agriculture and timber-growing in foreign countries, with a view to introducing the best methods into Germany.

In a report from Washington, U.S.A., the condition of the North American forests is described. The Weymouth fir, or white fir, is specially noted, on account of its excellent cleavage, low specific gravity, little shrinkage, &c. This fir grows well almost anywhere, and the wood of the young trees is in great demand, both for poles, &c., and for paper-making.

The report then compares the consumption of timber with the stock and produce. Accurate statistics cannot be obtained, but at the present rate of consumption, the American wood will be used up in from five to six years. Timber is also largely imported from Canada, and if the excessive consumption be not checked, it is estimated that it will last only from 40 to 50 years.—S. P. E.

*Normal Papers [Germany].* W. Herzberg. Mitt. k. tech. Versuchsanst. zu Berlin, 1897, **15**, [6], 322—323.

Those Government district offices in Germany using paper of less value than 15*l.* per annum, are now required to have the paper supplied, tested for quality. In 1895, 10 of these offices made application for the testing of 19 samples of papers, of which 9 samples did not come up to the required standard. In 1896, 208 samples were tested, of which 53 samples, or 25·5 per cent. were below the necessary standard.—S. P. E.

*Rag Half-stuff or Rag-pulp.* Papier-Zeit. 1897, **22**, [97], 3471.

Although frequently proposed, no mill has yet been erected for manufacturing rag half-stuff as a marketable article. A demand certainly exists for such material, and sample lots have been made at paper-mills. A difficulty experienced is the drying of the pulp; the writer only succeeded by draining and by the use of centrifugal machines in obtaining a pulp of 35 per cent. fibre. Rag-stuff cannot be dried like wood-pulp, owing to its forming compact board-like material, difficult for the paper maker to disintegrate in the beating engine. The high percentage of moisture makes the cost of transport heavy. Another writer who is about to erect and manage a rag half-stuff mill on the Rhine, for dealing with 5,000 kilos. daily, strongly condemns the methods adopted in previous attempts at this branch of manufacture. He sums up the necessary conditions for successfully manufacturing this material as follows:—

1. Cheap female labour, and plenty of it.
2. Water power, or cheap coal.
3. Plenty of suitable and clean water.
4. A favourable site near mills making a good quality of paper. Conveniences for water transport and exportation at low rates.
5. An experienced paper-maker who understands the nature and quality of rag half-stuff required for the manufacture of the various grades of paper.—S. P. E.

*Paper for Newspaper Printing, Manufacture of.* W. Herzberg. Mitt. aus den Königl. techn. Versuchsanst. zu Berlin, 1893, **16**, [1], 87—88.

Only the cheaper sorts of paper are used for newspaper printing, the chief desideratum being that the strength shall be sufficient to stand the strain of the printing. Wood meal is the chief constituent of such papers, but in some cases, wood cellulose and straw cellulose are used, and occasionally certain quantities of rag.—H. H. B. S.

*Soda, Recovery of.* Papier Zeit. 22, [101], 3614.

In an article on straw-pulp making, Küssel recommends the use of ovens in which the hot gases are used for evaporating the spent soda lye. This method is shown to cause a waste of fuel, and ovens should be used in connection with evaporating apparatus. A spent lye containing less than 15 per cent. of soda is not worth evaporating. Thus, it does not pay in the case of small mills to erect soda recovery plant, but the larger the mill, the more economical the erection of plant becomes.—S. P. E.

*Sulphite Liquor, Recovery of Waste from Wood Cellulose Factories.* S. Ferenczi. Papier-Zeit. 1897, 22, [100], 3575; [102], 3647; [103], 3679.

The Papier und Zellstoff Fabrik at Unterkochen-Wolfach, Germany, offers a prize of 10,000 marks for the best and most successful method of treating waste sulphite liquors so as to prevent pollution of the streams into which those liquors are now run. A number of mills in Germany have been closed by the authorities owing to the want of a method of preventing such pollution. The author deals with the several methods proposed and experimented with, for the purpose of solving this difficulty.

The following is the composition of the fresh and the waste liquors at the above sulphite mills:—

*Fresh Liquor.*

| | Per Cent. |
|---|---|
| Total $SO_2$ | 3·29 |
| Free $SO_2$ | 1·76 |
| $SO_2$ combined with CaO | 1·44 |

*Waste Liquor.*

| | Per Cent. |
|---|---|
| Water (at 105° C.) | 88·7 |
| Organic matter | 9·7 |
| Mineral ash | 1·6 |
| Sp. gr. at 17·5° C. = | 1·0515 |

The mineral ash in the waste liquor gives the following on analysis:—

| | Per Cent. |
|---|---|
| Calcium sulphate | 0·24 |
| Calcium sulphite | 0·91 |
| Magnesium sulphite | 0·24 |
| Magnesia combined with organic acids | 0·13 |
| Silica, alumina, oxide of iron, &c. | 0·50 |

The chief trouble at the above cellulose factory, as well as at other sulphite works, is due to the enormous growth of a fungus (*Beggiatoja alba*) which occurs in the streams into which the waste liquor flows. F. Zenk, in his book upon "Pollution of Watercourses," says "streams contaminated with waste sulphite liquor have a disagreeable odour, the water is turbid and dirty, and when running over cascades or falls a peculiar froth forms, owing to the escape of gases mainly sulphuretted hydrogen, produced by the putrefaction of the organic matter contained in the liquor. Green plants that generally grow in such streams when not polluted, disappear, and are replaced by a colourless fungus." This *Beggiatoja* growth is nourished by the organic matter containing sulphur and lime which is contained in the waste liquor. From the waste liquors it is easy to eliminate and recover the sulphites and free sulphurous acid, but great difficulty is experienced in dealing with the organic matter.

Alex. Mitscherlich has taken out a number of patents for the recovery of the organic matter from waste sulphite liquor and for the production of useful products therefrom. Ger. Pat. 4179, 1875 has for its object the extraction of a tannin material from the waste liquor. In German patent 34,420, also by Mitscherlich, there is described a process of using the tannin material as size for paper. By this process the solution containing the tannin (simply the waste lye) is added to the pulp in the beating engine, and when well mixed, a solution of gelatin is added, the result being an insoluble coating upon the fibres of tannin-size. In a later patent, the addition of resin-size is recommended. The transport of waste liquors to the paper mills proved to be too expensive, therefore Mitscherlich recommended (Ger. Pat. 54,206, 1890), the precipitation of the tannin in the waste liquor by means of a 10 per cent. solution of gelatin, and the transportation of the size thus formed, to the paper mills. Owing to the cost of gelatin, Mitscherlich proposed (Ger. Pat. 82,498, 1893) the use of horn and animal hoofs, &c. Several other Ger. Pats. (86,651 and 93,944) also treat of this method of producing a paper-sizing material from the waste sulphite liquor. Mitscherlich has published a paper upon the methods of using this form of tannin-size and also the results obtained by its use in a paper mill in Savoy. These results, although satisfactory, do not appear to have been realised in other mills. According to Mitscherlich's Ger. Pat. 72,161 of 1891, the inventor obtains by means of osmosis, a purer form of tannin suitable for tanning hides, and his patent No. 72,362 deals with the production of a good combustible material suitable for binding the carbon, &c., used in the manufacture of fuel briquettes. C. D. Ekman has obtained a patent (Ger. Pat. 81,643) for the production of a dressing compound for textile material which the inventor calls "Dextron." The liquor is neutralised and then concentrated by quadruple evaporators to 31° B. This concentrated liquid at a temperature of 85° C., is next saturated with magnesium sulphate. The solution of this salt throws out in the form of a scum upon the surface, the so-called "dextron" which is collected, dried and ground. The material, it is said, can be largely used in the place of dextrin. It contains tannin and possesses antiseptic properties, and is sold chiefly to cotton weaving mills and calico printers. From the mother liquor remaining after removing the "dextron," a substance may be obtained by the addition of gelatin, called by the inventors (Cross and Bevan, Eng. Pat. 1548, 1883) "gelalignosin," which is somewhat similar to Mitscherlich's tannin glue. This gelalignosin is insoluble in hot water, but soluble by the addition of a small quantity of alkali. Gelalignosin so dissolved and treated with pulp, produces a size upon the fibres when alum is added.

Frank recommends recovering the free gas by condensing and then saturating the waste liquor by means of milk of lime. Calcium sulphite is formed, which settles down; this is washed and freed from water by settling, or by means of filter presses. The calcium sulphite is used over again for the manufacture of fresh bisulphite liquor. The liquors from which the calcium sulphite has been removed, and which contain calcium hydrate and the organic matter, are run into tanks and a strong blast of air is blown into the liquid, the waste chimney gases being also blown in. The carbonic acid precipitates the lime as carbonate, and the oxygen of the air oxidises the organic matter, rendering it less suitable for the nourishing of fungus growth. Should there still be too much unoxidised organic matter present, it is advisable to run the waste liquor thus treated, upon open fields or waste ground and thence into the water courses.—S. P. E.

## PATENTS.

*Straw and other Fibrous Materials; Disintegrating, Bleaching, and like Treatment of; An Improved Process and Apparatus for the.* G. H. Pond, Ashburnham, Massachusetts, U.S. America. Eng. Pat. 19,797, Aug. 27, 1897.

Straw is packed in a suitable tank, containing a 5 per cent. solution of sodium chloride, in such a manner that there is a clear space between the straw, and anode and cathode respectively, which are placed in the solution.

Arrangements are made, by means of sliding gratings, to close the anode compartment, while the cathode compartment communicates with a central chamber containing the straw, and *vice versâ*. The central chamber is provided with a tube in which a small propeller is placed, and arrangements are made for the distribution of the liquid.

When the grating, connected with the cathode cell, is opened, and the current is turned on, caustic soda is formed, and made to circulate through the straw, while chlorine is given off at the anode. After three hours the cathode grating is closed and the anode grating opened. A solution of chlorine is thus made to circulate through the already disintegrated straw, and this bleaches it. After disintegration by the caustic soda, if desired, both anode and cathode gratings may be opened.—C. M.

*Marbling, Mottling, or Colouring Paper or Cardboard on one Side in the Paper Machine; An Improved Process for.* E. Lehmann, Foekendorf, Germany. Eng. Pat. 25,117, Oct. 29, 1897.

*See under* VI., *page* 244.

## XX.—FINE CHEMICALS, ALKALOÏDS, ESSENCES AND EXTRACTS.

*Monazite Sand of North Carolina.* O. Boudouard. Bull. Soc. Chim. 1898, **19**, [1], 10—13.

Monazite sand is found in considerable quantity in Brazil, Liberia, and North and South Carolina. The deposits in North Carolina are situated in the counties of Burke, McDowell, Rutherford, and Cleveland. The sands are met with in river beds together with other minerals, such as quartz, felspar, hornblende, magnetite, zircon, rutile, corindon, &c. They are disintegrated products of certain rocks, and by virtue of their specific gravity, the richest portions of the alluvial deposits are found in the upper reaches of rivers. They consist of the phosphates of rare earths (cerium, didymium, lanthanum, yttrium), and also contain the oxides of thorium, zirconium, and of titanium, together with silica in varying proportions. The ore forms crystalline spangles varying in colour from pale yellow to yellowish-brown. Its value depends upon the thorium oxide it contains, and the present paper gives a method applied by the author in analysing monazite sands of North Carolina. He states that sands from Brindletown contain 4—6·6 per cent. of thorium oxide, whilst those from Shelley contain 2·76 per cent. Operating with 20 grms. of the finely powdered ore, this was fused with twice its weight of sodium carbonate, extracted with water, and the residue ignited with sulphuric acid and afterwards treated with hydrofluoric acid. The portion remaining, which is always present, is termed insoluble residue. The aqueous alkaline extract serves for estimating the phosphates, whilst the solution of the sulphates is mixed with sodium sulphate and allowed to stand at least 24 hours, when the double sulphate is filtered off and washed with a cold saturated solution of sodium sulphate. The double sulphate of the cerium earths thus obtained, is decomposed with caustic soda, and the oxides are converted into nitrates. These are fused with five parts of potassium nitrate at a temperature not higher than 340° C., and when the evolution of nitrous vapours has ceased, the fused mass is treated with water, when the cerium remains as a dioxide. Ammonia precipitates lanthanum and didymium from the mother-liquor. These can be separated to a certain extent by fusing their nitrates at 160° C., which gives pure lanthanum oxide, although a portion remains with the subnitrate of didymium, which separates below that temperature. The mother-liquor from the double sulphates of the cerium earths is precipitated by ammonia; the filtrate contains the alkalis and the metals of the alkaline earths, with the exception of magnesium, whilst the precipitated oxides, after washing, are boiled with aqua regia, when the titanium separates as metatitanic acid. The neutralised liquor, on treatment with oxalic acid, gives a precipitate of the oxalates of thorium and the yttrium earths. Since the former gives a double sulphate of potassium and thorium, it can be easily separated. The mother-liquor contains zirconium, iron, aluminium, beryllium, magnesium, and the titanium which was not separated as metatitanic acid. Zirconium and titanium are precipitated as thiosulphates, and the former is separated from the latter as a double sulphate of zirconium and potassium. The iron, aluminium, and beryllium are precipitated by ammonia in presence of ammonium chloride, and the oxides are digested with ammonium carbonate to remove the beryllium. Finally the magnesium is precipitated as magnesium-ammonium phosphate. An analysis of monazite sand carried out in this way gave the following result:—$SiO_2 = 9·56$, $TiO_2 = 6·63$, $P_2O_5 = 39·48$, $ThO_2 = 2·42$, $ZrO_2 = 5·75$, $CeO_2 = 12·50$, $Di_2O_3 + La_2O_3 = 8·07$, yttrium bases $(M_2O_3) = 0·48$, $Fe_2O_3 + Al_2O_3 = 9·85$, $Be_2O_3 =$ traces, $MgO = 3·74$, $CaO =$ traces, Gas $= 0·20$, Insoluble residue $= 1·55$. Total $= 100·23$.

According to some chemists a difference is made between the terms "monazite sand" and "monazite," the latter being considered by some as only an integral part of the former. The following analysis from the Chem. Zeit. of Aug. 5, 1896, is thus expressed: Oxide of iron, 3·50; titanic acid, 4·67; silica, 6·40; monazite, 85·43. Total, 100·00. (See also this Journal, 1896, 675).—T. A. L.

*Thorium, The Separation of, from the Cerite Earths.* G. Wyrouboff and A. Verneuil. Comptes Rend. 1898, **126**, [4], 340—343.

Clève pointed out that the addition of hydrogen peroxide to a solution of thorium sulphate, produced a gelatinous precipitate of a salt having the formula $Th_4O_7, SO_3$. On studying this reaction, it was found that the whole of the thorium in a solution of the nitrate could be similarly precipitated as the salt $Th_4O_7N_2O_5$, and the following method was found suitable for the separation of thorium from the cerite earths:—

The solution of the nitrates, containing no more than 0·5 per cent. of oxides, is evaporated to dryness. Then 100 c.c. of water and 10 c.c. of hydrogen peroxide (10 volumes), free from non-volatile impurities, are added, and the whole is warmed for some minutes with stirring. The voluminous precipitate is transferred to a filter and washed until the washings no longer give a precipitate with ammonia. The precipitate is detached from the filter and dissolved in the warm in a few cubic centimetres of water to which 2 grms. of ammonium iodide and 2 c.c. of concentrated hydrochloric acid have been added. Reduction ensues at once, and the warm solution is passed through the filter, in order to dissolve adhering particles of precipitate, and the filter is washed. The solution is precipitated with ammonia, and the hydroxide, which need not be washed, is transferred to the same filter and ignited. The other earths are precipitated with ammonia in the filtrate from the peroxide. The precipitate from the hydrogen peroxide should be perfectly white; if it is not, it contains cerium, and the hydroxide must be redissolved in nitric acid, and the evaporation and precipitation repeated. For exact results, a double precipitation is recommended in the case of the other earths also, since they may contain traces of thoria. The results given in the third column were secured by only one precipitation.

| | I. | | II. | | III. | |
|---|---|---|---|---|---|---|
| | Taken. | Found. | Taken. | Found. | Taken. | Found. |
| ThO .......... | 0·3670 | 0·3655 | 0·3645 | 0·3640 | 0·0508 | 0·0475 |
| $C_3O_4$ .......... | 0·0231 | 0·0229 | 0·0214 | 0·0370 | 0·9485 | 0·9515 |
| LaO, DiO..... | .. | .. | 0·0140 | | .. | .. |

Dennis' method of precipitating thoria from a mixture of the rare earths by means of the potassium salt of hydrazoic acid is faulty, in that the precipitate contains appreciable quantities of cerium, which are not eliminated by a second precipitation. In one analysis, 3·34 per cent. of the ceria was carried down with the thoria, but on dissolving the latter in nitric acid and precipitating with hydrogen dioxide, the filtrate yielded almost exactly that amount of ceria. The method now proposed is therefore more exact than Dennis' process.—J. A. B.

*The Ferrates.* L. Moeser. J. Prakt. Chem. 1897, **56**, [22, 23, and 24], 425—437.

After a review of the various methods which have been proposed for the preparation of the salts of ferric acid, the author describes the process which he has developed from the methods of Foster and C. L. Bloxam, and gives a method for the partial purification of the potassium salt.

80—90 grms. of freshly-prepared ferric oxide, free from manganese, are dried between filter-paper, mixed with 100 grms. of water and 50 grms. of potash (purified from alcohol). To the cold mixture, 50 grms. of bromine are gradually added, and then potash to saturation, care being taken that the liquid does not become too hot. An excess of 20 grms. of potash is added and the liquid warmed to

60° C. for half an hour. Potassium ferrate, being insoluble in concentrated potash, separates, mixed with potassium bromide and bromate, and chiefly at the surface of the liquid. This is dried on a porous plate, washed free from potash by decantation with 96 per cent. alcohol, and, if desired, freed from potassium bromide by solution in a little water and pouring into 85 per cent. alcohol. So prepared, potassium ferrate is an almost black amorphous powder, still containing potassium bromate. It may be kept unaltered in a well-stoppered bottle; it is very soluble in water, forming an alkaline liquid, reddish-black and non-transparent when concentrated. The solution has the same ozone-like odour as potassium permanganate; its oxidising action on wood, paper, &c., is slower than that of permanganate. The concentrated solution quickly decomposes with a rapid evolution of oxygen, but when very dilute, keeps unchanged for hours. Potassium ferrate is insoluble in and is undecomposed by ether, chloroform, and alcohol (unless containing more than 20 per cent. of water). At 250°, oxygen is evolved, and a green mass of potassium ferrite remains, which is decomposed by water into potassium and ferric hydroxides; this action is probably caused by the bromate present. Acids, and salts of acid reaction, at once decompose the ferrate; ammonia is oxidised with evolution of nitrogen. Silver nitrate gives, with evolution of oxygen, a black precipitate of silver ferrite (probably), which is decomposed by washing into silver oxide and ferric hydroxide.

Barium ferrate is obtained as a red precipitate on the addition of barium chloride to the aqueous solution, or may be directly produced by treating a mixture of ferric and barium hydroxides with hypochlorites or hypobromites. Pure barium ferrate is a dark carmine-red amorphous powder, stable when dry, but slowly decomposed by water.

—A. C. W.

### *Mercuroso-Mercuric Iodide, Non-Existence of.* M. François. Jour. Pharm. Chim. 1897, **6**, [10], 443—448.

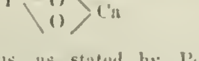
In 1827, Boullay described a "new iodide of mercury," mercuroso-mercuric iodide, $Hg_4I_6$. He gave the details of the method of preparation, which have been quoted in most works of chemistry to the present day. The salt was said to be obtained by adding a solution of iodide of potassium, containing free iodine equivalent to one half the amount combined with the alkali, to a slightly acid solution of mercurous nitrate, the addition of the liquid being stopped as soon as a red tint was developed.

Following Boullay's instructions, it is proved that the mercuric iodide at first precipitated is converted into the mercurous salt, and mercuric nitrate formed; the latter, when this first reaction is complete, is precipitated as mercuric iodide. It is evident, therefore, that the so-called mercuroso-mercuric iodide is nothing but a mixture of mercurous iodide, $Hg_2I_2$, and mercuric iodide, $HgI_2$.

—J. O. B.

### *Calcium Glycerophosphate.* Adrian and Trillat. Journ. Pharm. Chim. 1897, **6**, [11], 481—485. (See this Journal, 1898, 66.)

*Composition of the Salt before and after Precipitation by Heat.*—Crude calcium glycerophosphate was found to contain 25 per cent. of phosphoric acid, and 22·5 per cent. of lime. The same salt dissolved in water and precipitated once by heating to 70° C., contained 30 per cent. of phosphoric acid and 22·25 per cent. of lime. The precipitate thus obtained was found to be the pure salt in the form of microscopic regular needles, which fell to an amorphous powder when exposed to the air; this passage from the crystalline to the amorphous form was observed *sub lente* as the solution evaporated on a microscope slide. The salt thus obtained by precipitation was found to have the constitution—



$$P\left\langle\begin{matrix} O\,C_3H_5(OH)_2 \\ \left.\begin{matrix} O \\ O \end{matrix}\right\rangle Ca \end{matrix}\right.$$

and was anhydrous, as stated by Pelouze, containing neither two molecules of water as stated by Portes and Prunier, nor one molecule as Petit and Polonovski have recorded. The same result was obtained with the salt precipitated from boiling alcohol.

Contrary to the generally accepted opinion, it was found that calcium glycerophosphate was not sensibly decomposed by the prolonged action of heat; it might be heated to 100° C. for several hours without undergoing any material change in chemical composition. The solubility of the pure salt in distilled water was found to be 4·53 in 100 parts at 25° C.

*Preparation of Calcium Glycerophosphate.*—Equal parts (by weight) of phosphoric acid and glycerin are mixed in an enamelled vessel and heated on a sand-bath, to between 130° and 150° C. for 24 hours, when the mixture darkens and begins to evolve acrid fumes. Tribasic calcium phosphate is then added in excess, with which the free phosphoric acid combines to form the dibasic salt. Milk of lime is then added to saturation, which combines with the glycerophosphoric acid, while the dibasic calcium phosphate is reprecipitated as the tribasic salt, which may be used again in subsequent operations. After filtration, the liquid is rapidly evaporated, by direct heat, to a pasty consistence, and then poured into 10 parts of alcohol and boiled for an hour. The precipitate thus obtained. is collected, drained, if necessary again treated with alcohol, and finally dried on the water-bath.—J. O. B.

### *Iodoform, Specific Gravity of.* F. Bergerinck. Chem. Zeit. 1897, **21**, [82], 853.

The specific gravity of iodoform is generally given in text books as "about 2," although the fallacy of this figure is obvious when it is considered that the density of liquid methyl iodide $CH_3I$ is 2·25 at 25° C.; that of methylene iodide $CH_2I_2$, also a liquid, is 3·34 at 5° C., and that of carbon tetraiodide $CI_4$, is 4·32. These facts led the author to determine the specific gravity of iodoform, which he finds to be 4·008 at 17° C.—J. O. B.

### *Iodoterpene.* A. Lieven. Pharm. Zeits. f. Russl. 1897, **36**, 501; through Jour. Pharm. Chim. 1897, **6**, [11], 498.

*Iodoterpene*, to which the formula $C_{10}H_{16}I$ has been assigned, is obtained by the direct combination of iodine and terpene. The body thus produced is a dark-brown liquid, having a terebinthinous odour, a sp. gr. of 1·19 at 15° C., and a boiling point between 165°—175° C.; it is freely soluble in ether, benzene, light petroleum spirit, and in chloroform; in absolute alcohol it is only dissolved to the extent of 10 in 100. It combines in all proportions with oils, fats, and paraffins. From its powerful antiseptic action, it is considered that iodoterpene will be found to be valuable as a surgical dressing.—J. O. B.

### *Acetone, Oil of, and Methylethylketone; Manufacture of, from Fleece Washings.* A. and P. Buisine. Comptes Rend. 1898, **126**, [4], 351—353.

Oil of acetone is the oily residue remaining after the separation of the acetone in the manufacture of the latter, by the dry distillation of crude acetate of lime. It can also be obtained by the dry distillation of the lime salts of the fatty acids formed by the oxidation of fusel oil. Fleece washings contain the volatile fatty acids. Their lime salts, obtained by saturating the acids with lime, and evaporating to dryness, on dry distillation yield 45 to 50 per cent. of their weight of a lightly-coloured liquid of penetrating odour, and acrid burning taste. Its specific gravity is 0·838, and by rectification it yields over 60 per cent. of methylethylketone, indicating that the fatty acids of the fleece washings consist chiefly of propionic acid. About 15 litres of oil of acetone are obtained from a cubic metre of fleece washings of 11 B. The treatment of the washings produced in the towns of Roubaix and Tourcoing would yield about 10,000 kilos. of oil of acetone per day. There is therefore in fleece washings an abundant source of oil of acetone, which is a most efficacious agent for denaturing alcohol, and is used for the purpose in Switzerland.

—J. A. B.

*Rose Oils and Allied Essential Oils, the Constituents of.* J. Bertram and E. Gildemeister. J. Prakt. Chem. 1897, **56**, [22, 23, and 24], 506–514.

H. and E. Erdmann have stated (this Journal, 1897, 823), that pure geraniol (which Erdmann and Huth call rhodinol) cannot be obtained from its compound with calcium chloride. The authors have used this compound for years as a means of separating pure geraniol from rose, citronella, geranium, and other essential oils; none of the preparations contain even a trace of chlorine. Further details of the process are now given:—200 grms. of palmarosa oil are mixed with 200 grms. of dry finely powdered calcium chloride, when the mixture begins to solidify it is placed in a desiccator in a cool place. The solid mass is powdered, triturated with anhydrous ether, benzene or light petroleum spirit, filtered, and by repeated exhaustion with the solvent, freed from uncombined oil. The mixture is decomposed with water, and after two or three washings with lukewarm water, will be free from chlorine. By evaporation of the ether and distillation *in vacuo*, pure geraniol $C_{10}H_{16}O$ is obtained, boiling at 110°—111° C. under 10 mm., at 121° C. under 18 mm., and at 230° C. under atmospheric pressure. The calcium chloride compound may also be decomposed by water, and the geraniol purified by steam distillation without previous washing, but this operation must not be conducted over the open flame, since by superheating, organic chlorine compounds will be produced. Samples of Schimmel and Co.'s geraniol sent to independent chemists before the appearance of Erdmann's paper, have been examined by them and found to be free from chlorine. Erdmann and Huth's statement that commercial geraniol contains citronellol, may be disproved by Tiemann and Schmidt's reaction with phosphorus trichloride (this Journal, 1896, 564). In an earlier communication Erdmann and Huth maintained that the alcoholic constituent of pelargonium oils (the "rhodinol" of Monnet and Barbier, the "réuniol" of Hesse) is identical with geraniol (this Journal, 1896, 292). Barber and Bouveault (this Journal, 1896, 373, 469) have shown that these oils contain, in addition to geraniol, an alcohol $C_{10}H_{20}O$, which Tiemann and Schmidt (*loc. cit.*) have proved to be identical with *l*-citronellol obtained by reduction of the alcohol of citronella oil.

The authors oppose the proposed change of the name of the alcohol $C_{10}H_{18}O$ from geraniol to rhodinol, geraniol and citronellol were the names given to the alcohols $C_{10}H_{18}O$ and $C_{10}H_{20}O$ by their discoverers, Jacobsen and Dodge. Besides, the term rhodinol has been applied to many different substances,—Eckart's rhodinol is a mixture of 20—25 per cent. of citronellol with 75—80 per cent. of geraniol, Barbier and Bouveault's is identical with citronellol, Erdmann and Huth's with geraniol, Tiemann and Schmidt's with *l*-citronellol.—A. C. W.

*The Rhodinol Question.* T. Poleck. Ber. **31**, [1], 29—30.

A PROTEST against changing the name of the alcohol $C_{10}H_{18}O$ from rhodinol to geraniol, and especially against the action of Tiemann and Schmidt in giving the name rhodinol to *l*-citronellol, a body of different composition, and found only in small and varying amounts in rose oils.

—A. C. W.

*Lemon-Grass Oil.* Imp. Inst. J., March 1898, 77.

THIS essential oil is obtained from the plant *Andropogon citratus*, which grows in India and Ceylon. The supply is at present derived almost entirely from the Malabar coast of India, Ceylon distillers having given up the production of oil of lemon-grass in favour of citronella oil.

The odoriferous constituent of lemon-grass oil is *citral*, and this constituent may be present to the extent of from 75 to 80 per cent.

The use of citral for the manufacture of the artificial perfume of violets, called *ionone*, has led of late to an increased demand for lemon-grass oil, and, as a result, the price has risen very rapidly during the last few years.

The following table shows the exports of this oil from India for the past six years, and the prices ruling in London. It should be stated that a case contains 12 pint bottles of oil.

| Year. | Amount Exported. | Price in London. |
|---|---|---|
| | Cases. | *s.* *d.* |
| 1891-92 | 1,450 | 2 0 per lb. |
| 1892-93 | 1,863 | 2 4 ,, |
| 1893-94 | 2,332 | 2 2 ,, |
| 1894-95 | 2,370 | 2 4 ,, |
| 1895-96 | 3,070 | 3 4 ,, |
| 1896-97 | 3,000 | 3 4 ,, to 12*s.* |

The arrival of a considerable quantity of oil in London at the beginning of the year has again lowered the price to about 3*s.* 6*d.* per lb.

*Violets, Artificial Perfume of.* Imp. Inst. J., March 1898, 77.

A PATENT for the manufacture of *ionone*, or artificial essence of violets, was taken out five years ago. The substance is obtained by the action of acetone upon citral (obtained from lemon-grass oil). It is a volatile liquid, dilute solutions of which possess a strong odour of violets. Its composition is represented by the formula $C_{13}H_{20}O$.

It is not yet known whether the natural perfume of the violet is due to the presence of *ionone*, as the amount of oil obtained by distillation of violets is very small.

A substance possessing the odour of violets has been obtained from the rhizome of *Iris florentina*, to which the name *irone* has been given, and this has been shown to have a chemical constitution closely allied to that of *ionone*.

Ionone has recently become very popular, and its manufacture has caused a large demand for citral-yielding oils, such as oil of lemon-grass, and, as indicated in the previous abstract, the price of the latter oil has risen very much in consequence. In the *Chemist and Druggist*, Jan. 22, 1898, it is pointed out that there is another source of citral, *viz.*, the essential oils containing *geraniol*, which is the alcohol corresponding to the aldehyde citral, and yielding the latter by gentle oxidation with the usual reagents, but, as geraniol is somewhat expensive, this process could only be a profitable one in case the price of lemon-grass oil rose to a still higher figure, and there does not appear to be any likelihood of this occurring at present.

*Guaiacum Resin, The Constituents of.* J. Herzig and F. Schiff. Monatsh. für Chem. 1897, **18**, [10], 714—721.

IN a preliminary communication (this Journal, 1897, 352), the authors expressed the opinion that the acid of guaiacum resin contains two hydroxyl groups. The pure acid was isolated as follows:—The resin was treated with sodium bicarbonate solution and the mixture heated by the introduction of steam. After cooling, the resin was twice melted with water, air-dried, and three times extracted with ether. The ether extract was treated with hot benzene, and the solution precipitated by petroleum ether. The filtrate, on evaporation, leaves a residue, which, dissolved in alcohol, gives, after some time, a precipitate of the insoluble potassium salt by the action of alcoholic potash. The acid liberated from this salt and extracted by ether, on evaporation of the solvent, forms a mass of fine needles.

The acetyl-resin acid was obtained by the action of acetic anhydride and sodium acetate. It crystallises from alcohol in fine white needles of melting-point 108°—110° C. Analysis and molecular weight determination point to the formula $C_{18}H_{18}(OCH_3)_2(C_2H_3O_2)_2$.

The compound, which Doebner had described as the monobenzoyl compound of the resin acid, was obtained according to his process and found to contain two benzoyl groups.

The products of the distillation of the acid are pyroguaiacin and guaiacol; no tiglic aldehyde is formed.

—A. C. W.

*Peru Balsam.* K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. **4**, [23], 316—318.

THE author asserts that all commercial samples of this balsam are more or less adulterated, and that the qualitative (German) officinal tests are incapable of detecting any but the grosser cases of adulteration, and hence afford no indication of the relative value of the various qualities.

He subjected to quantitative examination, samples of the pure balsam obtained direct from a Honduras producer. These specimens were the natural exudation from the tree, and had neither been boiled, distilled, nor stored—operations which all commercial parcels of the balsam have to undergo. The data obtained are given below, particulars of the constants furnished by the commercial article being subjoined for comparison:—

| —— | | Acid No. | Saponification Value. | Ester No. | Cinnamein. Per Cent. | Resin Ester. Per Cent. |
|---|---|---|---|---|---|---|
| Natural Honduras balsam.. | No. 1 | 77·46 | 243·07 | 165·61 | 71·41 | 15·00 |
| | „ 2 | 76·92 | 214·34 | 137·42 | 77·56 | 13·18 |
| | „ 3 | 77·34 | 215·01 | 137·67 | 73·63 | 17·32 |
| Commercial balsams................ | | 68·8—75·0 | 260—270 | 188·7—196·8 | 65—70 | 28·00 |

It thus becomes evident that commercial samples of the balsam differ considerably from the pure specimens and must be regarded as "secondary" products, inferior by reason of their lower percentage of cinnamein and higher content of resin ester. All samples that fail to conform to a normal standard of 60—80 for the acid number, 240—270 for the saponification value, together with a high content of cinnamein and a low percentage of resin ester, must, in the author's opinion, be regarded as adulterated.—C. S.

*Camphor-Oil, Light, as an Adjunct to Petroleum.* L. Gans. Chem. Rev. Fett- u. Harz-Ind. **4**, [22], 304.

The author examined a sample of light Japan camphor oil, sp. gr. 0·886 at 15° C., and found that when mixed with petroleum for lighting purposes, it somewhat increased the illuminating power of the flame and diminished the depression of the latter, but increased the consumption of oil.

The camphor oil in question had an acid number of 0·7, and yielded on fractional distillation (Engler method), 81·8 per cent. by weight at below 180° C. (the bulk coming over at about 172° C.); 12·9 per cent. at 180°—200°; 4·9 per cent. above 200° C., and 0·4 per cent. of residue. The viscosity at 20° C. (Engler instrument), referred to water = 1; flashing point (Treumann), 52° C.; burning point, about 60° C.

Its presence in petroleum can be detected by the orange-red coloration resulting on agitation with sulphuric acid (sp. gr. 1·53), the red—turning to brown—coloration and brisk reaction it gives with nitric acid, and by its resinification under the influence of caustic potash.—C. S.

*Kolatannin: The Caffeine Compound in Kola.* J. W. T. Knox and A. B. Prescott. J. Amer. Chem. Soc. 1898, **20**, [1], 34—78.

The authors have previously shown the supposed glucoside of kola nut to be a combination of caffeine with tannin (this Journal, 1897, 366). The present research proves that the tannin thus combined is identical with the free kolatannin of the seeds. This *free kolatannin* was isolated as follows:—The nuts were sliced into boiling alcohol, to sterilize them, and thus prevent the formation of a red colouring body, which otherwise appeared. After a few minutes' boiling they were removed from the alcohol, and dried in a current of warm air, then ground, and extracted by percolation with alcohol of 50 per cent. The alcoholic extract thus obtained was concentrated *in vacuo* and filtered from the insoluble caffeine kolatannate and colouring matter, the rest of the caffeine compound left in solution was then removed by salting out with sodium chloride; the precipitate thus obtained was added to that first separated. The filtrate was washed out, first with chloroform to remove fat and alkaloids, then with ether; finally it was washed out with ethyl acetate, in successive portions, until no more tannin was removed; the solvent was then distilled off, the residual tannin dissolved in water, purified by precipitation with salt, again shaken out with ethyl acetate, and dried *in vacuo* or at 60° to 65° C. Thus obtained, kolatannin was a cream-coloured powder, freely soluble in water, acetone, and alcohol; sparingly soluble in ether; insoluble in chloroform and in petroleum ether.—J. O. B.

*Thyradene.* Pharm. Post. 1897, **30**, 457; through J. Pharm. Chim. 1897, **6**, [11], 503—504.

A PROPRIETARY preparation of thyroid gland.—J. O. B.

*Vanillin, Synthesis of.* L. Bouveault. Bull. Soc. Chim. 1898, **19**, [2], 75—77.

The author has succeeded in partially demethylating veratroyl-carboxylic acid by heating it in an autoclave with potash. The best results were obtained by heating at 160°—170° C., but in every case the vanilloyl-carboxylic acid obtained, was mixed with protocatechoyl-carboxylic acid and unaltered veratroyl-carboxylic acid. On boiling this mixture of acids with an excess of dimethylaniline for about an hour, there results a brown plastic mass with a strong odour of vanilla. This is dissolved in dilute boiling sulphuric acid (10 per cent.), and the solution is shaken out with several successive quantities of ether. The ethereal solution is washed with sodium carbonate solution, which removes the free acids and the vanillin, and leaves behind the veratroyl aldehyde and other neutral bodies. The alkaline solution is acidified and shaken with ether, and the ethereal extract washed with a solution of bisulphite, which removes the vanillin alone. The bisulphite solution is treated with hydrochloric acid, and the vanillin extracted with ether and purified by distillation *in vacuo*, and crystallisation from petroleum ether.—C. A. M.

*Ouabaïn [Strophanthin], Researches on.* Arnaud. Comptes Rend. 1898, **126**, [4], 346—349.

The author has already established the identity of ouabain and the strophanthin of *strophanthus glaber*. (Comptes Rend. **107**, 1011 and 1162.) It forms three crystallised hydrates. One is formed at the ordinary temperature and contains nine molecules of water, of which two are removed when the hydrate is kept in an exsiccator. It consists of quadratic uniaxial crystals. The hydrate which forms at about 30° C. contains four molecules of water, and the third which forms at about 60° C. contains three. The rotatory power of a 1 per cent. aqueous solution of ouabain is $[\alpha]_D = -30 \cdot 6°$. 100 c.c. of water dissolves the following number of grms. of anhydrous ouabain:—at 30° C., 1·57; at 14°·5, 0·93; at 8°, 0·66. Ouabain readily hydrolyses to rhamnose and a resin which is certainly formed by polymerisation of the second product of the hydrolysis. The amounts of rhamnose obtained, support the following equation for the hydrolysis:—

$$C_{30}H_{46}O_{12} + H_2O = C_6H_{12}O_5 + C_{24}H_{36}O_8.$$

The formula $C_{30}H_{46}O_{12}$, corresponding to the molecular weight 598, agrees with the results of observations, and the formation of a heptacetin, $C_{30}H_{39}[C_2H_3O]_7O_{12}$, points to there being at least seven hydroxyl groups in the molecule. —J. A. B.

*Podophyllum, A Chemical Examination of the Constituents of Indian and American.* W. R. Dunstan and T. A. Henry. Proc. Chem. Soc. 1898, [189], 12—13.

The authors find that the constituents of Indian podophyllum (*Podophyllum emodi*) and of American podophyllum (*Podophyllum peltatum*) are identical. The chief constituent is the *podophyllotoxin* of Podwyssozki and Kürsten, which the authors have fully examined. It is a neutral crystalline substance (m.p. 117°), to which the authors assign the formula $C_{15}H_{14}O_6$. It is strongly lævo-rotatory, and acts as a powerful purgative and intestinal irritant. When heated with alkalis, it is converted by hydration into the salt of an unstable, gelatinous acid, *podophyllic acid*, $C_{15}H_{16}O_7$, of which a crystalline sodium salt was obtained, and also silver and copper salts, which were analysed. This acid very readily loses water, and furnishes the crystalline *picropodophyllin* of Podwyssozki and Kürsten, which is isomeric with podophyllotoxin. It passes again into podophyllic acid when warmed with aqueous alkalis. It melts at 227°, and is optically inactive. Podophyllotoxin and picropodophyllin furnish identical decomposition products; when oxidised with nitric acid, *oxalic acid* is the principal product; when fused with alkalis, *orcinol* and *acetic acid* are produced. Both substances contain two methoxyl groups and no hydroxyl. It is concluded that picropodophyllin is the lactone of podophyllic acid, which is probably the hydroxy-carboxylic acid of *dimethoxymethyl-phenylhydro-γ-pyrone.*

The nature of the isomerism of podophyllotoxin and picropodophyllin remains to be determined. The latter substance is therapeutically inert.

The yellow colouring matter of podophyllum, called by Podwyssozki podophylloquercetin, is proved by the authors to be identical with *quercetin*, the colouring matter of quercitron bark.

An uncrystallisable resin, *podophylloresin*, was also isolated and found to exert a purgative action.

Estimations have been made of the amount of "podophyllin" (a mixture of resins with podophyllotoxin which is used in medicine) contained in the two plants. Indian podophyllum contains from 9 to 12 per cent., and American from 4 to 5 per cent. The two resins have been proved to be equally valuable therapeutic agents. The amount of crystalline podophyllotoxin in the Indian plant varies from 2 to 5 per cent., whilst representative samples of the American rhizome were found to contain rather less than 1 per cent.

Indian podophyllum is likely to be valuable both as a drug and as a dyestuff.

*Indian Hemp, Oxycannabin from.* W. R. Dunstan and T. A. Henry. Proc. Chem. Soc. 1898, [189], 44—45.

Oxycannabin is the name given by Bolas and Francis to a crystalline substance they obtained by acting on the pharmacopœial extract of Indian hemp with concentrated nitric acid (Trans. Chem. Soc. 1869, **22**, 417; Chem. News, 1871, **24**, 77). They obtained it in the form of yellow needles melting at 176°, and represented its composition by the formula $C_{20}H_{20}N_2O_7$. In the course of the examination of the constituents of Indian hemp, the authors have prepared the substance in this way, and have also obtained it by the action of fuming nitric acid on the "cannabinol" isolated by Messrs. Wood, Spivey, and Easterfield (Trans. Chem. Soc., 1896, **69**, 544) from the "charas" or resin of Indian hemp.

When quite pure, oxycannabin crystallises in colourless needles melting at 182°; it is insoluble in water, but soluble in hot alcohol, crystallising out on cooling. It sublimes when gently heated, and may be easily purified in this way. On combustion, the substance gives numbers agreeing with those required by the formula $C_{10}H_{10}NO_4$.

It does not dissolve in aqueous alkalis unless warmed with them in a closed tube. By acidifying the resulting solution, an acid is precipitated which is at present under investigation. "Oxycannabin" would therefore appear to be a lactone.

By the action of nitric acid on cannabinol, *normal butyric acid* was also produced, together with *oxalic acid.*

As the substance is thus proved to be a nitro compound, the name "oxycannabin" is inappropriate, but it would be premature to propose a new name until more is known about the constitution of the compound.

*Morphine in Opium, Estimation of.* C. Montemartini and D. Trasciatti. Gaz. Chim. ital. **27**, [2], 302; Chem. Centr. 1897, **2**, [25], 1164.

*See under* XXIII., *page* 282.

*Chloroform: Do Inhalations give Rise to the Presence of Carbonic Oxide in the Blood?* L. de Saint-Martin. Comptes Rend. 1896, **126**, [7], 533.

*See under* XXIV., *page* 283.

*Photography [Organic Developers], Recent Progress in.* A. Granger. Monit. Scient., Feb. 1898, **12**, 86.

*See under* XXI., *below.*

*Pepsin, A Proposed New Method of Examining.* A. H Allen. Pharm. J. 1897, **59**, [1435], 561.

*See under* XXIII., *page* 281.

## PATENTS.

*Methyl Chloride or other Volatile Liquids, Impts. in and relating to Vessels for containing and discharging.* H. Helbing, 63, Queen Victoria Street, London. Eng. Pat. 3803, Feb. 12, 1897.

The vessels, which are intended for use as "auto-sprays" for medical or surgical purposes, are made of non-corrodible material, such as earthenware, china, porcelain, &c., and are closed by a similar non-corrodible screw or bayonet-joint cap, with a packing disc of india-rubber, &c.—R. A.

*Pharmaceutical Preparation [Phenolated Camphor] of Antiseptic and Curative Properties for the Treatment of Sores, Glandular Swellings, and Affections of Mucous Membranes.* J. P. Cleghorn, Ballygunge, Calcutta India. Eng. Pat. 6062, March 8, 1897.

Phenol (one part), heated a few degrees above its melting point, is saturated with camphor (about 16 parts). The caustic properties of the phenol are neutralised by the camphor, and the resulting product is said to be safe for use medicinally, either internally or externally.—L. A.

*Ozone, Impts. in, and in Means for the Production of.* E. Böhme, Barnsbury. Eng. Pat. 7177, March 19, 1897.

*See under* XI. A., *page* 252.

*Guaiacol, Impts. in the Manufacture of.* Kalle and Co., Biebrich-on-Rhine, Germany. Eng. Pat. 7233, March 19, 1897.

The manufacture of guaiacol by diazotising *o*-anisidine and decomposing the diazo compound by boiling with sulphuric acid of not less than 35 per cent. strength, at a temperature above 135° C., is claimed. The guaiacol is thus at once removed from the further action of the diazo compound. It may be purified by distillation. It boils at 200° C., and becomes a solid crystalline mass at about 30° C.—A. C. W.

*Alkylated Uric Acids, Impts. in the Preparation of.* C. F. Boehringer and Soehne, Waldhoff, Mannheim, Germany. Eng. Pat. 7341, March 20, 1897.

In Eng. Pat. 10,005, 1896 (this Journal, 1897, 558), a process is claimed for preparing alkylated uric acids by the action of halogen alkyls on alkaline solutions of uric acid. The patentees now claim the process which consists in substituting for halogen alkyls, such acid esters as have a large electrical conductivity, *e.g.*, methyl nitrate, methyl benzene sulphonate, potassium methyl sulphate. See also this Journal, 1898, 68.—A. C. W.

## XXI.—PHOTOGRAPHY.

*Photography [Organic Developers], Recent Progress in.* A. Granger. Monit. Scient., Feb. 1898, **12**, 86.

Continuing their studies on the organic developers, Lumière and Seyewetz have recently given an account of diamido- and triamidoresorcinol, and also triamidophenol. As already stated, resorcinol is the only dihydroxybenzene which does not reduce the latent photographic image; but, provided the additional groups are suitably arranged, its amido derivatives nevertheless do so. The hydrochloride of 2 : 4-diamidoresorcinol may be used in 1 per cent. solution, mixed with three times its weight of anhydrous sodium sulphite; and, compared with *o*-*p*-diamidophenol (the only other developer of practical utility in the absence of alkalis), it gives very good results. The negatives are soft, the blacks intense, and the half-tones well graduated. It becomes more powerful if the sulphite be raised to 10 per cent. of the whole solution; but above that limit, fog is likely to ensue. Alterations in the proportion of the resorcinol compound, whether increases or decreases, diminish the reducing power, and make the prints weaker. Addition of alkali raises the speed, but not the power, of development; the greatest effect being obtained by the employment of 8 per cent. of a 10 per cent. solution of sodium carbonate. Too much alkali lowers its energy, and leads to the production of fog. Acids retard or even prevent reduction; but they do not permit of correction for over-exposure. The latter is best overcome by the use of 1 to 5 c.c. of 10 per cent. potassium bromide, which is rather a curious fact, seeing that it acts very slightly on the hydrochloride of diamidophenol. Solutions of diamidoresorcinol should not be kept for more than a few days. Experiments with the base itself show it to be a very weak developing agent, and though capable of improvement by the addition of sulphite, its want of stability renders it practically useless. The hydrochlorides of triamidophenol and triamidoresorcinol in 1 per cent. aqueous solution reduce the photographic image feebly by themselves, but their power is greatly enhanced by the introduction of 1·5 parts of sodium sulphite. The picture appears at first very quickly, but development soon stops; the negatives are weak, without half-tones, and generally inferior to those obtained as above. This is presumably due to a secondary reaction such as is observed with cuprous chloride: soluble imides are probably formed, which neutralise the effect of the rest of the developer. Triamidophenol and triamidoresorcinol, therefore, in spite of their theoretically favourable constitution, cannot be employed in photographic practice.

The same authors have previously shown that the introduction of the acid group $COOH$ lowers or destroys the developing power of an aromatic nucleus; that whereas pyrogallol and catechol are developers, gallic and protocatechuic acids, on the other hand, although containing two hydroxyls in the ortho position, will not reduce exposed silver salts till after esterification (ethylic gallate, for example, does develop). They have now investigated the influence of the ketone group $CO$, which also has acid properties; and they find that when a "developing" nucleus (*i.e.*, an aromatic compound containing one or more hydroxyls in the proper position) is joined by the group—$CO$— either to a fatty residue or to another aromatic radicle *not containing any* $OH$, the photographic power of the whole is not sensibly affected. But if one or more hydroxyls be introduced into the second ring, whatever may be their relative positions, the developing power is destroyed. Although it might be expected, therefore, from theoretical considerations, that the photographic activity of a polyphenol should be increased by uniting it, with the help of a ketonic group, to another phenolic nucleus, this is not the case. The following seven bodies accordingly are not developers even in alkaline solution:—2 . 3 . 4 . 2′- and 2 . 6 . 2′ . 5′-tetrahydroxybenzophenone, pentahydroxybenzophenone, δ morintannic acid, hexahydroxybenzophenone, hexahydroxydiphenylketone, and tetrahydroxyphenylnaphthylketone (Alizarin Yellow W); but di- and trihydroxyacetophenone, as well as trihydroxybenzophenone, do develop the latent image.

With reference to the suggested addition of acetone to the pyrogallol developer (this Journal, 1896, 920, and 1897, 259) in place of an alkali, various tests have been carried out with different proportions both of sulphite and of acetone; and as a result, the normal solution may be compounded as follows: "pyro," 1 grm.; anhydrous sodium sulphite, 5 grms.; acetone, 10 c.c.; water, 100 c.c. If there be any fear of over exposure, it is advisable, perhaps, to omit the acetone in the first instance, adding it gradually as development proceeds. The silver reduced, is of a warm black colour, the film has great elasticity, and the gelatin is not stained. Similar experiments with paramidophenol show that although satisfactory negatives may be produced, vigorous and free from fog, yet they are not so good as those obtained by the action of caustic lithia. The best formula is, paramidophenol, 0·7 grm.; sulphite, 10 grms.; acetone, 15 c.c.; water, 100 c.c.

Further investigation has been made by Liesegang of the processes for developing, after a very brief exposure to light, those citro-chloride papers which are primarily intended for "printing-out." Most of the ordinary developers will act in this manner, even if they are dissolved alone in water; but the final colour of the prints and their relative tone-values vary greatly according to the body selected and the way in which it is employed. The best results are given by gallic acid and quinol (hydroquinone); a little alkali or some fused sodium acetate (which is always faintly alkaline) may be added; but even then it is not a case of "alkaline development" in the strict sense of the word, as the paper invariably contains a large quantity of organic acid: true alkaline development would cause the whole surface to turn black. By reducing the acidity of the liquid, the shadows are made darker; by increasing it, the print becomes redder during fixing. In carrying out the operation it is necessary to take note of the amount of free silver nitrate in the paper; if the excess be large, the proofs will be dark and the proportion of acid high, so that less is required in the developer.

A cold-saturated solution of gallic acid brings up the picture in two minutes; it may be hastened by the addition of sodium acetate; but powerful alkalis (except in a very weak bath) and sodium phosphate, which behaves as an alkali, should be avoided. After fixing, the deposit is greenish-black in the shadows. Lead acetate has no effect beyond precipitating some of the gallic acid. Citric and tartaric acids keep the liquid clear for a long time, give a reddish image, which loses strength in the fixing bath, and tend to the production of "sulphur toning." Sodium sulphite acts as a strong retarder, and yields brown tones. Pyrogallol is best used quite alone, and in a sufficiently dilute solution. It produces brown or warm black tints. Quinol gives a print much of the usual colour, which loses intensity and changes to orange in the "hypo" bath. It can be employed alone, acidified with citric or acetic acid, and accelerated with sodium acetate or (more strongly) sodium phosphate. Toning, however, is always necessary. Paramidophenol by itself gives a brown colour, redder in presence of sulphite, whilst the prints must afterwards be treated in a "combined" toning and fixing bath. Metol works well, producing greens and browns. It must be employed alone in a weak solution. If the prints remain too long in the hypo, they are "sulphured." Other developers are either less advantageous or distinctly objectionable for the present purpose.—F. H. L.

### *Glycin* [*Photography*], *Developing with.* A. von Hübl. Wiener Phot. Bl. **5**, 3; Chem. Zeit. Rep. **22**, [1], 32.

Glycin (the chloride of *p*-oxyphenylamidoacetic acid) is universally applicable for developing, having good toning powers (at moderate temperatures), working clear, and being equally suitable for instantaneous and time exposures. The stock developing solution is prepared by dissolving 25 grms. of sodium sulphite in 40 c.c. of hot water, adding 10 grms. of glycin, boiling up, and then adding 50 grms. of potassium carbonate. For use, it is diluted as follows:—Normal or brief exposures, 1 part to 20 of water at 20°—25° C. (a little dilute sodium hydroxide (1 : 10) being occasionally added). Doubtful exposures, 1 part to 20 of water; or, if the peculiarities of the plate are unknown, 1 part to 80 of water, 1—2 c.c. of (1 : 10) potassium bromide being added per litre, and the whole cooled to 10 C. Development is very slow, but considerable irregularities of exposure can be adjusted by this means.—C. S.

### *Protalbin Paper.* E. Valenta. Chem. Zeit. Rep. 1898, **22**, [1], 8.

The author reports favourably on the new protalbin paper (vegetable albumin paper), which has been introduced into the market by Jolles, Lilienfeld, and Co., Vienna. The paper possesses extremely strong powers of resistance, and its other physical properties are quite equal to those of the modern collodio chloride emulsion papers. Its sensitiveness is $1\frac{1}{3}$ times greater than that of freshly silvered albumin paper, the shading of the resulting prints being equal to that produced by most commercial varieties of silver chloride emulsion papers. Prints can be treated separately, in toning and fixing baths, or in a combined bath. The rhodium gold toning bath produces purple and violet-black to almost black shades, which may be intensified by a more prolonged immersion in a similar toning solution, to which strontium chloride is added. The best results are obtained with weak alkaline gold toning baths. With these, prints can be obtained resembling albumin colours. The paper will not bear strong alkaline solutions. Gradation and shading are easily obtained in fixing baths containing gold salts. In baths without gold or in baths containing lead salts, only indifferent results are obtained. After toning, the prints should be thoroughly washed as usual.—J. O. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Explosion Figures.* J. Pinnow. J. Prakt. Chem. 1897, **56**, 520—521.

When mixtures of hydrogen and oxygen are exploded in dusty test tubes, water is deposited in fairly regular rings on the tube. This is due to the aggregation of the dust by the movements of the gases during the explosion, and is not observed in clean tubes. The distance between the rings depends on the diameter of the tube, and probably on the composition of the gaseous mixture. In the same tube, the width of the ring is always the same, and the note produced depends on the composition of the mixture. Thus, the number of single explosions in a given time varies with the composition.—A. C. W.

*Nitro-Derivatives, Chemical Changes caused by the Explosive Decomposition of, in Presence of Oxygen Compounds.* C. Göttig. Ber. 1898, **31**, [1], 25—28.

The author has examined the products of the decomposition of a powder composed of barium nitrate, 9·83; nitrotoluene, 22·22; and nitrocellulose, 67·96 per cent., and gives minute details to illustrate his method of examination in such cases. The residue, after explosion, was found to contain, per cent.—Organic matter (charcoal), 9·51; barium carbonate, 64·44; residue, insoluble in acids, alkaline carbonates, iron compounds, &c., 26·05. For the determination of the gases produced, the powder was exploded by electricity in a closed vessel and the gases collected and examined in the usual way. The amount of moisture they contained was determined partly by noting the increase of volume in the gases when the eudiometer was surrounded by steam and partly gravimetrically. The products of the decomposition were thus found to have the following percentage composition by weight:—Nitric oxide, 8·22; carbon dioxide, 30·89; carbon monoxide, 25·71; methane, 3·69; hydrogen, 0·10; nitrogen, 10·55; water vapour, 8·64; residue, 12·20.

On the basis of these figures the original powder was calculated to have the following molecular composition, in which Eder's formula was taken for nitrocellulose:—

| | | | Parts by Weight. |
|---|---|---|---|
| 11 molecules | $C_{12}H_{14}(NO_2.O)_6O_4$ | = 11·594 | = 6,534 |
| 10 „ | $C_6H_2(NO_2)_3.CH_3$ | = 10·227 | = 2,270 |
| 4 „ | $Ba(NO_3)_2$ | = 4·261 | = 1,044 |
| | | | 9,848 |

and the decomposition taking place on the explosion of the powder may be illustrated by the equation:—

$$11C_{12}H_{14}(NO_3)_6O_4 + 10C_6H_2(NO_2)_3.CH_3 + 4Ba(NO_3)_2 = 4BaCO_3 + 49H_2O + 9C + 93CO + 72CO_2 + 28NO + 10H + 24CH_4 + 76N.$$

—C. A. M.

*Carbohydrates, Nitration of.* W. Will and F. Lenze. Ber. 1898, **31**, [1], 68—90.

From time to time explosions of guncotton have occurred in magazines, the causes of which were obscure. Since cellulose yields sugar when acted upon by dilute acids, it occurred to the authors that it was not improbable that in these cases the guncotton contained nitrated sugars of less stability than nitrocellulose, and, with the object of throwing light upon this point, they have made a systematic examination of the nitro derivatives of various kinds of carbohydrates, but more especially of the sugars. As a rule these were prepared by dissolving the finely divided carbohydrate in nitric acid of sp. gr. 1·52 cooled to zero, and adding sulphuric acid (sp. gr. 1·84) drop by drop to the chilled mixture. Usually a turbidity occurred, in some cases immediately, in others after some time, followed by a separation of oily drops, whilst occasionally, as in the case of rhamnose, there was a solid precipitate. The nitration products were well washed with ice water, and were then usually solid and friable compounds, some of which remained solid at the ordinary temperatures, whilst others became syrupy when the temperature rose above zero. The majority of the nitro derivatives could be crystallised from alcohol, though in a few instances, amorphous precipitates were obtained. In general the nitro derivatives thus prepared were soluble in acetone, glacial acetic acid, and alcohol; insoluble in water and petroleum spirit. Concentrated nitric acid dissolved them readily, and concentrated sulphuric acid reprecipitated them. They were insoluble in cold hydrochloric acid, and decomposed on warming, with the evolution of chlorine, the monosaccharides more readily than the disaccharides. When boiled with water they were gradually decomposed, with the liberation of nitrogen oxides, and slowly brought into solution. Alkalis readily decomposed the esters, with the formation of products containing little or no nitrogen. They reduced Fehling's solution on warming, and rotated the beam of polarised light. They decomposed between 120° C. and 140° C. on slowly warming, and exploded when the temperature was suddenly raised to a higher degree. Decomposition gradually occurred at 50° C. after a considerable time, and in some cases at the ordinary temperature when the nitrated carbohydrate (sugar) was exposed to sunlight.

1. *Monosaccharides.*—The pentoses yielded tetranitrates—Rhamnose tetranitrate, $C_6H_8O_{13}N_4$, m.p. 135° C. $[\alpha]_D^{20°} = -68·4°$ in 2·3 per cent. methyl alcohol solution. On being kept at 50° C. it lost about 1·2 per cent. of its weight in a month. Arabinose tetranitrate, $C_5H_6O_5(NO_2)_4$, colourless crystals, melting at 85°, decomposing about 120°. $[\alpha]_D^{20°}$ in 4·4 per cent. alcoholic solution freshly prepared $= -101·3°$; after 20 hours $= -90°$. After 24 hours, lost in weight 1·5 per cent.; after four days, 40 per cent. Xylose yielded a mixture melting at 141°, and probably consisting of a tetranitrate and of a dinitrate of xylose anhydride, $C_5H_6O_4(NO_2)_2$, melting between 75° and 80°.

The aldose group of the hexoses yielded pentanitrates. Glucose pentanitrate, $C_6H_7O_6(NO_2)_5$, decomposed at 135°, and lost about 38 per cent. in weight after 24 hours at 50°. $[\alpha]_D^{20°}$ in 6 per cent. alcoholic solution $= +98·7$. Galactose gave two pentanitrates. Of these the less soluble melted at 115° to 116°, decomposed at 126°, and lost about 42 per cent. in weight after 10 days at 50°. $[\alpha]_D^{20°}$ in 6 per cent. solution $= +124·7$. The other pentanitrate melted at 72° to 73°, decomposed about 125°, and after 24 hours at 50°, lost 41·7 per cent. in weight. $[\alpha]_D^{20°}$ in 6·7 per cent. solution $= -57°$. Mannose pentanitrate melted at 81° to 82°, decomposed at 124°, and lost 46 per cent. in weight after 24 hours. Its $[\alpha]_D^{20°}$ in 5 per cent. solution $= +93·3°$. The ketose group of the hexoses behaved very differently to the aldoses on nitration, and instead of pentanitrates, only nitrated anhydrides could be obtained, owing probably to the ready decomposition of the compounds first formed. Lævulose (nitrated at 15° instead of 0°) yielded two trinitrates, $C_6H_7O_5(NO_2)_3$. The α-lævulosan trinitrate melted at 137° to 139°, decomposed at 145°, and after six months at 50° did not lose perceptibly in weight. $[\alpha]_D^{20°} = +62°$ in 1 per cent. solution. The β-trinitrate decomposed at 135°, and lost from 1 to 3 per cent. in weight after 8 days at 50°. $[\alpha]_D^{20°}$ in 5 per cent. solution $= +20°$. From sorbinose only one trinitrate was isolated, the composition of which corresponded to that of sorbinose anhydride trinitrate, $C_6H_7O_5(NO_2)_3$. This melted at 40° to 45°.

As a representative of the heptoses, Fischer's α-glucoheptose was nitrated. This yielded a hexanitrate, $C_7H_8O_7(NO_2)_6$, which melted at 100°, and had $[\alpha]_D^{20°}$ in 3·4 per cent. alcoholic solution $= 104·8°$.

2. *Glucosides.*—Representatives of simple glucosides containing no aldehyde groups were examined. α-methyl glucoside yielded a tetranitrate, $C_7H_{10}O_6(NO_2)_4$, which melted at 49° to 50°, decomposed at 135°, and lost about

0·7 per cent. in weight after 5 days at 50°. A similar tetranitrate was obtained from a methyl-*d*-mannosite.

3. *Bisaccharides.*—Cane sugar yielded an octonitrate, $C_{12}H_{14}O_{11}(NO_2)_8$, with $[\alpha]_D^{20^\circ}$ in 3·4 per cent. alcoholic solution = + 52·2. It melted at 28° to 29°, decomposed at 135°, and was very unstable at 50°, losing about 11 per cent. after three days. Lactose gave an octonitrate which decomposed at 135°, and when kept at 50°, lost in weight after eight days 0·7 per cent., and after 40 days, about 40 per cent. Attempts were made, by varying the method of nitration, to obtain the trinitrate described by Gélis (Journ. Russ. Phys. Chem. Ges. **14**, [1], 253), but unsuccessfully. From maltose an octonitrate, $C_{12}H_{14}O_{11}(NO_2)_8$, was obtained, melting with decomposition at 163°—164°. At 50° it lost 1·3 per cent. in weight after 11 days, and 23 per cent. after 43 days. $[\alpha]_D^{20^\circ}$ in 3·5 per cent. solution = + 128·6. Trehalose also gave an octonitrate, melting at 124°, and decomposing at 136°.

4. *Trisaccharides.*—From raffinose a compound containing 11 nitro groups was prepared, $C_{18}H_{21}O_{16}(NO_2)_{11}$. It melted between 55° and 65°, and had $[\alpha]_D^{20^\circ}$ in 3·6 per cent. solution of + 94·9°. It decomposed at 136°, and after three days at 50° lost 9 per cent. in weight.

5. *Starch.*—By their method of nitrating, the authors obtained a hexanitrate in the form of a white powder, which when perfectly pure, remained stable at 50° for six months. It decomposed at 194° without previously melting.

6. *Wood Gum.*—A pure nitro product was not obtained, but probably only a mixture of the mono- and dinitro derivatives.

The authors point out that of the various derivatives described above, the hexoses are as a rule less stable than the bioses, and the anhydrides more stable than the normal nitrates, and they consider that this fact may have a considerable bearing on the stability of guncotton, since the less stable impurities of the latter can only be completely decomposed and made soluble by continued boiling with water, whilst they are comparatively readily soluble in alcohol.—C. A. M.

## PATENTS.

*Explosives, Safety; Impts. in the Manufacture of.* S. A. Rosenthal, London: communicated from J. Stanislaus v. Romocki, Berlin. Eng. Pat. 4062, Feb. 15, 1897.

The addition is claimed of manganese dioxide, potassium permanganate or bichromate, or similar compound which gives up oxygen on heating, to a mixture of ammonium nitrate and trinitrotoluene.—W. M.

*Explosives, Improved Manufacture of.* G. Beneckè, Southport, Lancashire. Eng. Pat. 28,220, Dec. 10, 1896.

The inventor proposes to add sodium or potassium carbonate or bicarbonate to explosives consisting of resin and ammonium nitrate and sodium or potassium chromate or bichromate. The resin is first melted, and into this the carbonate and bichromate in a fine state of division are stirred. On cooling, the mass is finely ground and mixed with ammonium nitrate.—W. M.

# XXIII.—ANALYTICAL CHEMISTRY.

## *APPARATUS, ETC.*

*Gas Measurement, Four New Methods of.* O. Bleier. Ber. **30**, [18], 2753—2759. (See also this Journal, 1897, 166—167, 634).

I. *A Compensation Method.*—Fig. 1 represents an apparatus for the determination of carbon dioxide and oxygen in air. The two pipettes A and B are united by a capillary tube containing a globule of mercury or water, *g*, forming a differential manometer. At their lower extremity they are connected by india-rubber tubes with the three-way tap $h_3$. A contains, to the mark *a*, 100 c.c., and is divided at *b* into two parts, containing 79·5 c.c. and 20·5 c.c. respectively. B holds, to the mark *c*, 100 c.c., and the graduated portion *c*, *d*, containing 1 c.c., is divided into 100 parts. A water-jacket and stirrer keep the temperature constant.

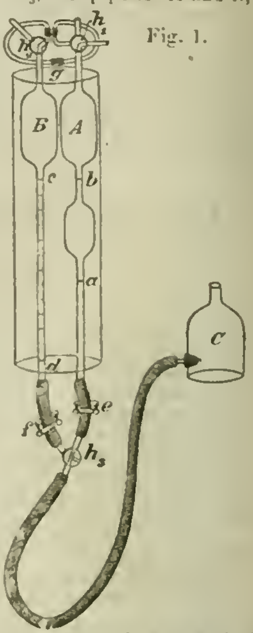
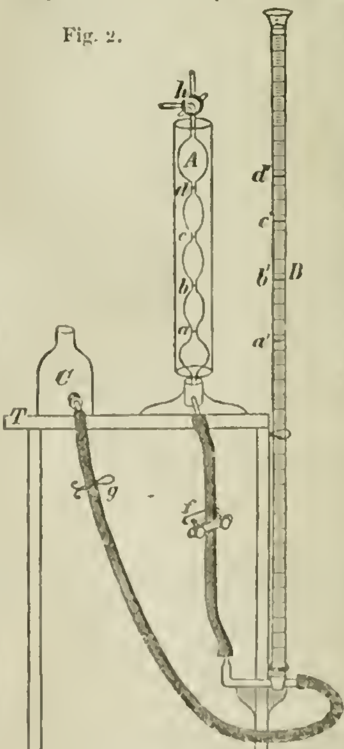


Fig. 1.

By means of the reservoir C, the three-way tap $h_3$, the clips *f* and *e*, and the taps $h_1$ and $h_3$, the pipettes A and B, are filled to the marks *a* and *c* with the gas to be tested and atmospheric air respectively. A momentary opening of the taps $h_1$ and $h_2$ brings the two gases respectively to the atmospheric pressure. On connecting the two pipettes by the taps $h_1$ and $h_2$, the globule *g* must occupy a central position. The gas in A, after absorption of the carbon dioxide in a suitable manner, is expanded to the mark *a* again. When the two pipettes are now reconnected by the taps $h_1$ and $h_2$, the globule in the manometer moves towards A. In order to establish equilibrium of pressure in the two vessels, the air in B must be expanded. This expansion is read off on the scale *c*, *d*, for example, 0·15 c.c. Then the volume of the carbon dioxide absorbed is $\frac{100 \times 0\cdot15}{100 + 0\cdot15} =$ 0·1498 per cent.

When determining oxygen, the amount of gas absorbed is so great that it is impossible to deal with it in the same manner. Instead of expanding to *a* after absorption, the liquid in A is brought to *b*, corresponding to 20·5 c.c., and then the reading is proceeded with as before. The calculation is the same, only now 20·5 c.c. must be added and the carbon dioxide found, subtracted from the result.

Fig. 2.



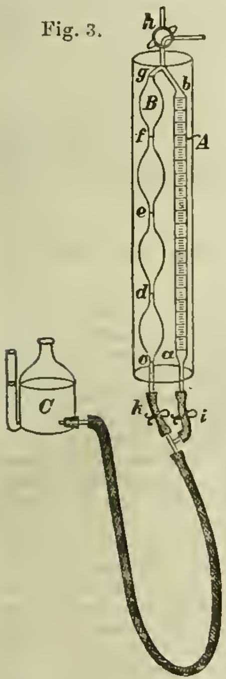
Such an apparatus can only be used for air analysis, but, by suitably modifying the pipettes A and B, it may be used for other gases.

II. *A Modification of Pettersson's Compensation Method.*—By filling the pipettes in the reverse order, B with the air to be examined and A with atmospheric air, the method of O. Pettersson and A. Palmquist (this Journal, 1887, 743) can be applied. After absorption of the oxygen, the compensating air in A is expended to *a*, so that the division *d* now denotes 20·5 c.c. The reading is proceeded with as before, and the value obtained must be multiplied by $\frac{79\cdot5}{101}$; then on adding 20·5 c.c. and subtracting the carbon dioxide, we obtain the volume of oxygen.

On account of the greatly diminished pressures under which this method has to be worked, rendering the use of water impracticable and greatly increasing the danger of leakage, the author prefers Method I.

III. *Gas Measurement by Determination of the Pressure.*—On the table T (Fig. 2) stands the gas burette A, surrounded by a water-jacket. By an india-rubber tube it is connected with the water manometer B, which carries a millimetre scale. Both are joined to the reservoir C by a tube. The burette is closed by a two-way tap *h*, and is divided by a number of constrictions, of known volume, between the marks *a*, *b*, *c*, *d*. On the manometer scale the marks $a^1$, $b^1$, $c^1$, $d^1$ are at corresponding heights.

Fig. 3.



The burette is filled, the tap *h* closed; by lifting C, the gas is compressed to *a*, and *g* closed. The fine adjustments are made, as in Method I., by the screw clip *f*. The pressure of the gas in A is now equal to the pressure of the atmosphere, *plus* that of the water column in the manometer above the mark $a^1$, *minus* the tension of aqueous vapour at the temperature at which the experiment is carried out. The absorption of the first component now takes place, and the subsequent measuring is conducted at constant volume, so far as the water-manometer scale admits. If the amount absorbed be greater than 10—12 per cent., the gas is compressed or expanded to the marks *b*, *c*, or *d*, and then measured by observing the pressure as before. The calculation follows as in Method I., only now pressures are substituted for volumes. The use of water and the moderate pressures to which the apparatus is subjected, renders this method, it is considered, preferable to that of Hempel (Neue Methoden zur Anal. der Gase, p. 108).

IV. *A New Gas Burette.*—Fig. 3 represents a gas burette with two limbs, A and B, the latter acting as a reservoir in which the main volume of the gas may be stored, while the excess is accurately measured in the graduated limb A, which contains between the marks *a* and *b* 20 c.c. The divisions *c d*, *d e*, and *e f*, on the limb B, contain each 20 c.c., while the division *f g* holds 18 c.c., and the space between *g* and *b* 2 c.c., making in all 100 c.c.

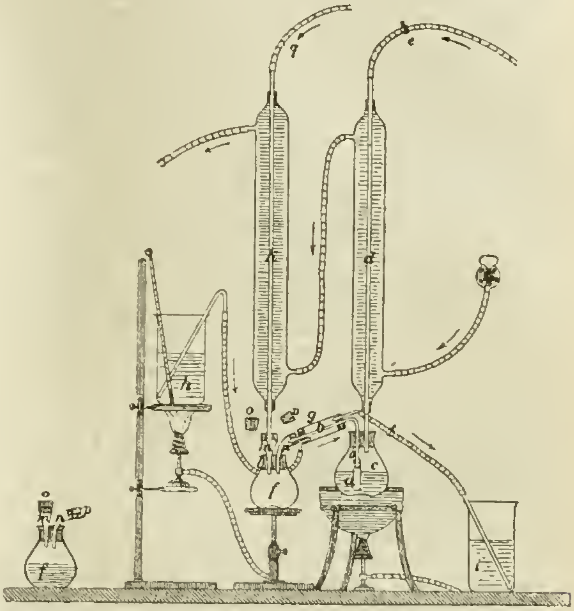
The gas being introduced into the apparatus by means of C and the clip *k* and *i*, the liquid is brought so that it stands at either *c*, *d*, *e*, *f*, or *g*, as the case may be, and the gas in excess of these volumes is measured off on the scale *a b* in the tube A. A narrow side limb on the reservoir C facilitates the adjustment of the pressures.

Thus, the apparatus can be used to measure accurately any volumes between 100 c.c. and 2 c.c. It may be used in combination with a compensator or with any form of gas analysis apparatus, and the constrictions on the limb B may be modified to suit special gases.—H. I.

*Solubility of Substances in Boiling Alcohol, A New Apparatus for Determining.* H. Göckel. Forschungs Ber. 1897, **4**, 173—177.

The author shows that in the usual method of determining the solubility of a substance in a boiling liquid (*viz.*: filtering the boiling saturated solution through a warmed and covered funnel into a weighed flask, and determining the residue in an aliquot portion of the filtrate), there is a considerable loss of the solvent by evaporation, and that the slightest cooling causes a separation of the dissolved substance on the funnel. With the object of avoiding these sources of error, he has devised the apparatus here illustrated.

The substance and solvent are placed in the flask, *c*, which holds about 25 c.c., and in which is also placed the filter-tube *a* which is filled with wool. The flask is heated for about 30 minutes on the water-bath, with *d* acting as a reflux condenser. Meanwhile the liquid in the flask is kept in motion, and prevented from entering the filter, *a*, by means of gentle air pressure supplied by bellows connected with the condenser, *k*, at *q*. After the 30 minutes' boiling, the bellows are detached from the top of *k* and connected with the condenser, *d*, by means of the tube, *e*. On now gently blowing, the boiling saturated solution passes through the filter, *a*, and tube, *b*, and falls into the flask, *f*, where loss by evaporation is prevented by the condenser, *k*. In order to prevent crystallisation in the tube, *b*, the latter is surrounded by a jacket, *g*, through which passes a current of water heated in the beaker, *h*, to a temperature of from 1° to 2° above that of the solvent. The tube *b* delivers into a wider tube, *n*, to which a stopper can subsequently be fitted, and a stopper is also provided for the tube *m*, when disconnected from the condenser, *k*. The flask, *f*, with the tubes, the combined weight of which, when empty, is known, is weighed when containing the saturated solution, and again after the evaporation of the solvent.



A table is given showing the solubility of caffeine in different solvents as determined with this apparatus and by the ordinary method (as given by Fresenius), and in each case the results obtained with the apparatus are much more concordant in duplicate, whilst the amount of solvent required is usually considerably less.—C. A. M.

## INORGANIC CHEMISTRY.—QUALITATIVE.

*Manganese, Rapid Detection and Colorimetric Determination of, in Plants and Earths.* P. Pichard. Comptes Rend. 1898, **126**, [7], 550.

From 0·5 to 1 grm. of the finely-powdered dry earth is incinerated at a dull red heat in a small platinum capsule, and then calcined with twice its weight of finely divided dry sodium or potassium carbonate. The fused mass is then detached from the crucible by the addition of a little dilute nitric acid. The mixture is transferred to a test tube. 0·5 grm. of red lead or peroxide of lead is added, followed by 4 c.c. of water and 2 c.c. of pure nitric acid. The mixture is boiled until the volume is reduced by one half. If manganese be present, the clear liquid, when the insoluble matter has subsided, will have a pink tint. Organic matter is dried, ground, and incinerated; the ash, a few decigrms. of which are sufficient, is treated as above. The amount of manganese present is determined colorimetrically by matching the tint produced with that of a known dilution of standard permanganate solution.—J. O. B.

# *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Acetylene as a Quantitative Reagent. II.* H. G. Söderbaum. Ber. 1897, **30**, [19], 3011—3017; see also this Journal, 1897, 562, 569.

*Separation of Copper and Cadmium.*—The mixture of salts taken (0·5—0·8 grm.) is dissolved in about 150 c.c. of water and 10—15 c.c. of strong sulphurous acid and 20—25 c.c. of ammonia (sp. gr. 0·96) are added. The liquid is warmed on the water-bath until it becomes light blue and then acetylene is passed into the hot liquid. The precipitate is washed two or three times by decantation with dilute ammonia saturated with acetylene, transferred to a filter and at once washed with hot water. For weighing, the acetylide is transformed into the oxide by decomposition with nitric acid and ignition of the nitrate. From the filtrate, cadmium is separated as sulphide by means of ammonium sulphide, or, after acidifying and evaporating to remove sulphurous acid, by electrolysis.

*Separation of Copper and Arsenic.*—When the arsenic acids are present as ammonium salts, the separation of copper as acetylide is incomplete; the precipitate carries down arsenic. This may be avoided by the addition of sufficient caustic soda to combine with the arsenic acids. Acetylene is passed into the hot solution until the precipitate has settled and the liquid has become completely clear. The filtrate is evaporated to dryness, the residue oxidised by nitric acid and precipitated by magnesium chloride. The acetylene used in these processes is washed with lead acetate and acid mercuric chloride solutions, the latter to remove phosphuretted hydrogen. In experiments where the mercuric chloride was not used, the precipitate was generally found to be free from phosphorus, occasionally scarcely ponderable traces of phosphomolybdate were obtained.—A. C. W.

*Halogens, Quantitative Separation of.* R. S. Swinton. Pharm. J., 1897, **59**, [1435], 562—564.

*Separate determination of Iodine in any Mixture of Halogens.*—An amount of the material, approximately equivalent to 0·2 grm. of iodine is convenient for the purpose, and is dissolved in 10 c.c. of water in a separator, about 5 c.c. of pure $H_2SO_4$ are slowly added, keeping the vessel cool by means of a stream of water, then thoroughly agitated, first with 5 c.c. of liquefied phenol of 90 per cent. strength, followed by 20 c.c. of saturated bromine water; after agitating, no free bromine should be left, the excess combining with the phenol to form tribromophenol. The liberated iodine is then removed by successive washings of chloroform, each of 10 c.c., until the solvent is colourless. The original solution is again agitated with another portion of liquefied phenol and bromine water, and again washed out with $CHCl_3$, when a further amount of iodine may be obtained. The combined solutions of iodine in chloroform are covered with water and titrated in the usual manner with decinormal thiosulphate.

*Separate Determination of Bromine in the presence of Chlorides.*—From 0·2 to 0·3 grm of the mixture is dissolved in 10 c.c. of water, in a separator and acidulated with 2 c.c. of a cooled mixture of equal volumes of sulphuric acid and water. About 20 c.c. of $CHCl_3$ are introduced, then excess of aqueous solution of potassium permanganate, and the mixture is well agitated. The separated chloroformic solution is then run into a stoppered bottle containing an alcoholic solution of potassium iodide, the original solution being again extracted with chloroform, until all the bromine is removed. The iodine displaced by the bromine is then titrated with thiosulphate in the usual manner.

*Determination of Iodides, Bromides, and Chlorides, in the presence of each other.*—About 10 grms. of the mixture is dissolved in 250 c.c. of water; 10 c.c. of the solution are taken for the determination of the iodide by the method first described. A second portion of 10 c.c. is taken for the bromine determination, the iodine present being eliminated by treatment with sufficient $\frac{1}{5}$-normal potassium permanganate, the amount requisite being one fifth the number of c.c. of the thiosulphate solution used up in the iodine determination. The liberated iodine is washed out with chloroform, and run into an alcoholic solution of potassium iodide. Then an excess of saturated solution of permanganate is added and the bromine liberated, removed by washing with chloroform, the chloroformic solution being added to that first obtained; and the whole titrated as usual. Then, when A = No. of c.c. of thiosulphate required by iodine in 10 c.c. of the solution in the first step and B, the number used up by the bromine and iodine in the second step. B − A = the number of c.c. equivalent to the bromine in the mixture.

Having thus determined the amount of iodine and bromine in the mixture, 10 c.c. of the original solution are titrated in the usual way with decinormal $AgNO_3$ solution and the equivalent for the iodine and bromine already determined deducted from the result, the difference representing the amount of Cl present.

Determinations by this method with mixtures of known quantities of haloid salts in widely different proportions have given results which are very satisfactory. Commercial specimens of alkali iodides and bromides were in many cases found to be far from pure.—J. O. B.

*Iodine, Bromine, and Chlorine, Separation and Estimation of.* A. Carnot. Comptes Rend. 1898, **126**, [3], 187—191.

The methods depend on the following reactions. In a solution containing iodides, bromides, and chlorides, the whole of the iodine is liberated on adding sulphuric acid containing nitrous anhydride. Addition of sulphuric and chromic acids, liberates the bromine; the action begins at the ordinary temperature, but is completed by heating for about an hour at 100°. Chlorides are not affected.

*Estimation of Iodine.*—The solution, diluted to about 200 c.c., is poured into a separating funnel, and treated with a few drops of sulphuric acid saturated with nitrous anhydride. Pure carbon bisulphide (10—15 c.c.) is then added, the funnel closed, and its contents vigorously shaken several times. The bisulphide is then run out into a wet filter, and the contents of the funnel extracted a second time, after adding three or four drops of the nitrous sulphuric acid. The second extract is added to the first, and the contents of the funnel extracted a third time with 2 or 3 c.c. of bisulphide and one or two drops of acid. The bisulphide is then well washed with water (the first washings being returned to the separating funnel), treated with 30 c.c. of 0·5 per cent. sodium bicarbonate solution, and titrated with decinormal or centinormal sodium thiosulphate solution.

*Estimation of Bromine.*—A few cubic centimetres of chromic acid of 10 per cent. strength, and 3 or 4 c.c. of sulphuric acid (diluted with an equal volume of water), are poured into the funnel, which is then closed and floated in a water-bath kept at 100°. After half an hour or an hour, the funnel is removed, allowed to become quite cold, and extracted three times, as already described, with carbon bisulphide. The extract is treated with a little dissolved potassium iodide and 30 c.c. of sodium bicarbonate, and vigorously shaken. The iodine, thus liberated, is titrated with thiosulphate, and the weight of iodine thus found, multiplied by the coefficient $\frac{80}{127}$.

*Estimation of Chlorine.*—The acid solution from the determination of the bromide is diluted to 500 c.c., treated with silver nitrate, and heated. When cold, the liquid is decanted, and the precipitate, which is yellowish, owing to the presence of a little chromate, purified by washing with warm water containing a little nitric acid. The determination is then finished in the usual manner.

The chlorine may also be determined by difference, as follows: — After separating the iodine, the solution is divided into two equal portions. In the one portion the bromine is determined as described, whilst the other portion is precipitated with an excess of titrated silver nitrate solution, and the excess of silver determined by titration with sulphocyanide. The amount of chlorine is then found by deducting the bromine.

Another method is to titrate 1/10th or 1/50th of the original solution with silver nitrate, using potassium chromate as indicator. The amounts of silver corresponding with the iodine and bromine, determined separately, are then deducted. This method is proposed for cases in which, as usually happens, the chloride is largely in excess of the other salts. To detect small quantities of iodides in presence of a great excess of chloride, the solution is treated with nitrous sulphuric acid and a few drops of carbon bisulphide. Very slight amounts of iodine produce a violet or rose colour. Bromides may then be detected by adding chromic and sulphuric acids to the solution, freed from iodine, and heating to boiling. Fluoresceïn paper, held over the flask, at once acquires a rose colour if traces of bromine be present.—N. H. J. M.

### *Hydrofluoric Acid, Determination of.* J. Zellner. Monatsh. für Chem. 1897, **18**, [10], 749—754.

Hydrofluoric acid may be volumetrically determined by adding some excess of alkali, boiling for a short time, and titrating the hot liquid with acid in the presence of phenolphthaleïn. The determinations made by this method, agree satisfactorily with gravimetric analyses of the liquid after the volumetric operation. If the titration be conducted in the cold, results are obtained which agree among themselves, but are about 1 per cent. below the results of the gravimetric process.

The weighing of the acid is accomplished by means of a vulcanised rubber vessel, suspended on the balance, connected below with a vulcanised rubber jet by means of rubber tubing closed with a clip.

The author describes an apparatus by which accurately measured volumes of hydrofluoric acid or similar liquids may be obtained. The specific gravity of the acid may be determined by means of a vulcanised rubber pyknometer or of a metallic sinker coated with pitch, in which a platinum wire is embedded. The results obtained by either method are exact to the third decimal place. The text books, according to Bineau, state that the acid of specific gravity 1·15 contains 35·35 per cent. of $HF$; the author finds for an almost pure acid of specific gravity 1·148, the acid content of 41·1 per cent., and for a less pure acid of specific gravity 1·162, the percentage 40·42. Hydrofluoric acid of different sources often shows tolerably different specific gravities with the same acid strength; this is due to the presence of hydrofluosilicic acid in varying amounts. —A. C. W.

### *Perchlorate in Nitrate of Soda [Chili], Method of Estimation of.* R. Selckmann. Zeits. f. angew. Chem. 1898, [5], 101—102.

The estimation of perchlorate in nitrate of soda, from the difference in the amounts of chloride present before and after ignition, apart from other disadvantages, occupies a considerable time (two hours for fusion). The author recommends a method in which a fusion lasting 15 minutes is sufficient, and for which he claims greater convenience than is to be found in the method depending on the decomposition which occurs with fuming nitric acid in sealed tubes (this Journal, 1897, 358).

The method, which depends on the greater ease of decomposition induced by the presence of some substance, such as lead, which can be oxidised by the oxygen evolved, is carried out as follows:—

Five or 10 grms. of the nitre, the chloride in which is known by a previous estimation, are gradually heated with three to four times their weight of lead turnings in a porcelain crucible of 30—40 c.c. capacity. The salt melts first, followed by the lead, and then begins a reaction between the two substances, gas being evolved and a film of lead oxide covering the fused salt. The mass is stirred with a bent copper wire so as to continually expose a fresh surface of lead to the salt, and the heat supply is regulated to allow of a slight evolution of gas, but not so as to stop it completely. After from 10—15 minutes, when the mass has, after thickening, become pasty, the heat is increased until the crucible bottom has reached dark redness. In a further one to two minutes, all the perchlorate will have been decomposed. A too strong or long heating is to be avoided, since it would cause a fusion together of the chloride and oxide to lumps which are decomposed with difficulty. The cooled melt, which contains only alkali chloride and nitrite, is washed with hot water into a beaker. If the fusion has been carried out at a very low temperature, the whole of the chlorine will be in solution, and red oxide of lead and metallic lead in small globules, will remain insoluble. Generally an insoluble compound of lead oxide and lead chloride is formed; this is recognisable from the yellow colour of the melt. This compound may be split up by dilute nitric acid, but it is preferable to add to the water, 2—3 grms. of sodium bicarbonate or 10 c.c. of a 20 per cent. solution of caustic soda free from chlorine, and gently warm.

The chlorine is estimated in the filtrate by adding silver nitrate and nitric acid, and weighing the silver chloride formed. The volumetric method cannot be used, owing to the presence of nitrous acid, which does not allow of the exact determination necessary in this case.

The author gives the results of a number of analyses of mixtures made by adding pure chloride and perchlorate in known quantity to sodium nitrate; the values calculated and found agree well.

Although the method proceeds easily with sodium nitrate, it is not so easy of manipulation with the potassium salt. In the latter case, about half an hour is required for the fusion, and great attention must be paid to the temperature, or a sudden glowing of the mass may occur.

The author recommends working with a Berzelius spirit lamp, giving a flame 4—5 cm. high.—J. T. C.

### *Cement Testing, Causes of Differences in the Results of.* M. Gary. Mitt. aus den Königl. tech. Versuchsanst. zu Berlin, 1898, **16**, [1], 1.

*See under* IX., *page* 247.

### *Sodium, The Volumetric Estimation of.* H. J. H. Fenton. Proc. Chem. Soc. 1898, [188], 21.

Dihydroxytartaric acid, in presence of dilute sulphuric acid, is readily oxidised by potassium permanganate at the ordinary temperature, the reaction affording a very convenient method for the estimation of the acid or its salts. A method has been devised for the quantitative estimation of sodium based upon this relation and upon the sparing solubility of sodium dihydroxytartrate. The solubility of the sodium salt at 0° is shown to be extremely small, and in presence of excess of a dihydroxytartrate, is practically negligible.

The substance to be examined, in concentrated neutral solution, is mixed with excess of potassium dihydroxytartrate and the mixture is kept at 0° for half an hour. The precipitated sodium salt is washed with a little ice-cold water, dissolved in excess of dilute sulphuric acid, and titrated with potassium permanganate.

The results obtained with sodium sulphate, chloride, nitrate, acetate, and with Rochelle salt, are accurate within 0·3 per cent. The presence of magnesium does not interfere with the accuracy of the results, but ammonium salts, if present in excess, lead to low results. Full details for working the process are given in the paper.

The potassium and ammonium salts of dihydroxytartaric acid have been prepared and examined, and, together with other derivatives of the acid, will be described in a future communication.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

### *Olive Oil (Portuguese): Coloration with Baudouin's Reagent.* A. J. F. da Silva. Bull. Soc. Chim. 1898, **19**, [2], 88—89.

Milliau first pointed out that certain kinds of olive oil contained a colouring matter derived from the aqueous part of the pulp of the fruit, which gave a rose coloration with Baudouin's reagent, and showed that the effect of this could be obviated by applying the test to the separated fatty acids instead of to the oil. Domergue established the same fact in the case of olive oil from Tunis and Algiers, whilst Villavecchia and Fabris found that certain kinds of Italian oil behaved in the same way. Similarly the author finds that the best varieties of Douro oil give the violet coloration with sugar and hydrochloric acid, but that the separated fatty acids from these give a negative result. The samples examined were perfectly normal in appearance and taste, and as regards their physical and chemical constants. As Tocher's reagent gave a negative result both with the oils and their fatty acids, the author prefers it to Baudouin's reagent for the detection of sesamé oil in olive oil. 15 grms. of the oil are shaken with an equal weight of a solution of 2 grms. of pyrogallol in 30 grms. of hydrochloric acid, and after being left for some time, the oily layer is decanted, and the hydrochloric acid warmed for five minutes. In the presence of sesamé oil, a reddish-purple colour is obtained.—C. A. M.

*Butter, Practical Use of the Refractometer in the Examination of.* A. J. Swaving. Landw. Vers.-Stat. **49**, 311—317; Chem. Centr. 1898, **1**, [5], 352.

In the examination of adulterated butter (butter mixed with a substitute), determinations of the saturation number (Sättigungszahl) and the refractometer number, are not always sufficient, and the use of the polarisation - microscope is necessary for determining whether any foreign crystalline fats are present. Pure butter may be distinguished from margarin in a simpler and quicker manner by means of the refractometer. Adulteration of natural butter with vegetable oils—linseed oil, rape-seed oil, sesamé oil, cotton-seed oil, earth-nut (arachis) oil, and olive oil—causes an increase of the refractometer number and a reduction of the saturation number. This is also the case with admixtures of natural butter with horse-fat, lard, or dog-fat.—A. S.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

*Ultimate Organic Analysis, Additional Remarks on the Simplification of.* M. Dennstedt. Ber. **30**, [18], 2861—2862 (this Journal, 1897, 765).

In carrying out the combustion in the manner described by the author in a previous paper (*loc. cit.*), it is often found that the halogen determinations come out too high, especially when dealing with substances containing nitrogen. This is due to the formation of silver nitrite or nitrate, and can be corrected by heating the silver boat in the Bunsen flame for a few moments to the fusion point of the silver haloid, previous to weighing. Even if nitrogen is known to be absent, it is, in fact, always advisable to do this.—H. I.

*Vinegar Analysis and some Characteristics of Pure Cider Vinegar.* A. U. Smith. (Contribution from the Chem. Lab. of Case School of Appl. Science, No. 30.) J. Amer. Chem. Soc. 1898, **20**, [1], 3—9.

The principal sources of vinegar, in the United States, are apple cider and distilled alcohol, the latter being obtained from malt and maize. These two vinegars differ very widely in character, cider vinegar giving from 2 to 4·5 per cent. of solids and 0·5 per cent. of ash, and, when old, has a peculiar, pleasant, fragrant odour and taste. The distilled spirit vinegar contains but little solid matter and but a trace of ash; the odour and taste are merely those of acetic acid and of the caramel used to colour it. The ash of true cider vinegar is fusible, and commences to volatilise at a comparatively low temperature, rendering it necessary, when making this determination, to incinerate several successive small quantities with as little heat as possible. The ash so obtained contains but little chlorides or sulphates and much carbonates and phosphates; the phosphates soluble in water being markedly greater in this vinegar than in other kinds. The addition of natural water to pure cider vinegar is found to materially lessen this figure, since the magnesium and calcium contained in the water give rise to the formation of insoluble phosphates by reacting with the alkali salt of that acid normally present.

Twenty-two samples of pure cider vinegar gave the following average figures for 100 grms.:—Acetic acid, 4·46; total solids, 2·83; ash, 0·39; alkalinity of ash (in c.c. of $\frac{1}{10}$N acid), 38·8; mgrms. of soluble $P_2O_5$, 19·1; mgrms. of insoluble $P_2O_5$, 10·1; original solids, 9·65. The average figures from 65 samples of spirit vinegar were: acetic acid, 3·84; total solids, 0·38; ash, 0·06; alkalinity of ash (in c.c. of $\frac{1}{10}$N acid), 1·1; phosphates, none.

—J. O. B.

*Acidity and Alkalinity, Estimation of, in Coloured Products.* F. Jean. Ann. Chim. Analyt. Appl. **2**, 445; Chem. Zeit. Rep. **22**, [4], 27.

In the case of wines, blacking, &c., if an alkaline reaction be observed, a weighed quantity of the substance is distilled with an excess of ammonium sulphate, the ammonia liberated—which is collected in a vessel containing titrated acid—forming a measure of the alkalinity of the substance. Bodies with an acid reaction are treated with a known excess of normal alkali and distilled in the above manner, the acidity being calculated from the difference between the amounts of alkali used and ammonia found.—C. S.

*Fat Analysis, Uniform Methods of.* F. Ulzer. Chem. Rev. Fett- u. Harz-Ind. **4**, [22], 299—301.

*Acid Number.*—In view of the great variation in the acid number of different classes of fats, no fixed invariable quantity of substance can well be decided upon, it being advisable to alter the amount in inverse ratio to the value of the acid number to be expected. In general, 5—10 grms. of substance (as proposed by Henriques) should be employed, being dissolved in ether, then mixed with half the volume of alcohol and titrated with $\frac{1}{2}$N (or for low numbers $\frac{1}{10}$N) aqueous or alcoholic alkali in presence of phenolphthalein. When the acid number of the oil is very low, 20—30 grms. should be covered with 100 c.c. of alcohol free from acid, well shaken up and titrated with $\frac{1}{2}$N or $\frac{1}{5}$N alkali.

*Saponification Value.*—In preparing the alcoholic potash, it is advisable to follow Henriques' plan of boiling the powdered alkali in 95 per cent. alcohol under a reflux condenser, since the use of dilute alcohol destroys the concordance of the results.

1. Warm saponification:—2—2·5 grms. of substance are treated with 25 c.c. of $\frac{1}{2}$N alkali and 25 c.c. of alcohol, then briskly boiled for half an hour under a reflux condenser, and the excess of alkali titrated with $\frac{1}{2}$N hydrochloric acid. In the case of waxes, 10 c.c. of petroleum spirit (b.p. 100° C.) should be added.

2. Cold saponification:—*a. Neutral Fats.*—3—4 grms. of substance are dissolved in 25 c.c. of cold petroleum spirit, 25 c.c. of $\frac{1}{2}$N soda solution being then added and the mixture shaken up, left for 12 hours at ordinary temperature, and titrated as above. *b. Waxes.*—Warm petroleum spirit (b.p. 100° C. minimum) is used, the saponifying lye being added immediately, warmed to effect solution, and left for 24 hours, the liquid being repeatedly warmed during the titration with $\frac{1}{2}$N hydrochloric acid.

*Iodine Number.*—Reliable results are obtained both with Hübl's and by Waller's reagent, and if the excess of iodine solution be maintained at double the quantity of iodine absorbed, the time of the reaction may be safely reduced to six hours. The iodine solution, which should be prepared from its components 48 hours before use, may be employed so long as 25 c.c. thereof require at least 35 c.c. of $\frac{1}{10}$N thiosulphate solution for their saturation.

To apply the test, 0·1—0·2 grm. of drying oils or fish oils—or 0·2—0·25 grm. of non-drying oils, 0·5 grm. of tallow fats, or about 1 grm. of cocoanut- or palm-kernel oil—is dissolved in 15 c.c. of pure chloroform, and 25 c.c. of the iodine solution is allowed to react thereon for six hours, 20 c.c. of (1:10) potassium iodide solution and 150 c.c. of water being employed for the titration. When a large number of estimations have to be made, it is desirable to determine by an additional titration, before commencing the back-titrations, whether and to what extent any alteration of "titer" occurs during the interval—the reduction, if any, to be taken into account in calculating the results. The conditions of experiment remain the same for the Hübl and for the Waller solution.—C. S.

*Soap, Analysis and Valuation of. Methods fixed by the Société Suisse des Chimistes Analystes.* J. Suisse de Chim. et Pharm. **35**, 491; Bull. Assoc. Belge des Chim. **11**, [7], 194—195.

Only the soda or "hard," and the potash, or "soft" soaps are affected by these regulations.

### A.—*Analytical Methods.*

1. *Determination of Moisture.*—5 to 10 grms. of a good average sample are dried for three or four hours at about 40°—or left in a desiccator for at least 12 hours in presence of sulphuric acid—and finally exposed to a temperature of 100° to 103° C. for six hours.

2. *Fatty Acids.*—5 to 8 grms. of soap or the residue from the preceding operation, are dissolved by warming in 30 to 40 c.c. of water, then treated with an excess of normal sulphuric acid and well shaken up with 100 c.c. of ether. The ethereal solution—or a measured portion thereof—is transferred to a tared glass flask and distilled, the residue being then left in the water oven for two hours and weighed. 96·75 per cent. of the resulting weight represents the total fatty anhydrides present.

3. *Combined Alkali.*—The fatty acids obtained in 2 are dissolved in 20 c.c. of alcohol and titrated with $\frac{1}{2}$N alcoholic alkali and phenolphthalein.

4. *Total Alkali.*—The excess of sulphuric acid in the aqueous solution obtained in 2 is determined by titration, the result being expressed in terms of $Na_2O$ for hard soaps, and of $K_2O$ for soft soaps.

5. When a preliminary test has shown that no free caustic alkali is present, the amount of alkali carbonates is found by the difference between the total and the combined alkali, and expressing the result as $Na_2CO_3$ or $K_2CO_3$ as the case may be.

6. *Free Alkali.*—The alkali is extracted by dissolving 5 grms. of soap in 100 c.c. of alcohol in a graduated test glass, which is then closed, weighed, and heated in the water bath to from 50°—60°. When the solution is clear, a portion—estimated by difference on re-weighing—is decanted and titrated with normal acid.

7. *Determination of Rosin* (*Hübl and Stadler Method*).—1 grm. of the mixed fatty acids and rosin is dissolved by heat in 20 c.c. of alcohol, and the solution neutralised completely with sodium hydrate, after which it is made up to 200 c.c. with water and precipitated by silver nitrate. The precipitate is washed with water on the filter, dried at 100° C., and extracted with ether in a Soxhlet apparatus, the resulting solution being then shaken up with a little dilute hydrochloric acid. After settling, the ethereal portion is decanted and distilled, the residue thus obtained being dried at 100° and weighed.

### B.—*Judging the Value of Soap.*

The essential determinations to be made are :—A. *Quantitative:* Moisture, fatty acids, total alkali, alkali carbonates. B. *Qualitative:* Detection of free alkali. A good soda soap should not contain more than a trace of free alkali, and never more than 0·5 per cent. of alkali carbonates. No free caustic alkali should be present in toilet or medicinal soaps; the latter must always contain the active medicinal ingredients they profess to contain.—C. S.

*Rubber Goods, Estimation of Mineral Matter in.* L. de Koningh. J. Amer. Chem. Soc. 1897, **19**, [12], 952—954.

5 grms. of the very finely divided sample are treated in a covered beaker with 50 c.c. of fuming hydrochloric acid, and after standing an hour, the whole is heated to 70° C. for another hour; 50 c.c. of water are then added, and the insoluble matter filtered off and washed with boiling water. The washing takes some time, owing to the swollen condition of the rubber, but about a litre of water is usually sufficient to render the residue free from acid. The residue is carefully transferred from the filter into a weighed porcelain dish, then dried on the open water-bath, and finally heated for three hours in an air-bath at 105° C. The loss of weight sustained by the rubber, indicates the greater part of the soluble mineral matter. The remainder of the ash is found by ignition. The author states that some samples which, on simple ignition, gave about 50 per cent. of ash, gave only 10 per cent. after the acid treatment, and the ash was then found to consist chiefly of barium sulphate.

It was found that in the treatment with fuming hydrochloric acid, 0·06 grm., or about 1 per cent., of organic matter was abstracted from 5 grms. of rubber.—A. S.

*Diastase of Barley, Observations on the Determination of.* P. Terrat. Journ. Pharm. Chim. 1897, **6**, [11], 494—498.

The codex directs that "1 decigrm. of diastase placed in contact with 100 grms. of starch jelly containing 6 per cent. of starch, at a temperature of 50° C. for six hours, should afford a liquid which will decolorise five times its volume of Fehling's solution." In this official test, nothing is said as to the quality of the water to be used in making the starch jelly. This has been found by the author to be of vital importance, since the trace of alkalinity due to bicarbonates generally found in ordinary drinking water, has a very marked influence in retarding the complete hydrolysis of the dextrin; so that a specimen of diastase which corresponded to the requirements of the official test when distilled water was employed, failed to attain, even approximately, the same reducing power on Fehling's solution when the starch jelly acted upon was made with ordinary water. Experiments showed that the process of hydrolysis of the starch was the same in the two experiments, but that the conversion of the dextrin thus formed, into maltose, was far less complete in the case where ordinary water had been employed. It is considered that the inhibitory influence of the alkali is only exerted upon the second phase of the process of hydration—the conversion of dextrin into maltose. It would appear, therefore, that the energy of the action of diastase on a molecule of a carbohydrate is in direct ratio to its condensation, the more condensed starch molecule being more readily attacked than that of the less complex dextrin. It is noted that the addition of iodine to the dextrins thus produced, does not affect their optical rotation, but, after the lapse of several days, renders them soluble in alcohol, so that they cannot be precipitated by that reagent.
—J. O. B.

*Wine, Chemical Analysis of. Decree of Instructions, issued 25th June 1896, by the German Federal Council.* Veröffentl. d. Kaiserl. Gesundheitsamtes, 1896, **20**, 557; Bull. Assoc. Belge des Chim. **11**, [7], 196—206; [8], 237—244.

1. *Specific Gravity.* — Determinations to be made by means of a pyknometer of 50 c.c. capacity, the mark being situated half-way along the neck, which is 6 cm. long and 6 mm. internal diameter. The instrument is weighed dry after resting 15—30 minutes in the pan of the balance; and after precisely gauging its capacity by three tests with distilled water, it is filled up to the mark with the sample of wine at 15° C. and carefully weighed.

2. *Estimation of Alcohol.*—The same sample (to which is added the rinsings from the pyknometer) is distilled in a 150—200 c.c. flask, along with a little tannin to prevent frothing. The distillate is collected in the pyknometer, and when 35 c.c. have passed over, the contents of the receiver are made up nearly to the mark with water, and kept for half an hour at 15° C., then filled up precisely to to the mark and weighed. The percentage of alcohol is read off by the aid of the Windisch table.

3. *Total Extractive Substances.*—The method employed depends on the proportion of extract; a preliminary calculation being made by the aid of the formula $x = 1 + d - d^1$, wherein $d$ = the density of the wine (1) and $d^1$ = density of distillate made up to original volume (2).

If the value of E (which is found from $x$, and is given in col. 2 of subjoined table) does not exceed 3, then 50 c.c. of the wine, measured at 15° C., are pipetted into a tared 75-c.c. platinum capsule (about 85 mm. in diameter and 20 mm. high), and evaporated to a syrup; then left for $2\frac{1}{2}$ hours in the (boiling) water oven and weighed after cooling in a desiccator.

When E = between 3 and 4, a sufficient quantity of the wine to yield not more than 1·5 grm. of extract is run into the capsule from a burette and treated in the foregoing manner, the weight of extract being in either case calculated to 100 c.c. of wine by the formula $x = 100 \frac{n}{m}$ grms.; $m$ being the number of c.c. taken and $n$ the weight of extract found. For values of E above 4, the table gives directly the number of grms. of extract per 100 c.c.

4. *Estimation of Mineral Matter.*—When less than 4 grms. of extract are present per 100 c.c., the residue from 3 is carefully carbonised in the capsule, and the carbonaceous residue crushed with a platinum rod and extracted several times with hot water. The solution is passed through a small filter, the ash content of which is known, and collected in a flask. The residual carbon is then dried and calcined along with the filter, and when the ash has become white the filtrate is poured in, evaporated to dryness, the residue moistened with ammonium carbonate and lightly calcined once more, and the whole weighed after cooling in a desiccator. Should the extract exceed 4 grms. per 100 c.c., 25 c.c. of wine are evaporated in a platinum capsule, the residue being carefully carbonised and treated as already described.

5. *Estimation of Sulphuric Acid in Red Wines.*—50 c.c. of wine are acidified with hydrochloric acid, and heated gently up to boiling point. A hot 10 per cent. solution of barium chloride (crystals) is added until precipitation is complete, the contents of the flask being then boiled up again and left to settle for six hours in a warm place, whereafter the clear liquid is decanted through a filter containing a known percentage of ash, the residue being washed with hot water, in the flask and on the filter, until the washings give no turbidity with silver nitrate. The precipitate and filter are then dried, incinerated in a tared platinum crucible, calcined, the residue moistened with a little sulphuric acid, evaporated, slightly calcined again, and weighed in the crucible after cooling in a desiccator.

6. *Total Acidity.*—25 c.c. of the wine are heated to boiling and titrated hot with $\frac{1}{4}$N caustic alkali. When normal alkali is employed, a 10-c.c. burette graduated to $\frac{1}{100}$ c.c. must be used. The final point is attained when a drop of the liquid will no longer cause dry, blue litmus paper to change colour. The free acid is calculated to tartaric acid, as follows:—If $a$ = number of c.c. of $\frac{1}{4}$N alkali required to neutralise 25 c.c. of wine, 100 c.c. of the latter will contain 0·075 $a$ grm. of free acid expressed as tartaric acid. If $\frac{1}{3}$N alkali be employed, the formula becomes 0·1 $a$.

7. *Volatile Acids.*—50 c.c. of wine are placed in a 200-c.c. flask and distilled by the aid of a current of steam introduced through a tube reaching to the bottom of the flask, heat being at the same time applied to reduce the volume of the liquid and maintain it constant at 25 c.c. When 200 c.c. of distillate have been collected, the contents of the receiver are titrated with a standardised solution of alkali in presence of phenolphthalein. If $a$ = the number of c.c. of $\frac{1}{10}$N alkali required to neutralise the volatile acids in 50 c.c. of wine, then 100 c.c. will contain 0·012 $a$ grm. of such acids, expressed as acetic acid.

8. *Fixed Acids.*—These are expressed as tartaric acid and calculated as follows:—If $a$ = grms. of total free acids (expressed as tartaric acid) per 100 c.c. of the wine, $b$ the volatile acids (found in No. 7), then $x = (a - 1·25b)$ grms. of non-volatile acids (= tartaric acid) per 100 c.c.

9. *Glycerin.*—(*a.*) Wines containing less than 2 grms. of sugar per 100 c.c. 100 c.c. are evaporated down to 10 c.c. in a porcelain capsule on the water-bath, and the residue treated with 1 grm. of quartzose sand and sufficient milk of lime (40 per cent. $CaH_2O_2$) to equal $1\frac{1}{2}$—2 c.c. per 1 grm. of extract present. After evaporating nearly to dryness, the residue is treated with 5 c.c. of 96 per cent. (vol.) alcohol, and stirred up with a pestle to a uniform pulp with a little more alcohol, then heated, with constant stirring, till boiling commences, and the turbid liquid decanted into a 100 c.c. flask. 10 to 12 c.c. of alcohol are employed to exhaust the residue in the capsule, and are then united to the contents of the flask, a process which is repeated until the latter amount to 95 c.c., whereupon they are cooled down to 15° C., made up to 100 c.c. with alcohol, well shaken up and passed through a folded filter into a graduated glass. 90 c.c. of the filtrate are evaporated (without boiling) in a porcelain capsule, the residue is taken up with a little absolute alcohol, which is then poured into a stoppered cylinder and the capsule washed with more alcohol until 55 c.c. are collected. This liquid is agitated thrice, each time with 7·5 c.c. of pure ether and left at rest until clear, after which it is evaporated in a tared glass—along with the rinsings (alcohol-ether 1 : 1·5) of the cylinder—at a temperature lower than its boiling point, the syrupy residue being dried at 100° C. for an hour, cooled in a desiccator and weighed.

If $a$ represent the weight of glycerin, so obtained, in grms. then 100 c.c. of the wine will contain 1·111$a$ grms. of glycerin.

(*b.*) In the case of wines containing 2 grms. (and over) of sugar per 100 c.c., 50 c.c. are heated on the water-bath in a large flask and treated with quartzose sand and milk of lime until the initial deep coloration disappears and an odour of alkali is manifest. When cooled down, 100 c.c. of 96 per cent. alcohol are added, and the precipitate filtered off and washed with alcohol. The whole is evaporated and treated as in the previous case, but the resulting weight, $a$, must be multiplied by 2·222 to obtain the weight of glycerin in 100 c.c.

10. *The Estimation of Sugar* is effected gravimetrically with Fehling solution. The reagents employed are: a solution of 69·278 grms. of crystallised copper sulphate per litre, and an alkaline solution containing 346 grms. of potassium-sodium tartrate and 103·2 grms. of sodium hydroxide per litre.

The approximate sugar content of the wine is ascertained by deducting 2 from the weight of extract, in grms. If the result does not exceed 1 grm. per 100 c.c., the direct method of estimation may be pursued; otherwise the wine must be diluted to bring the amount of sugar within the above limit, in which case the quantity taken must be such that the final volume on dilution is not less than 100 c.c.

To apply the test, 100 c.c. of the wine (pure or dilute, as the case may be) are neutralised by an alkali in a porcelain capsule, and concentrated to 25 c.c., tannin and colouring matters being then eliminated by shaking up and heating the residue with 5—10 grms. of bone-black and filtering into a flask with a 100 c.c. mark, the mass on the filter being washed until the filtrate nearly measures that volume, whereupon three drops of a saturated solution of sodium carbonate are added and the liquid made up to the mark after shaking. In case any turbidity is produced, the liquid must be set aside to clarify and filtered at the end of two hours. Lead acetate may also be employed to remove the tannin, &c. In such case, 160 c.c. of wine are neutralised and concentrated as already described, and diluted to the original volume, to which 16 c.c. of lead acetate are then added, the whole being shaken up and filtered. Eight c.c. of the aforesaid sodium carbonate solution, or of sodium sulphate (saturated at 20°) are agitated with 88 c.c. of the filtrate and the liquid is filtered once more. These additions having increased the volume of the wine by one-fifth, this circumstance must be taken into account in the subsequent calculations.

*Invert Sugar.*—Equal volumes (25 c.c.) of the copper sulphate solution, tartrate solution, and water are heated to boiling in a porcelain capsule, whereupon 25 c.c. of the wine filtrate are run in from a pipette and briskly boiled for a further five minutes. The cuprous oxide precipitate is removed by aspiration through a tubular asbestos filter of known weight, and is washed, first with water, and afterwards with alcohol and ether, then dried at 100° C., heated strongly in the air, and, the tube being connected with a hydrogen generator, gentle heat is applied until all the copper oxide is reduced to the metallic state. The metal is then weighed after cooling in the current of hydrogen.

*Cane Sugar.*—50 c.c. of the wine filtrate are introduced into a 100 c.c. flask and accurately neutralised with hydrochloric acid, 5 c.c. of a 1 per cent. solution of this acid being then added, and the whole is heated for $\frac{1}{2}$ hour on the (boiling) water-bath. The liquid is then neutralised, evaporated on the water-bath, rendered slightly alkaline with sodium carbonate, and filtered into a 50-c.c. flask, the precipitate being washed until the flask is filled. The inverted sugar is then determined in 25 c.c. in the same manner as before. In calculating the results, the weights of invert sugar per 100 c.c.—as determined from the precipitated copper—before and after inversion with hydrochloric acid, are expressed by $a$ and $b$, the formula $x = 0·95\ (b - a)$, giving the weight of cane-sugar present.

9. *Polarisation.*—Large and accurate polarimeters, giving readings to $\frac{1}{10}$th of a degree, are essential, and the results are expressed in angular degrees, referred to a 200-mm. tube, the readings being taken at 15° C.

*White Wines.*—60 c.c. of the wine are neutralised, concentrated to one-third, made up again with water to the original volume, and treated with 3 c.c. of lead acetate solution, the precipitate being filtered off. 31·5 c.c. of the filtrate are mixed with 1·5 c.c. of saturated sodium carbonate (or sodium sulphate) solution, filtered, and polarised. The increase in volume is taken into account in calculating the results.

In the case of *red wines*, decolorisation being necessary, 60 c.c. are neutralised, concentrated to one-third, filtered, made up with water to initial volume, treated with 6 c.c. of lead acetate solution, and filtered, 33 c.c. of the filtrate being treated with 3 c.c. of a saturated solution of potassium carbonate (or sodium sulphate), filtered again, and the filtrate examined in the polarimeter. In this case the volume of the wine has been increased by one-third.

When lead acetate does not completely decolorise the wine, bone-black must be resorted to, and in that case, 50 c.c. of wine are neutralised, concentrated to one-half, which is mixed and heated with 5—10 grms. of bone-black, filtered, and the bone-black washed with hot water until the filtrate measures 75—100 c.c., according to the amount of sugar present. The liquid is then concentrated to 30—40 c.c., filtered, and made up to 50 c.c. with the washings and water, and polarised. In this case no increase in volume occurs.

12. *Detection of Impure Dextrose by Polarisation*:—(*a*). If the amount of reducing sugar present be found not to exceed 0·1 grm. per 100 c.c. of wine, and the polarimeter reading is lævo-rotatory or of not more than 0·3° dextro rotation, then the sample is free from impure dextrose.

(*b*.) If, with the same limits of sugar content, a dextro-reading of 0·3°—0·6° is obtained, dextrin may possibly be present. In such case it is sought for by the method given in § 19, and at the same time the method below, (*d*), is employed to detect unfermented impure dextrose.

(*c*.) When the total sugar does not exceed 0·1 grm., and the deviation is more than 0·6° to the right, dextrin is sought for as in § 19, and if found, the unfermentable constituents of the impure dextrose are tested for, according to *d*.

(*d*.) When more than 0·1 grm. of total sugar per 100 c.c. is found, dextrose is sought for in the following manner:—

α. The residue from 210 c.c. of wine concentrated to one third, is diluted until the sugar content does not exceed 15 per cent., and completely fermented at 20°—25° C. by means of 5 grms. of strong yeast free from any optically active constituents.

β. Some quartz sand and a few drops of a 20 per cent. solution of potassium acetate are added to the fermented liquid which is then evaporated to a syrup and stirred up with 200 c.c. of 90 per cent. alcohol added by degrees. After distilling off the bulk of the alcohol and evaporating the remainder, the residue is diluted with water to 10 c.c. and well mixed with 2—3 grms. of bone-black, then filtered, and the filtrate made up with washings of hot water to exactly 30 c.c. (at 15° C.). A dextro-rotation of more than 0·5° in the polarimeter indicates the presence of unfermentable constituents of impure dextrose. If the reading is exactly + 0·5° or but slightly in excess of this figure, the bone-black is washed again until 30 c.c. of washings are produced, and the polarimeter reading of this quantity is added to the first reading; and should the second reading amount to more than $\frac{1}{5}$th of the first, another washing is made as before, and a third reading taken.

*Note.*—A dextro deviation may also be due to the presence of certain constituents of some kinds of honey.

13. *Detection of Extraneous Colouring Matters in Red Wines.*—Tests should always be made for the presence of coal-tar dyes; and the wines examined for their behaviour towards lead acetate. Wool fibres, mordanted with alum and sodium acetate should be boiled in the wine, and the dye, thus fixed on the fibre, tested by various reagents. (See Tables of Lehne and Rusterholz running through this Journal for the year 1895.)

14. *Total Tartaric Acid; Free Tartaric Acid; Potassium Bitartrate; Tartrates of the Alkaline Earths.*—(*a*) *Total Tartaric Acid*: 100 c.c. of wine are agitated with 2 c.c. of glacial acetic acid, 3 drops of a 20 per cent. solution of potassium acetate, and 15 grms. of powdered potassium chloride, until the latter is dissolved. After adding 15 c.c. of 95 per cent. alcohol and inciting precipitation, the liquid is left in repose for at least 15 hours at the ordinary temperature, and then passed through a perforated Gooch crucible, fitted with an asbestos filter resting on platinum tissue, or through filter papers supported on Witt porcelain plates, filtration being accelerated by a vacuum in the receiver. A mixture of 15 grms. of potassium chloride, 20 c.c. of 95 per cent. alcohol and 100 c.c. of distilled water is used to rinse and wash the precipitate, the operation being performed in three stages, and the total washing liquid not exceeding 20 c.c. The precipitate is then dissolved in boiling water (free from alkali), heated to boiling, and titrated with $\frac{1}{4}$N alkali, blue-violet litmus paper being used as indicator. The volume (c.c.) of $\frac{1}{4}$N alkali consumed being represented by $a$, 100 c.c. of the wine will contain $0{\cdot}0375\ (a + 0{\cdot}6)$ grms. of total tartaric acid.

(*b*) *Free Tartaric Acid*: Fifty c.c. of completely fermented wine (or 25 c.c. if rich in sugar) are incinerated as described in § 4. The ash is treated with 20 c.c. of $\frac{1}{4}$N hydrochloric acid and 20 c.c. of water, and the whole gently raised to boiling, whereupon it is titrated with $\frac{1}{4}$N alkali in presence of blue-violet litmus paper.

If $a$ = c.c. or wine taken; $b$ = c.c. of $\frac{1}{4}$N alkali used, and $c$ = grms. of total tartaric acid per 100 c.c., then 100 c.c. will contain $c - \frac{(3{\cdot}75\ (20-b)}{a}$ grms. of free tartaric acid. When $a = 50$ c.c., $x = c + 0{\cdot}075b - 1{\cdot}5$; if $a = 25$ c.c., $x = c + 0{\cdot}15b - 3$.

(*c*) *Potassium Bitartrate*: The ash obtained from the same quantity of wine and in the same manner as in *b*, is extracted with hot water, the solution filtered, and filter and capsule well washed, the liquid being then heated gently to incipient boiling, along with 20 c.c. of $\frac{1}{4}$N hydrochloric acid, and finally titrated as in *b*. The volume of wine being represented by $d$, that of the alkali by $e$, and the total tartaric acid by $c$, the value $n$ is estimated by the formula $n = 26{\cdot}67c - \frac{100\ (20-e)}{d}$. If $n = o$ or a negative value, 100 c.c. of wine contain $1{\cdot}2533c$ grms. of bitartrate; but if $n$ has a positive value, the content will be $\frac{4{\cdot}7\ (20-e)}{d}$ grms.

(*d*) *Alkaline Earth Tartrates*: The tartaric acid in this form of combination is calculated from the foregoing results as follows:—If $n = o$ or a negative value, the content is *nil*; but if $n$ has a positive value, 100 c.c. of the wine will contain $\frac{3{\cdot}75\ (e-b)}{d}$ grms. of tartaric acid so combined.

15. *Sulphuric Acid in White Wines.*—Same method as for red wines. (§ 4.)

16. *Sulphur Dioxide.*—A 400-c.c. flask, connected with a $CO_2$ generator and a condenser discharging into a bulbed (Péligot) U-tube, is employed. The air, being expelled by the current of carbon dioxide (which is passed continuously during the operation), 50 c.c. of a solution of iodine (iodine, 5 grms.; potassium iodide, 7·5 grms.; water, 1 litre) are placed in the U-tube and 100 c.c. of wine in the flask, the latter being distilled—with addition of 5 grms. of syrupy phosphoric acid—until half the liquid has passed over. The contents of the U-tube are acidified with a little hydrochloric acid, heated, and precipitated by barium chloride, the barium sulphate being then treated as in § 5. The total $SO_2$ content per 100 c.c. of the wine will then be $0{\cdot}2748a$ grm., $a$ representing the weight of barium sulphate obtained.

As an alternative method, 50 c.c. of wine may be run in from a pipette into a 200-c.c. flask containing 25 c.c. of a (56 grms. per litre) solution of potassium hydroxide, and the contents left to stand for 15 minutes, after mixing. 10 c.c. of dilute (1 : 3) sulphuric acid and a few c.c. of starch solution are added, and the liquid is titrated with $\frac{1}{50}$N iodine solution, added quickly until the blue coloration is persistent after agitation. In this case the quantity of iodine solution being expressed by $a$, the total $SO_2$ content of the wine will be 0·00128*a* grm. per 100 c.c.

A portion of the sulphurous acid in wine being in organic combination and the remainder either free or as alkali bisulphite, the free acid is determined by pouring 50 c.c. of wine from a freshly opened bottle into a 100-c.c. flask filled with carbon dioxide, and titrating it with $\frac{1}{50}$N iodine as before, in presence of 5 c.c. of dilute sulphuric acid. Taking the volume of iodine solution as *a*, the content of free $SO_2$ will be 0·00128*a* grm. per 100 c.c. of wine. The difference between this quantity and the total $SO_2$ gives the amount in combination with organic substances.

17. *Estimation of Saccharin.*—100 c.c. of wine, mixed with coarse, clean sand, are evaporated, and the residue is treated with 1—2 c.c. of a 30 per cent. solution of phosphoric acid, and extracted at a moderately warm temperature with a mixture (1 : 1) of ether and petroleum spirit, until the volume of (filtered) extract attains 200 or 250 c.c. After distilling off the bulk of the solvent, the remainder is evaporated and the residue taken up with dilute sodium carbonate, filtered, evaporated to dryness in a platinum crucible, and introduced by degrees—after mixing with 4—5 times its weight of dry sodium carbonate—into potassium nitrate in a state of fusion. The mass is then dissolved, acidified with hydrochloric acid, and the sulphuric acid precipitated by barium chloride. The weight of this precipitate, multiplied by 0·7857, gives the saccharin content in grms. per 100 c.c. of wine.

18. *Salicylic Acid.*—50 c.c. of wine are mixed—but not sufficiently to form an emulsion—with an equal volume of ether and petroleum spirit (1 : 1) in a tapped funnel, the ethereal layer being then drawn off and the solvent removed by evaporation after passing through a dry filter. A violet-red coloration ensuing on treating the residue with a few drops of ferric chloride indicates salicylic acid. If the coloration be black or red-brown, a drop of hydrochloric acid is added, the mixture taken up with water, and the entire treatment repeated.

19. *Gum Arabic and Dextrin.*—A preliminary test is made by treating 1 c.c. of wine with 10 c.c. of 96 per cent. alcohol. A slight flocculent turbidity may be disregarded, but if a copious, viscous precipitate be formed, the following method is employed:—

100 c.c. are concentrated to 5 c.c., and treated with 96 per cent. alcohol, until a precipitate ceases to form. After two hours this is filtered off, re-dissolved in 30 c.c. of water, and saccharified by three hours' boiling on the water-bath with 1 c.c. of hydrochloric acid (sp. gr. 1·12) in a 100-c.c. flask, the cork of which is traversed by an open glass tube 1 metre in length. When cooled, and rendered alkaline by sodium hydrate, the liquid is diluted and the sugar estimated by means of Fehling solution, as in § 10. Wines free from gum arabic and dextrin, give merely traces of reducing sugar under this treatment.

20. *Tannin.*—100 c.c. of wine, freed from $CO_2$, are neutralised so as to leave 0·5 grm. of free acid; and after adding 1 c.c. of a 40 per cent. solution of sodium acetate, a 10 per cent. solution of ferric chloride is added drop by drop so long as a precipitate forms. Each drop precipitates 0·05 grm. of tannin. The exact determination of this substance is then performed by any of the usual methods.

21. *Chlorine.*—50 c.c. of wine are rendered alkaline by sodium carbonate, heated in a closed glass to drive off all the $CO_2$, and evaporated, carbonised, and incinerated in a platinum capsule as in § 4. The ash, moistened with 1 drop of nitric acid, is extracted with hot water, filtered, and the filtrate precipitated by a sufficiency of silver nitrate solution (1 : 20). After heating on the water-bath and cooling in the dark, the precipitate is collected on a filter of known ash-content, washed with hot water until free from acid, dried at 100° C., and incinerated in a covered crucible. When cold, 1 drop of hydrochloric acid is added, and driven off again by careful heating, the residue being then strongly heated to incipient fusion, cooled in the desiccator, and weighed.

If the weight of silver chloride be expressed by *a*, 100 c.c. of the wine will contain 0·4945*a* grms. of chlorine or 0·816*a* grms. of sodium chloride.

22. *Phosphoric Acid.*—Fifty c.c. of wine are treated with 0·5—1 grm. of a mixture of potassium nitrate and sodium carbonate (1 : 3) and evaporated to a syrup, the residue being

*Table Indicating the Value E., which Determines the Choice of the Method Employed for Estimating the Extractive Matter of Wine.*

| *x.* (§ 3.) | E. | *x.* | E. | *x.* | E. | *x.* | E. | *x.* | E. | *x.* | E. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1·0000 | 0·00 | 1·0195 | 5·04 | 1·0390 | 10·09 | 1·0585 | 15·16 | 1·0780 | 20·26 | 1·0975 | 25·38 |
| 1·0005 | 0·13 | 1·0200 | 5·17 | 1·0395 | 10·22 | 1·0590 | 15·29 | 1·0785 | 20·39 | 1·0980 | 25·51 |
| 1·0010 | 0·26 | 1·0205 | 5·30 | 1·0400 | 10·35 | 1·0595 | 15·42 | 1·0790 | 20·52 | 1·0985 | 25·64 |
| 1·0015 | 0·39 | 1·0210 | 5·43 | 1·0405 | 10·48 | 1·0600 | 15·55 | 1·0795 | 20·65 | 1·0990 | 25·78 |
| 1·0020 | 0·52 | 1·0215 | 5·56 | 1·0410 | 10·61 | 1·0605 | 15·68 | 1·0800 | 20·78 | 1·0995 | 25·91 |
| 1·0025 | 0·64 | 1·0220 | 5·69 | 1·0415 | 10·74 | 1·0610 | 15·81 | 1·0805 | 20·91 | 1·1000 | 26·04 |
| 1·0030 | 0·77 | 1·0225 | 5·82 | 1·0420 | 10·87 | 1·0615 | 15·94 | 1·0810 | 21·04 | 1·1005 | 26·17 |
| 1·0035 | 0·90 | 1·0230 | 5·94 | 1·0425 | 11·00 | 1·0620 | 16·07 | 1·0815 | 21·17 | 1·1010 | 26·30 |
| 1·0040 | 1·03 | 1·0235 | 6·07 | 1·0430 | 11·13 | 1·0625 | 16·21 | 1·0820 | 21·31 | 1·1015 | 26·43 |
| 1·0045 | 1·16 | 1·0240 | 6·20 | 1·0435 | 11·26 | 1·0630 | 16·33 | 1·0825 | 21·44 | 1·1020 | 26·56 |
| 1·0050 | 1·29 | 1·0245 | 6·33 | 1·0440 | 11·39 | 1·0635 | 16·46 | 1·0830 | 21·57 | 1·1025 | 26·70 |
| 1·0055 | 1·42 | 1·0250 | 6·46 | 1·0445 | 11·52 | 1·0640 | 16·60 | 1·0835 | 21·70 | 1·1030 | 26·83 |
| 1·0060 | 1·55 | 1·0255 | 6·59 | 1·0450 | 11·65 | 1·0645 | 16·73 | 1·0840 | 21·83 | 1·1035 | 26·96 |
| 1·0065 | 1·68 | 1·0260 | 6·72 | 1·0455 | 11·78 | 1·0650 | 16·86 | 1·0845 | 21·96 | 1·1040 | 27·09 |
| 1·0070 | 1·81 | 1·0265 | 6·85 | 1·0460 | 11·91 | 1·0655 | 16·99 | 1·0850 | 22·09 | 1·1045 | 27·22 |
| 1·0075 | 1·94 | 1·0270 | 6·98 | 1·0465 | 12·04 | 1·0660 | 17·12 | 1·0855 | 22·22 | 1·1050 | 27·35 |
| 1·0080 | 2·07 | 1·0275 | 7·11 | 1·0470 | 12·17 | 1·0665 | 17·25 | 1·0860 | 22·36 | 1·1055 | 27·49 |
| 1·0085 | 2·19 | 1·0280 | 7·24 | 1·0475 | 12·30 | 1·0670 | 17·38 | 1·0865 | 22·49 | 1·1060 | 27·62 |
| 1·0090 | 2·32 | 1·0285 | 7·37 | 1·0480 | 12·43 | 1·0675 | 17·51 | 1·0870 | 22·62 | 1·1065 | 27·75 |
| 1·0095 | 2·45 | 1·0290 | 7·50 | 1·0485 | 12·56 | 1·0680 | 17·64 | 1·0875 | 22·75 | 1·1070 | 27·88 |
| 1·0100 | 2·58 | 1·0295 | 7·63 | 1·0490 | 12·69 | 1·0685 | 17·77 | 1·0880 | 22·88 | 1·1075 | 28·01 |
| 1·0105 | 2·71 | 1·0300 | 7·76 | 1·0495 | 12·82 | 1·0690 | 17·90 | 1·0885 | 23·01 | 1·1080 | 28·15 |
| 1·0110 | 2·84 | 1·0305 | 7·89 | 1·0500 | 12·95 | 1·0695 | 18·03 | 1·0890 | 23·14 | 1·1085 | 28·28 |
| 1·0115 | 2·97 | 1·0310 | 8·02 | 1·0505 | 13·08 | 1·0700 | 18·16 | 1·0895 | 23·28 | 1·1090 | 28·41 |
| 1·0120 | 3·10 | 1·0315 | 8·14 | 1·0510 | 13·21 | 1·0705 | 18·30 | 1·0900 | 23·41 | 1·1095 | 28·54 |
| 1·0125 | 3·23 | 1·0320 | 8·27 | 1·0515 | 13·34 | 1·0710 | 18·43 | 1·0905 | 23·54 | 1·1100 | 28·67 |
| 1·0130 | 3·36 | 1·0325 | 8·40 | 1·0520 | 13·47 | 1·0715 | 18·56 | 1·0910 | 23·67 | 1·1105 | 28·81 |
| 1·0135 | 3·49 | 1·0330 | 8·53 | 1·0525 | 13·60 | 1·0720 | 18·69 | 1·0915 | 23·80 | 1·1110 | 28·94 |
| 1·0140 | 3·62 | 1·0335 | 8·66 | 1·0530 | 13·73 | 1·0725 | 18·82 | 1·0920 | 23·93 | 1·1115 | 29·07 |
| 1·0145 | 3·75 | 1·0340 | 8·79 | 1·0535 | 13·86 | 1·0730 | 18·95 | 1·0925 | 24·07 | 1·1120 | 29·20 |
| 1·0150 | 3·87 | 1·0345 | 8·92 | 1·0540 | 13·99 | 1·0735 | 19·08 | 1·0930 | 24·20 | 1·1125 | 29·33 |
| 1·0155 | 4·00 | 1·0350 | 9·05 | 1·0545 | 14·12 | 1·0740 | 19·21 | 1·0935 | 24·33 | 1·1130 | 29·47 |
| 1·0160 | 4·13 | 1·0355 | 9·18 | 1·0550 | 14·25 | 1·0745 | 19·34 | 1·0940 | 24·46 | 1·1135 | 29·60 |
| 1·0165 | 4·26 | 1·0360 | 9·31 | 1·0555 | 14·38 | 1·0750 | 19·47 | 1·0945 | 24·59 | 1·1140 | 29·73 |
| 1·0170 | 4·39 | 1·0365 | 9·44 | 1·0560 | 14·51 | 1·0755 | 19·60 | 1·0950 | 24·72 | 1·1145 | 29·86 |
| 1·0175 | 4·52 | 1·0370 | 9·57 | 1·0565 | 14·64 | 1·0760 | 19·73 | 1·0955 | 24·85 | 1·1150 | 29·99 |
| 1·0180 | 4·65 | 1·0375 | 9·70 | 1·0570 | 14·77 | 1·0765 | 19·86 | 1·0960 | 24·99 | .. | .. |
| 1·0185 | 4·78 | 1·0380 | 9·83 | 1·0575 | 14·90 | 1·0770 | 20·00 | 1·0965 | 25·12 | .. | .. |
| 1·0190 | 4·91 | 1·0385 | 9·96 | 1·0580 | 15·03 | 1·0775 | 20·12 | 1·0970 | 25·25 | .. | .. |

carbonised, extracted with nitric acid, filtered, washed several times, and incinerated with the filter; then moistened with nitric acid, taken up with hot water, and the (filtered) liquid added to the first extract. The liquid is treated with 25 c.c. of molybdic solution (150 grms. of ammonium molybdate in 1 litre of dilute [1 per cent.] ammonia) and 25 c.c. of nitric acid (sp. gr. 1·2), and heated to 80° C. After six hours' repose in a warm place, the clear liquid is decanted on to a filter, and the precipitate washed four or five times by decantation with dilute molybdic solution (100 vols. of above solution, 20 vols. of nitric acid (sp. gr. 1·2), and 80 vols. of water); then dissolved in concentrated ammonia, passed through the same filter, and treated, together with the (ammoniacal) washings, with hydrochloric acid until the precipitate first formed is re-dissolved. When cold, 5 c.c. of ammonia are stirred in, followed by 6 c.c. (drop by drop) of magnesium mixture—magnesium chloride 68 grms., and ammonium chloride 165 grms. [both in solution], mixed with 200 c.c. of ammonia [sp. gr. 0·96], and made up to 1 litre—40 c.c. of ammonia being then added, and the whole left for 24 hours, whereafter it is filtered and the precipitate washed with dilute ammonia (1 : 3) until the washings cease to become turbid in presence of acidified silver nitrate. After drying and incineration, the precipitate is cooled, moistened with nitric acid, and calcined, cooled, and weighed. The weight of magnesium pyrophosphate, when multiplied by 1·2751, will give the weight of $P_2O_5$ present in 100 c.c. of the wine.

23. *Nitric Acid.*—1. White wines: (*a.*) Ten c.c. of wine are freed from alcohol and colouring matter, and filtered, a few drops being then tested with diphenylamine and sulphuric acid for the nitric colour reaction (blue). (*b.*) More minute traces of nitric acid may be detected by evaporating 100 c.c. of wine to a syrup, which is then treated with absolute alcohol, so long as a precipitate forms, filtered, heated to drive off the solvent, and after decolorising the residue and concentrating it down to 10 c.c., employing the above test (*a*) to the filtered liquid.

2. Red wines: 100 c.c. of wine are treated with 6 c.c. of lead acetate, filtered, and mixed with 4 c.c. of concentrated magnesium sulphate solution and a little bone-black. After standing awhile and filtering, the liquid is tested in the same manner as white wine.

24 *and* 25. *Barium and Strontium.*—The ash from 100 c.c. of wine, incinerated as in § 4, is taken up with dilute hydrochloric acid, the solution evaporated to dryness, and the residue examined by the spectroscope. If either metal be present, a quantitative estimation is made by the usual methods.

26. *Copper.*—This metal is estimated by electrolysing ½—1 litre of wine, the deposited copper being weighed, dissolved in nitric acid, and tested in the usual manner.

—C. S.

### *Beer and other Liquids containing Acid Phosphates. Determination of Acidity in.*

A. Ott. Zeits. für d. ges. Brauwesen, **20**, [51], 667—668; [52], 682—685. (See also this Journal, 1898, 188.)

*Determining the Acidity of Sour Beer; and Detection of added Neutralising Agents.*—The amount of N alkali used in titrating the liquid with violet-red litmus indicator, increases regularly with the quantity of free acid present, whilst with violet-blue litmus, the increase of alkali is slightly more, though the *ratio* between the two quantities of alkali required diminishes. In view of this the author proposes to use litmus papers (red and blue) of definite shades, ascertain the exact difference in the quantities of alkali consumed in either case, and compare this difference with the amount required with the red paper: the more free acid present, the higher will be the figures for the red paper in proportion to the said difference.

For detecting added neutralising agents, he suggests the distillation of 100 c.c. of the liquid *per se*, and of a similar quantity after addition of a little N-sulphuric acid or pure phosphoric acid, titrating the distillates with $\frac{1}{10}$N alkali and phenolphthalein, a higher figure for the second titration indicating the presence of such neutralising agents. As an alternative method, the distillate (driven over by steam) is collected in fractions of 100 c.c., and as each fraction will contain less acetic acid than its predecessor, the $\frac{1}{10}$N alkali required for neutralisation will be a continually decreasing quantity. On distilling further in presence of a little (about 1 c.c.) of N-sulphuric acid or phosphoric acid, the next fraction collected will—if neutralising agents have been added to the beer—contain an exceptionally large proportion of acetic acid. In this manner the author has succeeded in detecting as little as 0·042 grm. of added sodium bicarbonate per 100 c.c. of beer.

In addition to the particulars already given, the author states that the acid reaction of normal beer is mainly due to acid phosphates, free acids playing only a subsidiary part, but other bodies—probably amido-acids—are also concerned. In normal worts, the same conditions prevail, except that free acids are absent. He also considers that the method of estimating the acid content of beer, adopted by the Bavarian Chemists' Association—*viz.*, titrating with normal alkali at 40° C., with a "neutral" (violet) litmus paper as indicator and characterising as permissible acidity, the equivalent of 3 c.c. of N-alkali per 100 c.c. of beer—requires modification, as do also the opinions hitherto held with regard to the percentage of acetic acid in normal beers.—C. S.

### *Phenol, Estimation of, in Disinfectants in presence of Soaps.*

W. Spalteholz. Chem. Zeit. 1898, **22**, 58.

A DETERMINATION of phenol in commercial carbolic acid is usually effected by means of the phenolates of the alkalis, but calcium phenolate is not suitable for this purpose since it is easily decomposed in an aqueous solution. It is not possible to determine the phenols by the ordinary saponification method in those cases in which the phenol preparations, like creolin and lysol, &c. contain soaps. The following method has given results within 0·5—1 per cent. of the calculated value, which is sufficiently close for the valuation of a disinfectant material. The substance to be examined is carefully heated to 200° C. in a small iron boiler, the temperature being gradually raised to 220° C. and the mixture steam-distilled so long as oil drops pass over with the water. Substances containing olein soaps must not be heated above 210° C., as above this temperature they are also decomposed. The distillate is then taken up with benzene, and the phenol which dissolves, is estimated with soda lye. Some samples of lysol examined by the author, contained from 50—60 per cent. of phenol, whilst samples of creolin contained from 0—20 per cent. The bactericidal properties of creolin, however, are partly due to the presence of coal tar hydrocarbons, and even a creolin free from phenol was found to be active in this respect. It is therefore necessary to value different sorts of creolin according to the amount of phenol contained, in conjunction, however, with its property of forming an emulsion with water. A creolin which gives an oily deposit on mixing with water has the same disadvantages as the common crude carbolic acids which come into commerce mixed with tar oils.—T. A. L.

### *Pepsin, A Proposed New Method of Examining.*

Alfred H. Allen. Pharm. J. 1897, **59**, 1435, 561—562.

IN order to determine the actual peptic value of pepsin, the amount of the true peptone formed, as distinct from the other products of the action of pepsin and acid on egg albumin, is suggested as forming a reliable means of obtaining satisfactory data in the critical examination of pepsin. The details of the process provisionally suggested, are as follows:—About 1 grm. of egg albumin in scales is powdered and dissolved in 20 c.c. of warm water. It is then heated in the boiling water bath to coagulation, and cooled to a temperature not exceeding 40° C.; 0·100 grm. of the pepsin to be examined, is next added, followed by 25 c.c. of decinormal HCl, the liquid is then warmed to 40° C. and kept at that temperature for 3 hours. A volume of decinormal sodium carbonate exactly equivalent to the acid employed, is then added, and the mixture heated to 90° C. on the water bath for 10 minutes. It is then cooled, diluted with water to 100 c.c., and passed through a dry filter. 50 c.c. of the filtrate is then saturated in the cold, with powdered zinc sulphate (about 60 grms. being required), allowed to stand with occasional agitation for half an hour and filtered. The precipitate thus produced may be washed with a cold saturated solution of zinc sulphate, a preferable method being, however, to weigh the liquid containing the precipitate, pass through a dry filter and weigh the filtrate. The subsequent result, derived from the portion of the filtrate thus obtained, is then calculated to the total weight of the liquid. This method of precipitation with zinc sulphate, due to Bömer, removes all the proteids except peptone. The filtrate is then acidulated with hydrochloric acid, and treated with excess of bromine water: the mixture is stirred to cause the yellowish precipitate to aggregate, allowed to stand for half an hour, then decanted through a plug of glass wool covered with pulped asbestos, fitted in a cylindrical funnel. The precipitate adhering to the sides of the beaker is washed with cold water, the washings being poured through the filter.

The contents of the filter tube, with the plug, are returned to the beaker, and the whole decomposed with 20 c.c. of sulphuric acid and treated by Gunning's modification of Kjeldahl's method for the determination of nitrogen. Where Kjeldahl's method cannot be conveniently employed, the precipitate obtained by bromine may be dissolved in hot 97 per cent. alcohol, the solution evaporated to dryness at 100° C., and the residue weighed. The albumoses may be deduced from the nitrogen found in the zinc precipitate, after washing with saturated solution of zinc sulphate.

—J. O. B.

*Morphine in Opium, Estimation of.* C. Montemartini and D. Trasciatti. Gaz. chim. ital. **27**, [2], 302—335; Chem. Centr. 1897, **2**, [25], 1164.

10 GRMS. of powdered opium (dried at 100° C.) are macerated for 1 hour with 90—100 c.c. of a 20 per cent. sodium chloride solution, and the solution is filtered. The residue is again triturated with 60 c.c. of the sodium chloride solution and then filtered through the same filter, this process being repeated until the filtrate appears colourless, or until a few drops give no colour reaction with Froehde's solution. The united filtrates are evaporated by means of gentle heat and with frequent stirring, so that a fine powder is left. This is exhausted with about 300—350 c.c. of absolute alcohol in small portions, until a fresh portion of the alcohol gives no longer the reaction with Froehde's reagent. [Froehde's reagent is a freshly prepared solution of 0·01 grm. of sodium molybdate in 1 c.c. of concentrated sulphuric acid.] The alcoholic extract is now evaporated to dryness and the residue covered with 15 c.c. of water, that has been rendered faintly alkaline, and allowed to stand for 24 hours. The precipitate is then collected on a tared filter, washed with water containing morphine, until the runnings are colourless, and then dried at 100° C. The paper and the precipitate are then placed in a separating vessel and repeatedly extracted with chloroform, till a few drops of the latter give no cloudiness on evaporating, taking up with hydrochloric acid, and treating with caustic soda. Finally the chloroformic solution is evaporated to dryness and the residue dried at 100° C. and weighed. This method takes 4 days, but the authors are seeking to modify it, in order to shorten the time occupied in carrying it out.

On an examination of the different methods for estimating morphine in opium, it was found that the above method, and those of Perger and Squibb (Ephemeris, 1882 and following Nos.), gave the best and most concordant results; the volumetric methods were found to be wholly unreliable.

—A. S.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Graphite in Siberia.* R. Helmhacker. Eng. and Mining J. 1897, **64**, [26], 756.

THE most important deposits of graphite in Siberia occur (1) on the Bogodol *golec*, situated in the mountains of Toonkinsk, in the government of Irkutsk; (2) in the district of Tourookhansk, in the northern portion of the government of Yenisseisk; and (3) in the eastern part of the Kirghiz steppes in the foothills of the Tarbagatai.

The Bogodol mountain deposit presents several large vein-like pockets in a heavy bedded granite, also in syenite and in crystalline limestone; the pockets are sometimes 25 m. thick. The mine opened by Aliber, the discoverer of the deposit, has been worked since 1856 under the name of the Mariinski mine; and from the indistinctly defined graphite body, about 1650 tons have been prepared for working. When the first deposit was exhausted, few attempts were made to discover others, and the claim being deemed unworthy of further investigation, the mine was closed. The present limited output is obtained by hand picking from the old workings. The chief difficulty is that of transport. An analysis of the Bogodol graphite by Pouzirevsky and Koutorgo showed 84 to 90 per cent. of carbon, with 10 to 15 per cent. of silicious ash. The Tourookhansk deposits occur embedded in clay slate; they show a slaty or radiated texture, and it is probable that the layers of graphite are in some places the result of the alteration by heat of the coal of the Jurassic formation. In 1861, when the deposit on the banks of the Kooreika river was discovered, 70,000 pouds of graphite were extracted; 2,000 pouds were exported in 1877, and in 1891, about 10,000 pouds were taken out. The graphite obtained has been stated to be better than the Ceylon graphite, and to be equal in quality to the Cumberland variety. The deposits at Tarbagatai rank among the richest and most promising in the world, but the production is variable, there being no shipment at all in some years. The greatest production took place in 1875, when 18,500 pouds (303,030 kilos.) were exported (see also this Journal, 1896, 300).

—A. S.

*Strontium Sulphide, Phosphorescent, Production of, from Strontium Sulphite and Thiosulphate.* J. R. Mourelo. Comptes Rend. 1898, **126**, 420—423. (See also this Journal, 1898, 189.)

PHOSPHORESCENT sulphides have already been obtained by Forster (Poggendorff Annalen, **133**, 94) from strontium thiosulphate and sulphite. Going over the same ground, the author finds that these salts, although completely converted into sulphate when strongly ignited, are decomposed, when cautiously heated, into SrS, $SrSO_4$, and free S (in the case of the thiosulphate). The products exhibit yellow-green and blue-green phosphorescence respectively, the intensity being proportionate to the percentage of SrS present (maximum, 14—15 per cent.). By the addition of alcohol to mixed solutions of $SrCl_2$ and $Na_2S_2O_3$, or $Na_2SO_3$, crystalline precipitates of the corresponding strontium salts, intimately mixed with sodium chloride, are obtained. These impure salts, when ignited at a bright red heat for some hours, are decomposed in the same way as the pure salts at a lower temperature, yielding fused white masses, which, when coarsely powdered, become strongly phosphorescent (yellow- and blue-green as before) after a short exposure to daylight. A mixture of the impure strontium salts behaves similarly. The strontium sulphide (13 per cent.) present, is, however, gradually oxidised on exposure to the air, and the products finally become inert.

—H. T. P.

*Ozone, Experiments with.* M. Otto. Ann. Chim. Phys. **13**, [7], 77—144; Chem. Centr. 1898, **1**, [5], 307.

As a result of a number of experiments with regard to the construction of the producers, tension of current, distance between electrodes, &c., the author concludes that the yield of ozone is proportional to the number of periods of the alternating current employed.

The action of ozone on several organic bodies was examined. From methane, formaldehyde and formic acid were obtained, and from ethylene, acetaldehyde and acetic acid, but there was no production of alcohol or glycol. Ozone reacts with acetylene, developing both heat and light, sometimes with explosive violence. Benzene gives "Ozobenzene" ($C_6H_6O_6$). Eugenol, safrol, estragol, and their isomers form the corresponding aldehydes. Thus, isoeugenol yields vanillin, isosafrol, and piperonal. Alcohols react but slightly with ozone, forming usually the corresponding aldehydes and acids; phenols also are only slightly attacked. Aniline gives quinone and azobenzene, and *p*-toluidine gives azotoluene.—A. S.

### *Ammonia and Hydrochloric Acid, Reaction between Dry and the Vapour Density of Dry Ammonium Chloride.*

S. Gutmann. Annalen, 1898, **299**, [3], 267.

Baker has stated (this Journal, 1894, 767—768) that perfectly dry ammonia does not combine with perfectly dry hydrochloric acid gas, and also that dry ammonium chloride volatilises undissociated at 360° C. The author has repeated Baker's experiments, using apparatus which is said to have offered greater guarantees for perfect dryness, than the apparatus used by Baker, but he is unable to confirm the results of the latter. He found that the most thorough drying of the gases did not prevent their chemical union, although it rendered the reaction slower and less energetic. Baker stated that the ammonia and hydrochloric acid may be completely dried over phosphoric anhydride, if they are previously dried, the former over burnt lime or by Stas's mixture of the oxides of copper and potassium, and the latter by passing through a series of flasks containing concentrated sulphuric acid. The author found, on the contrary, that it is impossible to dry the gases unaltered, over phosphoric anhydride, as ammonia is rapidly and hydrochloric acid slowly absorbed by that substance, if the gases had been carefully dried beforehand. The absorption of hydrochloric acid takes place so slowly that it may be dried over phosphoric anhydride, but for drying the ammonia, Stas's mixture should be used.

Finally, the author found, on estimating the vapour density, and using a somewhat modified form of Victor Meyer's apparatus, with anthraquinone as the heating agent, that, in opposition to Baker's results, dry ammonium chloride actually dissociates at 280° C.—A. S.

### *Glycerin and Dioxyacetone, A New Total Synthesis of.*

O. Piloty. Ber. **30**, [19], 3161—3169.

Henry (Bull. acad. belge, **3**, [30], 25) showed how nitroisobutylglycerin, $(CH_2.OH)_3C.NO_2$, might be synthetically prepared from formaldehyde and nitromethane. Starting from this nitro compound, the author formed the hydroxylamine compound $(CH_2.OH)_3C.NH.OH$, which, by the action of mercuric oxide, he transformed into the oxime of dihydroxyacetone $[(CH_2OH)_2C:N.OH]$, and from this, by the action of bromine in aqueous solution, obtained the pure crystalline dihydroxyacetone—

$$2(CH_2.OH)_2C:N.OH + 2Br_2 + H_2O =$$
$$2(CH_2.OH)_2CO + N_2O + 4HBr.$$

Dioxyacetone has a sweet taste, is extremely soluble in cold water, readily soluble in hot alcohol, less so in hot acetone, hardly soluble in hot ether, and insoluble in petroleum spirit. When dried *in vacuo* over sulphuric acid, it melts between 68° and 75° C.—a variation which the author attributes to polymerisation changes. It forms an osazone, melting at 132° C., and reduces Fehling's solution strongly, even in the cold. It is unfermentable by beer yeast. On treating dihydroxyacetone with a solution of aluminium sulphate, cooling the liquid to zero, and adding sodium amalgam, it is reduced to glycerin.

The author regards dihydroxyacetone as the triose which, in the formation of fructose, furnishes the ketose group. He suggests that if, as is held by von Baeyer and others, formaldehyde is to be regarded as the first assimilation product when carbon dioxide is absorbed by plants, and the sugars produced by condensation of this, the formation of fructose may be explained on the assumption that in the condensation of the formaldehyde both the trioses, glyceraldehyde and dihydroxyacetones, are formed as products of intermediate stages, and that the fructose is the product of the condensation of these—

$$CH_2(OH).CH.(OH)CH.O$$

Glyceraldehyde.

$$CH_2(OH).CO.CH_2OH$$

Dihydroxyacetone.

$$= CH_2(OH).CH.(OH).CH.OH$$
$$CH.(OH).CO.CH_2.OH.$$

—C. A. M.

### *Ammonia Derivatives, Oxidation of, by Soil Ferments.*

E. Demoussy. Comptes Rend. 1898, **126**, [3], 253—256.

Methylamine is converted by soil organisms first into ammonia and then into nitrous and nitric acids. Trimethylamine is oxidised more slowly; ammonia could be detected after 13 days, but the carbon was not completely oxidised in 18 days. The slowness of the change is attributed partly to the greater complexity of the molecule, as compared with monomethylamine, and partly to its injurious effect on the microbes. As no monomethylamine could be detected, it is assumed that all three methyl groups are simultaneously attacked.

Aniline yielded ammonia in 18 days in very dilute solutions; in stronger solutions, ammonia could not be detected for two months. Pyridine was oxidised still more slowly, and it was difficult to detect ammonia owing to nitrification, although the latter was also retarded by the poisonous effect of the still untransformed base. Quinoline yielded some ammonia after four months.

Contrary to what was expected, the results indicate that even amines must be converted into ammonia before nitrification can take place. The more complex the substance, the longer the time necessary for its oxidation.—N. H. J. M.

### *Glucose, The Action of Benzhydrazide on.*

G. Pinkus. Ber. 1898, **31**, [1], 31—37.

Davidis (Ber. **29**, 2310) described a class of bodies which he called benzhydrazides, which he obtained by acting on sugars, in alkaline solution, with benzhydrazide. The author was led to re-examine this reaction and finds that the product is really a mixture of the benzylosazones of glyoxal and methyl-glyoxal. As these bodies have not been previously described, he prepared them synthetically and found them to agree in every respect with the bodies obtained from glucose. He explains the presence of glyoxal in the products of the action of alkali on glucose, as an intermediate product between glycerin-aldehyde and lactic acid, which latter is known to be a product of the reaction. In the presence of the hydrazine, the sparingly soluble compound of glyoxal, is precipitated.—A. L. S.

### *Tartrates, A New Ferment of: Bacillus Tartricus.*

L. Grimbert and L. Ficquet. J. Pharm. Chim. 1898, **7**, 97—100.

This energetic ferment of calcium tartrate is a small bacillus 1—2 μ long, with active movements capable of anaerobic or aerobic life, but more active in the latter condition. It attacks many carbohydrates—glucose, lactose, maltose, saccharose, dextrin, and mannite among others. The products of fermentation of calcium tartrate are acetic and succinic acids, hydrogen and carbon dioxide. The decomposition of ammonium tartrate produces the two acids without evolution of gas.—A. C. W.

### *Benzene, A Combination of Phosphoric Anhydride with.*

H. Giran. Comptes Rend. 1898, **126**, [8], 592—593.

The author treated benzene (3 parts) with phosphoric anhydride (2 parts) in a sealed tube at 110°—120° C. for four to five hours. A solid brick-red substance was formed, which dissociated rapidly in the air; it could, however, be preserved in an atmosphere of benzene. Water decomposed it into benzene and phosphoric acid. An alcoholic solution slowly attacks barium carbonate, a gelatinous mass being formed. The same result is obtained if, instead of barium carbonate, an alcoholic solution of barium ethylate be added until the liquid becomes neutral. This white salt, when washed with alcohol and dried over caustic potash, was soluble in water. When dried at 110°—120° C. it gave numbers on analysis agreeing with the formula $C_6H_2P_8O_{20}Ba_2$. The acid, which is called *benzenetetradimetaphosphoric acid*, results from the union of 1 molecule of benzene with 4 of phosphoric anhydride.—J. L. B.

### *Chloroform: Do Inhalations give rise to the Presence of Carbonic Oxide in the Blood?*

L. de Saint-Martin. Comptes Rend. 1898, **126**, [7], 533—536.

The author combats the statement of Degrez and Nicloux (Comptes Rend., Dec. 6, 1897) that the carbonic oxide obtained by them to the extent of 5 c.c. per litre from the blood of animals narcotised with chloroform, was due to the action of that anæsthetic. Experimenting with the blood of animals which had been treated by chloroform inhalations, and with normal blood, it was found by the author that both afford minute traces of carbonic oxide varying in extent from 0·88 to 0·2 per cent. by volume. This is not considered to be a normal constituent of the blood, but is regarded as being derived from the action of an organic acid on a normal constituent of that fluid, the liberation being analogous to that of the formation of traces of carbonic oxide observed in the determination of oxygen by means of alkaline pyrogallates. In these experiments the author determined the amount of carbonic oxide by means of the spectroscopic method, and by the direct absorption of the gas with cuprous chloride, previously described (Comptes Rend. **119**, 8).—J. O. B.

*Mercurous Iodide, Amorphous; The Colour of.* Maurice François. J. Pharm. Chim. 1897, **6**, [12], 529—533.

On precipitating mercurous nitrate with solution of potassium iodide in the presence of free nitric acid and excess of the mercurous salt, the resulting precipitate is found to be pure mercurous iodide in an amorphous condition. The colour of this is bright chrome yellow. To ensure the completion of the reaction, the precipitate is left in contact with the solution for 24 hours, in the dark, with occasional agitation; it is then collected and dried at 50° C. in the dark. Analysis shows this to be pure mercurous iodide, $Hg_2I_2$. The prevalent opinion has been that the colour of pure mercurous iodide should be yellowish-green, and the name "green iodide" is an accepted synonym for the preparation. The author shows that this is inaccurate; the colour of the pure compound being bright yellow, the green tint observed in commercial samples of the iodide, and in that prepared by the official process of the Codex, by triturating together mercury and iodine, is due to the presence of uncombined mercury. Further, on triturating a little mercury with pure yellow mercurous iodide, a greenish mixture is obtained, similar in all respects to the official article. By subjecting these green samples to the action of dilute nitric acid (20 grms. of pure acid in 80 grms. of water), they may be converted into the pure yellow iodide by the removal of the free metallic mercury.—J. O. B.

## PATENT.

*Printers' Roller Composition and the like, Impts in.* W. J. J. Hawkins, London. Eng. Pat. 1112, Jan. 19, 1897.

The inventor claims that, by the addition of powdered mica to the ordinary roller composition, a material is produced which is far less susceptible of change under varying atmospheric conditions. It is said to be practically impervious to moisture, not affected by heat, and exceedingly tough. His formula is:—Glue, $2\frac{1}{2}$ lb.; water, $1\frac{3}{4}$ lb. (about): glycerin, $6\frac{1}{8}$ lb.; sugar, $2\frac{3}{4}$ lb.; mica (finely powdered), 3 oz. The modified composition is said to be equally applicable to the manufacture of the well-known gelatinous printing surfaces.—F. H. L.

# New Books.

The Arrangement of Atoms in Space. By J. H. van 't Hoff. Second revised and enlarged Edition, with a Preface by Prof. **Johannes Wislicenus**, and an Appendix, Stereochemistry among Inorganic Substances, by Alfred Werner, Professor of Chemistry at the University of Zurich. Translated and edited by Arnold Eiloart. Longmans, Green, and Co., 39, Paternoster Row, London; also at New York and Bombay. 1898. Price 6s. 6*d.*

Small 8vo volume, containing prefaces to first and second editions, table of contents, introduction, and subject-matter filling 195 pages, pages 185 to 199 being devoted to Prof. Werner's Appendix on "Stereochemical Isomerism of Inorganic Compounds." The work closes with a copious alphabetical index.

The nature of the subject-matter and its classification may be judged of from the following summary:—Stereochemistry of Carbon. I. The Asymmetric Carbon Atom. II. Division of the Inactive Mixture. Temperature of Conversion. III. Compounds with several Asymmetric Carbon Atoms. IV. Determination of the Position of the Radicals in Stereomers. V. The Unsaturated Carbon Compounds. VI. Ring Formation. VII. Numerical Value of the Rotatory Power. Stereochemistry of Nitrogen Compounds. I. Trivalent Nitrogen. II. Compounds containing Pentavalent Nitrogen.

The work is illustrated with 19 geometric figures for graphic representation.

Handbook on the Workmen's Compensation Act, 1897. With approved Schemes of Compensation; Statutes referred to; Notes on Accidents, Negligence, and Misconduct; Employers' and Workmen's Liability; Actions Independent of the Act; Arbitration; Forms of Notices; List of Stamp Duties; Official Form of Application for a Contracting-out Certificate, &c.; together with the Principal Explanatory Remarks of the Lord Chancellor, Lord Herschell, the Home Secretary, the Colonial Secretary, the Attorney General, Mr. Asquith, and other Statesmen. By M. Roberts-Jones, Barrister-at-Law, of Gray's Inn and the South Wales Circuit, &c., &c. Western Mail, Limited, Cardiff, and 82, Fleet Street, London. 1898. Price 2s. 6*d.*

Commences with prefaces to the 1st and 5th editions, list of Statutes referred to, Table of Contents, Table of Cases, Introduction and text filling 65 pages. An Alphabetical Index ends the work. The matters treated of are as follows:—

The Workmen's Compensation Act, 1897. First Schedule. Second Schedule. Personal Injuries by Accident. Serious and Wilful Misconduct. Liability of Workmen. Actions Independent of this Act. Schemes of Compensation. Model Rules. Appendix A.—Statutes referred to in the Act. Appendix B.—Forms, Stamp Duties, &c.

Chemisch-technisches Repertorium. Uebersichtlicher Bericht über die Neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 36. Jahrgang. 1897. Zweites Halbjahr. Erste Halfte. Mit in den Text gedruckten Illustrationen. R. Gaertner's Verlagsbuchhandlung, Hermann Heyfelder, Schönebergerstrasse 26, Berlin, S.W. H. Grevel and Co., 33, King Street, Covent Garden, London.

This number, for the second half-year of 1897, and the first half thereof, treats of the following subjects of Applied Chemistry:—Building Materials, Cements, Artificial Stone. Dyestuffs, Dyeing and Calico Printing, and Coal-tar Products. Fats, Oils, Illuminating and Heating Materials. Fermented Liquors. Tanning, Leather and Glue Manufacture. Textiles. Glass and Earthenware. Wood and Horn. India-rubber and Gutta-percha. Cements and Adhesives. Lakes, Varnishes, and Paints. Metals.

Nozioni di Analisi Chimica e Cenni sull' Analisi delle Acque Potabili. Del Dott. Icilio Guareschi, Professore ordin. di Chimica Farmaceutica e Tossicologica nella R. Università di Torino. Unione Tipografico-Editrice, Turin, Via Carlo Alberto 33. 1898. Price, Lire 5.

A system of Qualitative Chemical Analysis, followed by a System of Analysis of Potable Waters. The work is divided into six principal parts, appropriated as follows:—

General Methods:—Part I. Special Reactions of the various Elementary Bodies and their Compounds. Part II. Systematic Methods. Tables and Tabulated Schemes of Analysis, I. to XVII., and A. to C. Elementary Outlines of Microchemical Analysis. Part III. Outlines of Methods for the Analysis of Potable Waters. The work is of large 8vo size, contains 169 pages of subject-matter, illustrated with 123 well-executed engravings.

NOZIONI DI ZOOCHIMICA. Del Dott. ICILIO GUARESCHI. Professore ordin. di Chimica Farm. e Tossicolog. nella R. Università di Torino. Unione Tipografico-Editrice, Turin, Via Carlo Alberto 33. 1898. Price, Lire 7.50.

A SYSTEM of the outlines of Animal Chemistry. The work, like the preceding, is of large 8vo size, and contains introduction, 258 pages of subject-matter, illustrated by 104 excellent wood engravings, and followed by a table of contents, index of illustrations, and alphabetical index of subject-matter. The text is subdivided as follows:—FIRST PART. I. CONSTITUENTS OF THE ANIMAL ORGANISM. Gases. Water. Solid Mineral Substances, and Compound Organo-mineral Substances. II. ALBUMINOIDS OR ALBUMINOUS SUBSTANCES AND THEIR IMMEDIATE DERIVATIVES. i. True Albuminoids. ii. Proteids, &c. iii. Fermentation-Albuminoids, &c., &c. III. NITROGENOUS NON-ALBUMINOID COMPOUNDS. IV. NON-NITROGENOUS SUBSTANCES. SECOND PART. ORGANS. SECRETIONS AND ANIMAL FLUIDS OF THE ORGANISM. I. Blood, &c. II. Tissues, Bones, &c. III. Glands and Digestive Fluids, &c. IV. Urine, Sedimentary Matters, and Urinary Calculi. V. Animal Concretions, &c.

CHEMICAL ANALYSIS OF OILS, FATS, AND WAXES, AND OF THE COMMERCIAL PRODUCTS DERIVED THEREFROM. Founded on Benedikt's Second Edition of "Analyse der Fette." By Dr. J. LEWKOWITSCH, F.I.C., F.C.S. Second Thoroughly Revised and Enlarged Edition. Macmillan and Co, Limited, London and New York. 1898. Price 25s.

IT is announced that the subject-matter of Chapters I. to VIII. of the first edition of this work, has been entirely re-arranged, amplified, and re-written in framing this second edition. Separate chapters have been assigned to the subjects of the Saponification of Fats and Waxes, and the Chemistry of Resin. Special attention has also been paid to Chapters IX. to XII., and nearly all the tables therein have been either re-cast or enlarged. Ample footnotes furnish references to various sources where new methods or new lines of research have appeared, likely to lead to extended knowledge, and these footnotes refer in the first instance, it is announced, to the pages of *this Journal*. The work is one of 822 pages, illustrated with 48 wood engravings, and terminating with a copious alphabetical index of subject-matter, the 1st edition containing 673 pages and 42 illustrations.

The subject-matter is subdivided as follows:—I. Physical and Chemical Properties of Fats and Waxes. II. Their Saponification. III. Constituents. IV. Determination of Foreign Matters of a Non-Fatty Nature, and Preparation of the Fatty Substance for Analysis. V. Physical Methods of Examining Fats and Waxes. VI. Chemical Methods of Examining them. (i.) Ultimate Analysis, (ii.) Qualitative Examination, and (iii.) General Methods of Quantitative Analysis. VII. Detection and Quantitative Determination of Unsaponifiable Matter in Fats and Waxes. VIII. Detection and Quantitative Determination of Resin. IX. Application of Physical and Chemical Methods to the Systematic Examination of Fatty Oils and Liquid Waxes. X. Application of Physical and Chemical Methods to the Systematic Examination of Solid Fats and Waxes. XI. Description of Natural Fats and Waxes: Methods of Examining them and Detecting Adulterations. XII. Technical and Commercial Analysis of the Raw Material and Products of the Fat and Oil Industries. XIII. Examples.

A special and valuable feature of the work is to be found in the German and French equivalents which are given of the English technical terms for the various Oils, Fats, and Waxes. Thus, the advantage of a Technical Glossary of these terms in the three languages is afforded for the reader —an advantage not to be found in the use of an ordinary dictionary.

The author announces that, in the issue of a future edition, he has under consideration the advisability of adding a further chapter, to be devoted to the subject of "Reagents," and "A Series of Tables frequently required in the Laboratory."

# Trade Report.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

### FRANCE.

*Modification of Duties on Lead, &c.*

*Bd. of Trade J., March* 1898, 313.

The *Journal Officiel* contains a Presidential Decree promulgating a law for the modification of the duties of the minimum tariff on lead, &c., as follows:—

| No. in the Tariff. | Lead, &c. | | New Tariff. | | Old Tariff. |
|---|---|---|---|---|---|
| | | | General. | Minimum. | Minimum. |
| | *Ores, Matt, and Scoria of all sorts.* | | Francs. | Francs. | Francs. |
| 222 | 30 per cent. of metal or less | Per 100 kilos. | Free | Free | Free. |
| | More than 30 per cent. of metal | ,, | 2·50 | 1·25 | |
| | In the rough, pigs, bars, or sheets. | | | | |
| | Argentiferous (containing 25 grms. and more of silver for every 100 kilos. of lead). | ,, | 3·00 | 2·50 | |
| | Non-argentiferous (containing less than 25 grms. of silver per 100 kilos. of lead):— | | | | |
| | (*a.*) From countries where argentiferous leads are free of export duty. | ,, | 4·00 | 3·50 | |
| | (*b.*) From other countries | ,, | 4·00 | 3·50<br>In addition to the amount of export duty paid in the country of origin. | |
| | Alloys of antimony in the rough | ,, | 7·00 | 6·50 | 3·00 |
| | Beaten or rolled | ,, | 7·00 | 6·50 | 3·00 |
| | Filings and old scrap | ,, | 4·00 | 3·50 | Free. |
| 239 | Minium | | 8·50 | 5·15 | 0·00 |
| | Litharge and other oxides | | 8·75 | 5·30 | 0·00 |
| 225 *bis* | Salts of lead, and chemical products of a lead base, not distinguished. | | 5 % *ad val.* plus 2·40 frs. per 100 kilos. | 5 % *ad val.* plus 2 frs. per 100 kilos. | Not specified. |
| 256 | Acetate of lead | | 11·50 | 7·00 | 5·50 |
| 262 | Carbonate of lead | | 8·50 | 5·00 | 2·00 |
| 266 | Chromate of lead | | 25·00 | 18·50 | 16·00 |
| 271 *bis* | Pyrolignite of lead | | 9·50 | 5·10 | 3·00 |
| 306 | Green colours from the mixture of chromate of lead and Prussian blue. | | 8·00 | 7·00 | 5·00 |
| 308 *bis* | Carbonate of lead, which has undergone a preparation similar to colours mixed with oil. | | 11·50 | 9·00 | 6·00 |
| 576 | Piping and other manufactures of lead of all sorts | | 8·00 | 6·50 | 3·00 |
| 576 *bis* | Electric accumulators | | 21·00 | 16·50 | 13·00 |

### SWITZERLAND.

#### *Tariff Decisions.*

A notice has been recently issued by the Swiss Customs Department with regard to the classification of certain articles in the Swiss Customs Tariff.

| No. | Article. | Duty. |
|---|---|---|
| | | Francs. |
| 11 | Orrisroot | 3·00 |
| 12 | Do. | 8·00 |
| 15 | Aldehyde | 8·00 |
| 45 | Manganese in paste | 0·40 |
| 88 | Mastic of "Kalkolithe" | 0·60 |
| 209 | Photographic negatives | 30·00 |
| 250 | Apparatus for the production of acetylene | 4·00 |

### QUICKSILVER FOR THE CAPE.

*Chem. and Druggist, March* 19, 1898, 460.

From March 1 the Cape import duty upon quicksilver is reduced from 5 per cent. to 3 per cent., so as to bring the Customs dues on this commodity into line with those of Delagoa Bay. It is thus classed with cyanide of potassium.

## *POST OFFICE NOTICES.*

### TRANSMISSION OF LIQUIDS, GLASS, &c., BY POST.

*Bd. of Trade J., March* 1898, 365.

Articles of the undermentioned classes are now transmissible by letter post within the United Kingdom, under the conditions given below :—

| | |
|---|---|
| GLASS. | LIQUIDS. |
| GREASE. | COLOURING POWDERS. |

The conditions are :—

1. Nothing inflammable, corrosive, or explosive may be sent, nor anything having an odour so strong as to effect the contents of other packets in the mails.

2. No article liable to Customs duty may be sent by letter post to or from the Channel Islands or the Isle of Man.

3. The following regulations as to packing must be complied with :—

*Glass* must be securely packed (in boxes of metal, wood, leather, or cardboard) in such a way as to prevent all risk of injury to the mails or the officers of the post office.

*Liquids* must be enclosed in glass bottles, firmly sealed ; the bottles must be enclosed in a box of wood, leather or metal, and packed securely in a box by means of corrugated paper or other suitable material, a sufficient quantity of sawdust or some spongy substance being added to absorb all the liquid contents in case of the bottle being broken. Special attention must be paid to the packing round the shoulders of the bottle ; and the box must open at one end only, and have a tightly-fitting lid or a screw lid if made of metal, and if made of wood a screw lid with india-rubber washer round the neck.

*Semi-liquids* and *Greases* must have an inner covering securely closed, and an outer covering of metal, wood, or leather sufficiently strong to prevent any risk of the contents escaping during transit.

*Powders* must have an inner covering securely closed, and a strong outer covering of metal, wood, leather, or cardboard.

4. Packets of all the classes named, are admitted to the letter mails at the sender's risk, the Postmaster General accepting no liability in the case of either loss or damage, even if the packets are registered.

5. Packets of the classes named which are found to be insecurely packed will be stopped. The public are warned that such insecurely packed packets come within the category of articles "likely to injure other postal packets in course of conveyance or an officer of the Post Office." Such articles are prohibited from being sent by post, and the senders thereof are liable to penalties under the Post Office (Protection) Act, 1884. These provisions will apply to insecurely packed packets of glass, liquids, grease, and colouring powders.

The public are asked to note that, although articles of the classes named are admitted to the Inland Letter Post, they can in all cases be sent more safely by Parcel Post.

If such articles are sent by Letter Post to or from places marked "a" in the List of Provincial Post Offices in the Post Office Guide, they should be registered and be marked "Fragile, with care," in order that they may be withheld from transmission in mails transferred to or from trains in motion.

All the articles mentioned above will also be transmissible by sample post under special conditions between the United Kingdom and other countries where they are admitted, provided that the packets also conform to the general regulations of the sample post, as set forth in the Post Office Guide. They must be so made up that they can be easily opened for purposes of inspection. In other respects the conditions as to packing, &c., will be those applicable to the articles concerned when intended for inland transmission.

Packets from abroad containing liquids liable to customs duty on entry into the United Kingdom will be stopped and returned to the country of origin.

### GLASS SAMPLES FROM THE UNITED STATES.

*Chem. and Druggist, March* 19, 1898, 460.

The U.S.A. Post Office Department has announced that after March 1, samples of glasses, powders, liquids, and other substances of a similar character will be admitted to the mails to Great Britain.

## *DIPLOMATIC AND CONSULAR REPORTS.*

### MANURES, SULPHUR, KAOLIN, AND SILICA IN ITALY.

*Foreign Office Annual Series*, 1898, *No.* 2033, *p.* 23.

The use of artificial fertilisers is spreading conspicuously in Italy, and the works of the German firm of Walter and Co. are annually turning out more and more of their chemical manures, which they suit to the soil upon which they are to be used by analysing its constituents beforehand. Among their other publications is a popular catechism on the use of chemical manure, worded in the simplest possible style, so that even a peasant proprietor can understand it and gain information from it The idea is a commendable one, and may be useful to our manufacturers at home. Most works on these subjects are so elaborate that it is only technical students who can follow them, and this checks the use of chemical manures among small owners and occupiers. The chemical fertilisers are much cheaper to spread than farmyard manure, and if 25 kilos. of the former are equal in fertilising power to 1 ton of farmyard manure, and only cost 11 lire 20 c., the gain in their adoption must be considerable. The price of a load of manure is about the same, but it is doubtful if an Italian cart carries as much as a ton.

The following gives an idea of the annual output of the firm :—Sulphuric acid, 6 tons per diem, about 2,000 tons per year ; muriatic acid, 350 tons ; sulphate of soda, 400 tons ; sulphate of iron, 500 tons ; nitric acid, 50 tons ; superphosphate of Algerian phosphates, 500 tons ; chlorate of potash (imported), 50 tons ; nitrate of soda (imported), 100 tons ; sulphate of copper (imported), 150 tons ; sulphate of ammonia, 100 tons ; sulphur, winnowed for vineyards, 500 tons. The firm finds sulphate of copper cheaper to buy than to make, and imports it in increasing quantities.

As is well known, powdered sulphur is blown on to the vines when they are flowering to keep off the disease. This is done by bellows which are filled with the powder which is then applied to the leaves. This plan obviously causes considerable waste, as a cloud of sulphur is blown into the air, and something like 50 per cent. of it falls upon the ground, where, of course, it does no good. It occurred to the inventor that the finer the sulphur the less would be the waste, and he accordingly invented a machine by which the sulphur should pass through a swift current of air which would blow all the finest of it into a receptacle, and allow the coarser matter to run off into a mill which grinds it again and brings it round automatically to the fan. The results have proved very satisfactory.

Hitherto the chief market for silica has been at Fontainebleau, and for china clay at La Lissolle, in France, where the product fetches 25 fr. per ton, considerable expense being incurred in separating the silica from the kaolin or silicate of clay. The same firm has discovered a vast deposit of pure silica in Calabria, and has caused a shipload of 160 tons to be brought to the works. In another place near it has found a deposit of kaolin, but it is to the pure silica that it attaches the greatest importance.

Both articles have to undergo a process of cleaning and separation from impure material. By a simple washing apparatus Dr. Walter separates the silica from the kaolin. In order to show that both materials rival the finest silica of Fontainebleau and the best china clay of La Lissolle, some samples have been sent to the Association of Chambers of Commerce. The present cost of raising 1 ton of raw material and conveying it to the coast is 15 fr. By means of a short wire-rope line the cost will be reduced to 2 fr. One ton of raw material gives 700 to 800 kilos. of silica, and from 200 to 300 kilos. of kaolin. One ton of silica is worth 25 fr. f.o.b. in Italy, and 1 ton kaolin is worth 50 fr.; so that a ton of raw material costing 15 fr. will produce about 30 fr.; and when the transport is perfected, and the raw material costs but 2 fr., the profit will be largely increased. There is practically no limit to the quantity obtainable, and as Italy alone consumes 34,000 tons of silica annually there is an abundant local market, besides what may be done in exports. The separating machine will be in full work before the end of February.

### IMPORT TRADE OF SERVIA FOR 1896—1897.

*Foreign Office Annual Series*, 1898, *No.* 2029.

Rock salt, mostly from Roumania, was valued at 36,651*l.*, and coal and lignite (chiefly Austro-Hungarian) at 12,614*l.* Other small importations were bricks, tiles, and earthenware, 4,346*l.*; porcelain, 2,464*l.*; and window glass, 6,080*l.* English glass was estimated at 300*l.* only.

There was an importation under the head of chemicals, &c., in 1896, amounting to 52,197*l.*, which was about 2,000*l.* higher than the figures for 1895. The principal distribution of this trade was Austria-Hungary, 33,277*l.*: and Germany, 11,647*l.* British trade consisted chiefly of soda, ammonia, and varnish, valued altogether at 2,237*l.* Chemicals and drugs of various sorts came to 10,226*l.*, a falling-off of 4,355, and made-up medicines and chemical preparations to 12,468*l.* Dyes were valued at 15,542*l.*, of which 5,980*l.* represented raw colours. For the first nine months of 1897 the importation of drugs, chemicals, and colours was valued at 39,575*l.*

Articles under the head of fatty produce were imported in 1896 to the value of 67,987*l.*, a decrease over the preceding year's figures of 30,466*l.*, this decrease being accounted for by a falling-off of no less than 37,505*l.* in the purchase of petroleum. The total importation of mineral and vegetable oils in 1896 amounted to 51,906*l.*, and included a sum of 10,548*l.* for British cotton-seed oil. Soap was valued at 7,663*l.*, mostly common unscented, and candles at 5,815*l.*

Under the head of fatty produce, Austria-Hungary furnished in 1896 to the amount of 29,313*l.*; Russia, 14,723*l.*; and Great Britain, 11,693*l.* During the first nine months of 1897 the value of this importation was 35,712*l.*

### NEW PETROLEUM WELLS IN TEXAS.

*Bd. of Trade J., March* 1898, 340.

A report, dated 28th January last, has been received at the Foreign Office from Her Majesty's Consul at Galveston, stating that a company has been organised for the purpose of working and putting upon the market the oil recently discovered at Sour Lake, Texas.

The wells are already producing freely, what is said to be a fine quality of lubricating oil, and a refinery is about to be built.

It is stated that the Sour Lake fields give every indication of being remarkably fine. They are situated near the sea coast, like the oil wells of Russia, and, like these latter, the oil comes from a bed of sand, and not from rock, like the Pennsylvanian oil.

The oil is said to be heavier than any found in America, and refines with but little waste.

### FERTILISER TRADE OF CHARLESTON, U.S.A.

*Foreign Office Annual Series*, 1898, *No.* 2031.

During the past commercial year the entire southern country has suffered from general trade depression, which has naturally affected the fertiliser industry.

Shipments of fertilisers from Charleston last year were 374,494 tons, compared with 257,715 tons during the previous season of 1895-96; being a gain of nearly 50 per cent. The total output of the mills is estimated to have been nearly 400,000 tons, some of the companies manufacturing from 20,000 to 80,000 tons yearly, and using in the process such materials as sulphur, kainit, blood, tankage, cotton-seed meal, potash, and phosphate rock, all of which are cash articles.

It is expected that the State of Georgia alone will use this season 400,000 tons of commercial fertilisers—more than the quantity consumed by any other two States combined, much of which will be supplied by Charleston mills. North Carolina's consumption closely approximates 200,000 tons, the result being that nearly the whole of last year's South Carolina product of 400,000 tons was sold when the new season opened on September 1, 1897.

The following comparative statement of shipments of fertilisers from the three principal ports in this Consular district of Charleston, Savannah, and Port Royal, will show the relative importance of each, and also give some idea of the total business :—

*Shipments and Sales of Fertilisers for the last Two Years.*

| From | Quantity. | |
|---|---|---|
| | 1896. | 1897. |
| | Tons. | Tons. |
| Charleston | 257,715 | 374,494 |
| Savannah | 90,000 | 120,000 |
| Port Royal | 35,000 | 35,000 |
| Total | 382,715 | 529,494 |

The imports of chemicals used in the manufacture of fertilisers at Charleston during the year ending August 31, 1897, were 107,828 tons, valued at 810,941 dols., compared with 58,938 tons, imported the previous year, with a valuation of 433,400 dols.; showing an increase last season of 48,890 tons, of 377,541 dols. value. The principal chemical imports consisted of :—

| — | Quantity. |
|---|---|
| | Tons. |
| Kainit | 42,349 |
| Brimstone | 8,900 |
| Muriate of potash | 5,289 |
| Sulphur pyrites | 20,570 |
| Nitrate of soda | 1,095 |
| Manure salt | 718 |
| Sulphate of potash | 121 |
| Sulphur ore | 28,786 |

In addition to the above imports of chemicals at Charleston, there were also imported to Savannah 120,000 tons, and to Port Royal 35,000 tons during the season.

Of the above-mentioned imports, the bulk of the pyrites and sulphur ore came from Spain and Portugal; the brimstone from Italy; the kainit, potash, and manure salt from

Germany; and the nitrate from the nitrate beds of Chili; little or nothing now being imported direct from Great Britain.

The shipments of phosphate rock from the port of Charleston during the year ending September 1, 1897, were only 2,240 tons to foreign countries, but to ports and places in the United States they amounted to 126,779 tons. Shipments from Beaufort were 82,376 tons foreign, and 5,044 to places and ports in the United States.

The total amount of phosphate mined in this country last year was 993,507 tons, of which South Carolina supplied 445,017 tons; Florida, 516,160 tons; Tennessee, 25,000 tons; and North Carolina, 7,000 tons. The total valuation of this whole product at present prices is estimated at 3,600,000 dols., equivalent in sterling to 720,000*l*.

The phosphate rock which has been discovered in Tennessee has within the past three or four years come forward as a strong competitor with the Carolina and Florida product. It, however, varies greatly from the Carolina and Florida article. Tennessee rock is darker in complexion and denser; it does not require washing and drying, and as a general rule is of good grade, but its analysis does not run uniformly, often differing considerably in the same deposits. After it is crushed it grinds easily and also slacks up in drying.

Many of the deposits are situated, however, in places more or less remote from railway and steamboat transportation facilities, this being particularly the case with the early discoveries, but the newer discovered fields are all very near the railways.

Florida is by far the most important and dangerous rival that South Carolina has to contend with in the phosphate business; varied and valuable deposits have been discovered and are now worked in that State, each good of its kind, and in qualities are as follows: hard and soft rock, land and river pebble, and river rock.

Most of the Florida rock is shipped to foreign countries; as yet she does not manufacture fertilisers, and consequently has not, in this respect, come into competition with Charleston's large fertiliser industries; but owing to the cheapness and good quality of the rock it is expected that, in time, numerous fertiliser-manufacturing establishments are sure to be established in the State, which will then be able to compete with South Carolina's fertiliser business as it does now in the phosphate rock trade.

The harmful effect of the phosphates of Algiers and Tunis on the South Carolina industry have not been so apparent during the past year as the competition nearer home from Florida and Tennessee; although the unfavourable influence has been material in affecting prices in foreign markets for American shipments with which the African mines compete. From the latest reports received, it appears that those mines have made but slow progress during the last year, and have not kept pace with the development of similar new industries in this country. There seems to have been a want of harmony among the companies engaged in the business, and political interference, from which they have also suffered, has tended to restrict and hamper the successful prosecution of the work. Notwithstanding all drawbacks, however, it is claimed that the Algerian and Tunis mines turn out 300 tons of phosphate rock daily, and that the output last year was 142,340 tons, which it is expected will be increased next season to 175,000 tons.

Most of last year's output was shipped to the following countries — England, France, Italy, Germany, Austria, Japan, Belgium, and Spain; quantities shipped being in the order named, and varying from 54,005 tons sent to England to the smallest shipment, which was 250 tons taken by Spain. The market for phosphate rock at the end of the year closed firmer with quotations for crude of 3 dols. at mines, per ton; hot air dried, 3 dols. 25 cts. per ton, f.o.b., vessels in Ashley River; 3 dols. 15 cts. per ton, f.o.b., City. Ground rock in bulk, 5 dols. 50 cts. per ton, f.o.b., vessel at City. The total exports of phosphate rock from this port from September 1 to December 31, 1897, were 29,950 tons, compared with 32,901 tons for the previous year, of which none went abroad.

## GENERAL TRADE NOTES.

### THE MINERAL PRODUCTION OF CANADA IN 1897.

*Eng. and Mining J.*, *March* 5, 1898, 278.

We are indebted to Mr. Elfric Drew Ingall, head of the Section of Mineral Statistics of the Geological Survey of Canada, for an advance copy of the statistics of the Dominion for 1897. The figures are given in the following table:—

*Mineral Production of Canada in* 1897.

| Product. | | Quantity. (b) | Value. (a) |
|---|---|---|---|
| | | | Dols. |
| METALLIC. | | | |
| Copper (fine, in ore, &c.) | Lb. | 13,300,802 | 1,501,660 |
| Gold | .. | .. | 6,190,000 |
| Iron ore | Tons | 71,451 | 178,716 |
| Lead (fine, in ore, &c.) (c) | Lb. | 39,018,219 | 1,396,853 |
| Mercury | „ | 688 | 324 |
| Nickel (fine, in ore, &c.) (d) | „ | 3,997,647 | 1,399,176 |
| Platinum | .. | .. | 6,600 |
| Silver (fine, in ore, &c.) (e) | Oz. | 5,558,416 | 3,322,905 |
| Total metallic | | .. | 13,996,234 |
| NON-METALLIC. | | | |
| Asbestos and asbestic | Tons | 25,262 | 324,700 |
| Chromite | „ | 2,637 | 32,474 |
| Coal | „ | 3,876,201 | 7,286,257 |
| Coke (f) | „ | 78,811 | 209,920 |
| Feldspar | „ | 1,275 | 3,506 |
| Fireclay | „ | 1,923 | 3,779 |
| *Grindstones | „ | .. | 40,000 |
| Gypsum | „ | 239,691 | 244,531 |
| *Limestone for flux | „ | .. | 40,000 |
| *Mica | .. | .. | 75,000 |
| Mineral pigments:— | | | |
| Baryta | Tons | 571 | 3,060 |
| Ochres | „ | 3,905 | 23,560 |
| *Mineral water | Galls. | .. | 140,000 |
| Moulding sand | Tons | 5,485 | 10,931 |
| Natural gas (g) | .. | .. | 325,873 |
| Petroleum (h) | Bbls. | 709,857 | 1,011,546 |
| Phosphate (apatite) | Tons | 908 | 3,984 |
| Pyrites | „ | 38,910 | 116,799 |
| *Salt | „ | .. | 180,000 |
| Sundry minerals, partly estimated, including actinolite, graphite, manganese, soapstone, and tripolite. | | .. | 10,000 |
| Total non-metallic | | .. | 10,097,831 |
| STRUCTURAL MATERIALS AND CLAY PRODUCTS. | | | |
| Cement, natural | Bbls. | 85,450 | 65,893 |
| „ Portland | „ | 119,763 | 209,380 |
| Flagstones | | .. | 7,190 |
| *Granite | | .. | 75,000 |
| *Pottery | | .. | 125,000 |
| Sewer pipe | | .. | 161,250 |
| Slate | | .. | 42,800 |
| Terra-cotta | | .. | 155,395 |
| Building material, including bricks, building stone, lime, sands and gravels, and tiles (estimated as for previous year). | | .. | 3,600,000 |
| Total structural materials and clay products. | | .. | 4,445,108 |
| All other non-metallic | | .. | 10,097,831 |
| Total non-metallic | | .. | 14,542,939 |
| Total metallic | | .. | 13,996,234 |
| Estimated value of mineral products not returned. | | .. | 250,000 |
| Total, 1897 | | .. | 28,789,173 |

* Partly estimated.

(a) Quantity or value of product marketed. The ton used is that of 2,000 lb.

(b) Copper contents of ore, matte, &c., at 11·29 c. per lb.

(c) Lead contents of ores, &c., at 3·58 c. per lb.

(d) Nickel contents of ore, matte, &c., at 35 c. per lb.

(e) Silver contents of ore at 59·79 c. per oz.

(f) Oven coke, all the production of Nova Scotia and British Columbia.

(g) Gross return from sale of gas.

(h) Calculated from inspection returns at 100 galls. crude to 42 refined oil, and computed at 1·42½ dols. per bbl. of 35 imp. galls. The barrel of refined oil is assumed to contain 42 imp. galls.

The total value of the mineral products of the Dominion for 12 years past has been as follows :—

| | Dols. | | Dols. |
|---|---|---|---|
| 1897 | 28,789,183 | 1891 | 18,976,616 |
| 1896 | 22,609,825 | 1890 | 16,763,353 |
| 1895 | 20 715,319 | 1889 | 14,013,913 |
| 1894 | 19,933,857 | 1888 | 12,479,550 |
| 1893 | 20,035,082 | 1887 | 11,365,705 |
| 1892 | 1,6628,417 | 1886 | 10,221,255 |

The increase in the items silver and lead in 1897 is practically all to be credited to British Columbia, to which province is also largely due the increase in the production of copper. The largest increase in this latter metal is, however, due to the increased shipments of nickel-copper mattes from the Sudbury mines in Ontario. The third copper-producing province, Quebec, also contributed a fair amount to the increase shown. The silver, copper, and lead increases, as far as British Columbia is concerned, represent the largely extended activities in the South Kootenay districts of Slocan, Nelson, and Trail Creek.

The most significant item, however, is to be found in the increase of nearly 123 per cent. in the gold. All the gold-producing districts of the Dominion show gratifying increases, although by far the largest part is due to the discoveries of rich placer mines in the Yukon country, and to the increased output of Trail Creek and other districts in British Columbia. The former increase amounted to about 2,250,000 dols., and the latter to nearly 1,000,000 dols.

The remaining metal of importance is nickel, which is seen to have shown an increase of nearly 18 per cent. As in the past, this is all from the Sudbury district.

Thus, the aggregate result of the increases in the metallic products is nearly 6,000,000 dols., or a proportion of about 74 per cent.

An interesting result of the mining developments and discovery of 1897 is shown in the increase in the proportion of the value of the total mineral production to be credited to the metals. In 1896 these constituted about 36 per cent., whilst in 1897 this proportion was increased to nearly 49 per cent.

Turning to the non-metallic products, we find that, as far as the data at present available permit of a conclusion being arrived at, the total value shows under 2 per cent. increase over that of the previous year. The only considerable change to be noted is that given under the heading asbestos, where the quantity has more than doubled, whilst the value has fallen off almost 25 per cent. This is due to the fact that the output of the new by-product, asbestic, is included. This constitutes over half the weight of the whole, but, being a low-priced article, brings down the price per unit very much. The returns, however, show also a falling off in the prices of the usual grades of the mineral apart from the above influence.

In coal, the variation is but little. The falling off in the production of the higher-priced mineral of British Columbia and the increase in that of Nova Scotia at a lower price, causes a fall in the total value. In gypsum, the increase in the value is larger than that shown in the quantity. This is due to the larger proportion of the higher-priced product, plaster of Paris, included in the returns for 1897. The lower average price ruling during that year for petroleum caused the decrease in the value of this article to be much greater than that in the quantity.

### INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

The third meeting of this Congress will be held in Vienna in July next. The work is divided into 10 sections, as follows :—1. General analytical chemistry. 2. Chemistry of foods and medicines. 3. Agricultural chemistry. 4. Chemistry of agricultural products. 5. Chemistry of wines. 6. Chemical industries based on inorganic materials. 7. Metallurgical chemistry, including also mines and explosives. 8. Chemical industries based on organic materials. 9. Chemistry of the graphic industries. 10. Questions of research and of general interest. One of the chief questions before the Congress will be the adoption of uniform methods of analysis for commercial products and raw materials.

### BEETROOT CULTIVATION IN THE UNITED STATES.

*Bd. of Trade J., March* 1898, 348.

The United States Department of Agriculture has recently issued a report, from which it appears that the production of sugar, which only amounted to 800 tons in 1886, has steadily increased since that period, and exceeded 40,000 tons in 1896. There are eight factories in the country, which are well supplied with all the necessary plant, and it is owing to the successive and rapid establishment of these factories that the national production rose from 2,800 tons in 1890 to 20,453 tons in 1893, and that it shows since 1894 an annual progression of 10,000 tons. If a comparison be drawn between the production of sugar and the consumption during the last 10 years, a still more marked advance is evident. The consumption, which was 1,355,809 tons in 1886, gradually increased until it amounted to 1,960,086 tons in 1896—an increase of about 50 per cent., while the population has only increased by 20 per cent. at the most during the same period.

### ALKALI MANUFACTURE IN MICHIGAN.

*Chem. Trade J., March* 12, 1898, 187.

The manufacture of alkalis in the Detroit district, Michigan, U.S.A., is growing very rapidly, and the Pennsylvania Salt Company have bought land down the river and intend going into the manufacture of soda ash, caustic, and bicarbonate of soda by the ammonia-soda process. According to the *Oil, Paint, and Drug Reporter*, this will be another attack on the trade of the oldest company in the United States, and the views of a prominent manufacturer down the river seem to indicate that there cannot possibly be any rise in prices until a combination takes place. The corporation which commenced the manufacture of alkali on the Detroit river has been allowed to extend and increase its works so rapidly that it can now demand a combination to put prices on a remunerative basis. Its output has crept up since 1894 from 60 tons to 200 tons per day, and it is reported that there will be a still further increase in the output. The bicarbonate plant is now producing largely, and the caustic plant is running full time and turning out a caustic of very fine grade. In the near future we can look for a drop in the price of caustic, and then a combination is almost assured.

The United Alkali Company, Ltd., has contracted for its wells, the contract having been made with T. Percy, of Ludington, who drilled the first wells for the ammonia-soda trade down the river.

A sample of iron pyrites running 38 per cent. sulphur has come to hand, and in the near future, if prospects materialise in finding even better than this, sulphuric acid will be made in Michigan. Strontium sulphate has also been found in abundance, and a market for this will be found in the manufacture of strontium. Calcium carbonate searches have been made, and an 88 per cent. oxide has so far been made. This will assist in the manufacture of caustic soda, and as the carbonate enters largely into the manufacture of beet-sugar, factories for which are now being exploited, a large demand may be expected.

The Joy Morton Salt Works are making progress, and Wayne county has now reached the highest point in its history for the manufacture of salt. It will soon surpass all other counties in Michigan.

The Detroit Soap Works are now increasing their plant and are putting in a plant for the manufacture of caustic soda and the recovery of glycerin.

The Solvay Works are not running full, having only placed about one-half their output. They have contracted to supply Delray with electric light and are assisting toward a fire department for the newly incorporated village. Reports say that the output of limestone from the Sibley limestone quarry has now reached 1,000 tons per day, the Wyandotte Works taking 500 tons per day. There is a good field open for other quarries in this vicinity, but none have branched out.

The new Detroit and Lima R. R. will prove very beneficial to alkali works along the Detroit river, opening up fresh fields for fuel and general supplies.

### NATIVE SODIUM CARBONATE.

*W. P. Blake. Eng. and Mining J., Feb.* 12, 1898, 188.

The Arizona School of Mines has recently completed an investigation of the "trona," or crude carbonate of soda, brought in from a deposit near the head of the Gulf of California. It is known as the Santa Helena Soda Deposit, and has been taken up under the mining laws of Mexico by Messrs. Andres and Joseph Rebeil, of Tucson, Arizona. The location is in the northernmost portion of the State of Sonora, Mexico, in the mining district of Altar, about 2½ miles from Adair Bay, and directly upon the shore of the Gulf. It is thus not far from the international boundary line, and is most directly accessible by water from Yuma or the Colorado, or overland from Gila Bend station on the Southern Pacific Railroad. The shores of the eastern side of the Gulf at the place are generally low and shelving, and broad areas are left bare at low tide, but at this particular point it is claimed that the water is deep enough for vessels of moderate draft to lie safely at anchor, where they can take in cargoes of soda from lighters.

The soda is found in the central portion of a basin-shaped depression, described as a dry lake, about one mile in length and breadth. The soda is spread out in a white sheet or layer over an area of some 60 acres, and resembles a mass of snow and ice, or the surface of a frozen lake. The few tests that have been made by digging and boring down into this deposit show that it has a thickness varying from 1 to 3 ft., partly overlaid by a stratum of sandy earth some 3 ins. thick. The lowest portions of the soda bed are saturated with water, or rather with a solution of soda in water, and this seems to underlie the bed for a depth of 2 ft. The solution is so strong that, when exposed to the air and sun, soda rapidly crystallises out and forms in layers to a depth of nearly 3 ins. in less than 30 days.

The valley appears to have several permanent springs of water of small volume, but in the very centre of the deposit there is a spring, or water-hole, where potable water can be had in quantity. No investigation has yet been made of this spring water, which may on analysis be found to contain enough soda in solution to give origin to the great deposit by long continued evaporation. The existence of water fit to drink is a most important advantage in that arid region.

The purity of the sodium carbonate of this deposit is remarkable. It is brought in from there in large mains almost free from sediment or earthy impurities. It may be obtained in clean crystals and crusts by solar evaporation of the concentrated or saturated water from the bottom of the bed, but in digging in the large way, there appears to be a notable mixture of insoluble clay and very fine sand, an earthy impurity which probably has been blown in upon the soda lake by the winds from the surrounding hills of Colorado river silt, as shown by the analysis of the average sample:—

*Composition of the Average Sample.*

| | Per Cent. |
|---|---|
| Sand, silt, &c., insoluble in water .......... | 13·00 |
| Iron oxide and alumina..................... | 2·80 |
| Lime ............................................ | 1·14 |
| Salt.............................................. | 4·70 |
| Sulphate of soda .............................. | 1·70 |
| Carbonate of soda............................ | 75·86 |
| Total........................... | 100·00 |

One avoirdupois pound of this sample leached with cold water gave 75 per cent. of clean white sodium carbonate, including the small quantity of other soluble salts. Hot water gives a larger product. There appears to be an increase of weight by absorption of water. The weight and bulk of the product by lixiviation is much reduced by ignition, thus driving off a considerable amount of water. It would appear to be desirable to dissolve the trona and to filter or decant to get rid of the insoluble impurities, but this would perhaps remove the product from the free list and subject it to a considerable import duty in the ports of the United States.

Another sample of selected crystals was analysed by Prof. Robert H. Forbes and Mr. M. H Walker, in the chemical laboratory of the agricultural experiment station of the University. They found the specific gravity to be 2·33. The qualitative examination showed, in addition to the carbonate of soda, a trace of chlorine and sulphates, no sulphides and no lime, barium, or silica, no potassium or ammonia. Some of the sodium was in the form of bicarbonate. The equivalent of 71·96 per cent. of $Na_2Co_3$ was found by titration with standard $H_2SO_4$.

The owners estimate that this deposit will yield 1,000 lb. of soda to each square yard of area, and that the soda can be dug and put on ship board for about 2 dols. per ton. In one test it was found that one man could throw up four tons of the soda in one day.

The height of the deposit above mean tide is about 27 ft. The plan is to construct a tramway to the water of an inlet navigable for lighters drawing less than 6 ft. of water, by which the mineral can be transferred in bulk to a vessel lying in deep water.

It is proposed to ship this trona to San Francisco, where an important use for it will be found in making borax from the crude tincal, or boracic acid, of Nevada. It has been suggested, also, that the borate of lime—Colemanite—of Nevada might be brought to Yuma, and there meet the carbonate of soda and produce borax. There is a scarcity of fuel at this locality, but for purposes of evaporation the ardent rays of the sun and the dry air are satisfactory substitutes.

### HINTS TO TRADERS WITH CENTRAL AFRICA.

*Bd. of Trade J., March* 1898, 267.

The following information relative to the trade of Uganda and neighbouring districts has been received from a well-informed source:—

It appears that certain cotton cloth sent from this country to Central Africa is so covered with size or some other preparation that the texture is subject to rapid deterioration. It is, moreover, not so durable as the American article, and is dearer and heavier, the latter a serious consideration where goods have to be conveyed by the most expensive of all kinds of transport, *viz.*, on men's heads. It is contended that English firms are unable to produce the article in demand, but the Americans appear to be able to do so.

It is not possible to state the annual value of this trade, but it must be very great and growing daily with the march of civilisation and the consequently increased desire for clothing. Another point which should not be lost sight of by shippers to Central Africa is the operation of a special style of packing. As an example, it has been stated that in the colony of Victoria the trade in tacks has been lost to British firms because they failed to pack in cardboard boxes instead of in paper parcels; the same trade has probably suffered in other places from the very same cause.

It should be impressed on the British manufacturer that everything should be packed, as far as possible, so that the cases can be made use of after the contents have been used. As an example, tea may be packed in compact little tins with close fitting lids, in which botanical specimens or anything else might be afterwards kept without fear of the damp. The firm that starts packing jam, butter, flour, &c. in similar tins will be amply rewarded for any expense they may incur. It may be added that jars or tins with screw lids like those which prune jars have, are most convenient.

It should be noticed that close packing, so that space is not uselessly taken up by quantities of straw, is very important, as this means useless payment of freight.

### THE MINERAL PRODUCTION OF SPAIN IN 1897.

*Eng. and Mining J., March* 12, 1898, 313.

From the advance statement prepared by Senor Roman Oriol, and published in the *Revista Minera*, we obtain the following interesting figures in relation to the mineral production of Spain for the year 1897:—

*Copper.*—The production of copper was stimulated by the demand for the metal and the high prices. Nearly all the important companies maintained their production, only one—the Compania de Sotiel Coronada—reporting a considerable decrease, its output having been 68,725 metric tons of ore and 1,425 tons of precipitate, against 103,311 tons ore and 2,467 tons precipitate in 1896. The Rio Tinto Company gives the following statement of the output of its mines, in metric tons:—

| — | 1896. | 1897. |
|---|---|---|
| Copper ore | 1,437,332 | 1,388,392 |
| „ precipitate | 19,912 | 20,503 |
| „ matte | 16,378 | 16,159 |
| Sulphate of copper | 4,192 | 3,100 |
| Silver-lead ore | 714 | 1,466 |
| Iron ore | 22,426 | 28,631 |

The exports, as compared with 1896, were 822,570 tons of copper ore, an increase of 193,030 tons; 36,055 tons of precipitate, an increase of 2,061 tons; and 15,432 tons of matte, an increase of 3,391 tons.

*Iron Pyrites.*—The exports were 217,545 tons in 1897, a decrease of 182,943 tons. The difference was largely due to the smaller production of the Aguas Teñidas Company, which mined only 155,000 tons in 1897, a decrease of 50,000 tons.

*Lead and Silver.*—The production of lead showed a material increase. That of silver also showed a gain, as was to be expected, nearly all the Spanish silver being obtained from lead ores. The output by provinces was as follows, lead in metric tons and silver in kilos.:—

| Province. | Lead. | | Silver. | |
|---|---|---|---|---|
| | 1896. | 1897. | 1896. | 1897. |
| | Tons. | Tons. | Kilos. | Kilos. |
| Murcia | 81,922 | 90,000 | 15,852 | 70,000 |
| Jaen (Linares) | 36,169 | 40,000 | 5,030 | 10,500 |
| Cordoba | 32,971 | 30,000 | 39,700 | 45,500 |
| Almeria | 11,658 | 11,000 | .. | .. |
| Guipuzcoa | 4,297 | 5,000 | 3,208 | 4,000 |
| Guadalajara | .. | .. | 764 | 1,000 |
| Totals | 167,017 | 176,000 | 64,554 | 131,000 |

The exports of lead were 171,774 tons, an increase of 3,189 tons; of lead ore, 8,267 tons, an increase of 1,982 tons.

*Manganese Ore.*—The exports of manganese ore were 95,756 tons, a gain of 7,889 tons. The production, which was 90,000 metric tons in 1896, increased to 100,000 tons in 1897. The gain was chiefly in the carbonate ores from Huelva.

*Quicksilver.*—The production was 49,540 flasks, of which 47,357 flasks were from Almaden and 2,203 from other mines; this was an increase of 6,027 flasks from Almaden, and a decrease of 371 flasks from the other mines; a net increase of 5,656 flasks. The exports were 1,742,325 kilos., an increase of 184,270 kilos.

*Zinc.*—The exports of zinc ore were 41,040 metric tons, an increase of 5,164 tons; those of metal were 2,170 tons, a decrease of 1,390 tons. The production of zinc ore diminished, however, having been 60,000 tons in 1897, or 4,828 tons less than in 1896. The decrease was in the output of the Real Compania Asturiana, which reports 20,000 tons from the Reocin mines and 7,200 tons from Udias and La Florida, or 4,000 and 25 tons respectively less than in 1896.

*Antimony.*—The production of antimony was very small; 20 tons of ore were exported, against 120 tons in 1896.

### Glauber Salts in the Caspian Sea.

*Eng. and Mining J., March* 12, 1898, 310.

Russian chemists have recently been investigating a salt deposit found in the Bay of Kara-Bougos, an inlet of the Caspian Sea, on the eastern shore, nearly opposite Baku. The bottom of the bay was found to be covered with a crust of crystallised Glauber salt, in many places 1 ft. thick. The waters of the Caspian Sea are very salt, and in the bay named, which is nowhere 50 ft. deep, evaporation goes on very rapidly. As fuel can readily be obtained from Baku, and transportation by water can be used, it is proposed to establish a factory to make sodium sulphate on a large scale.

### Algerian Phosphate Production.

*Eng. and Mining J., March* 12, 1898, 313.

The following are the shipments of Algerian phosphates from the port of Bone for the full year 1897, as compared with 1896. The shipments are given in metric tons:—

*Shipments of Algerian Phosphates from the Port of Bone.*

| Shipped to | 1896. | 1897. |
|---|---|---|
| Algeria | 1,321 | 598 |
| Austria | 4,240 | 600 |
| Belgium | 1,500 | 2,000 |
| England | 54,005 | 73,430 |
| France | 34,661 | 70,155 |
| Germany | 17,490 | 24,770 |
| Holland | 2,900 | 6,000 |
| Italy | 20,170 | 18,950 |
| Japan | 3,500 | 8,014 |
| Roumania | .. | 1,300 |
| Russia | .. | 100 |
| Spain | 250 | 1,165 |
| Totals | 140,040 | 207,082 |

Of the total production in 1897, Crookston Brothers report 96,547 metric tons, the Constantine Phosphate Company 82,145 tons, and the Société Generale de Phosphates Française 28,390 tons, making in all 207,082 metric tons. These figures represent the total production of Algeria and Tunis. Some deposits in the last-named country will enter in the production of 1898, and their shipments will be made from the port of Sfax.

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 28th February. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,830,336 | 1,645,382 |
| Chemicals and dyestuffs | 673,474 | 550,715 |
| Oils | 711,481 | 655,755 |
| Raw materials for non-textile industries. | 2,795,651 | 2,680,854 |
| Total value of all imports | 37,245,164 | 35,770,874 |

### Summary of Exports.

| Articles. | Month ending 28th February. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 2,542,282 | 2,366,351 |
| Chemicals and medicines | 745,880 | 702,140 |
| Miscellaneous articles | 2,414,666 | 2,481,014 |
| Total value of all exports | 17,864,682 | 17,641,849 |

### IMPORTS OF METALS FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Copper :— | | | | |
| Ore .............. Tons | 2,271 | 2,330 | 27,032 | 22,868 |
| Regulus ........ ,, | 9,148 | 6,721 | 273,661 | 186,354 |
| Unwrought ..... ,, | 4,019 | 5,255 | 202,854 | 263,754 |
| Iron :— | | | | |
| Ore............. ,, | 586,168 | 488,153 | 449,146 | 357,002 |
| Pig............. ,, | 9,904 | 8,562 | 25,436 | 22,122 |
| Bolt, bar, &c. ... ,, | 3,307 | 2,358 | 25,143 | 18,842 |
| Steel, unwrought.. ,, | 7,358 | 2,358 | 39,417 | 17,883 |
| Lead, pig and sheet ,, | 3,004 | 13,012 | 150,374 | 160,613 |
| Pyrites ........... ,, | 54,671 | 48,482 | 81,785 | 77,195 |
| Quicksilver ....... Lb. | 1,257,023 | 822,716 | 115,359 | 76,812 |
| Silver ore....... Value £ | .. | .. | 95,000 | 57,007 |
| Tin ............... Cwt. | 27,938 | 38,646 | 84,284 | 121,554 |
| Zinc .............. Tons | 5,008 | 8,690 | 86,031 | 114,952 |
| Other articles ...Value £ | .. | .. | 174,714 | 148,424 |
| Total value of metals | .. | .. | 1,830,336 | 1,645,382 |

### IMPORTS OF OILS FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut......... Cwt. | 21,275 | 14,233 | 24,115 | 16,115 |
| Olive.............. Tuns | 1,852 | 1,427 | 59,760 | 47,468 |
| Palm .............. Cwt. | 62,906 | 59,933 | 65,313 | 63,850 |
| Petroleum ........ Gall. | 17,663,525 | 23,804,310 | 322,603 | 378,299 |
| Seed .............. Tons | 3,196 | 2,701 | 68,911 | 54,704 |
| Train, &c. ........ Tuns | 254 | 1,132 | 4,869 | 19,343 |
| Turpentine ....... Cwt. | 75,539 | 3,906 | 77,081 | 4,459 |
| Other articles .. Value £ | .. | .. | 88,829 | 71,517 |
| Total value of oils... | .. | .. | 711,481 | 655,755 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,147 | 7,454 | 2,316 | 16,804 |
| Bristles.......... Lb. | 387,902 | 222,601 | 42,779 | 33,463 |
| Caoutchouc ...... Cwt. | 40,311 | 36,775 | 508,496 | 471,857 |
| Gum :— | | | | |
| Arabic......... ,, | 4,407 | 4,180 | 10,270 | 7,214 |
| Lac, &c. ....... ,, | 13,392 | 9,092 | 63,938 | 32,317 |
| Gutta-percha .... ,, | 3,846 | 5,724 | 37,069 | 49,763 |
| Hides, raw :— | | | | |
| Dry............ ,, | 27,940 | 51,333 | 70,679 | 149,261 |
| Wet ........... ,, | 40,700 | 70,402 | 87,999 | 138,731 |
| Ivory ............ ,, | 577 | 237 | 24,902 | 8,581 |
| Manure :— | | | | |
| Guano ......... Tons | 726 | 2,105 | 3,413 | 7,880 |
| Bones ......... ,, | 3,679 | 3,590 | 13,998 | 11,806 |
| Nitrate of soda... ,, | 9,561 | 6,139 | 79,263 | 43,543 |
| Phosphate of lime .. | 26,047 | 23,715 | 41,141 | 36,157 |
| Paraffin.......... Cwt. | 68,874 | 97,256 | 61,536 | 75,940 |
| Linen rags....... Tons | 1 789 | 1,634 | 18,925 | 15,780 |
| Esparto.......... ,, | 19,001 | 20,890 | 83,850 | 81,012 |
| Pulp of wood :— | | | | |
| Chemical ...... ,, | 24,629 | 9,948 | 121,831 | 79,310 |
| Mechanical .... ,, | | 18,034 | | 51,306 |
| Rosin............ Cwt. | 291,729 | 169,837 | 72,340 | 22,472 |
| Tallow and stearin ,, | 78,129 | 89,119 | 79,196 | 87,458 |
| Tar ..............Barrels | 1,253 | 3,537 | 270 | 1,504 |
| Wood :— | | | | |
| Hewn ......... Loads | 123,072 | 120,745 | 237,300 | 199,789 |
| Sawn .......... ,, | 76,451 | 69,060 | 213,562 | 180,137 |
| Staves......... ,, | 5,907 | 5,195 | 30,339 | 34,854 |
| Mahogany ..... Tons | 6,441 | 5,626 | 59,531 | 51,682 |
| Other articles....Value £ | .. | .. | 830,678 | 790,233 |
| Total value ....... | .. | .. | 2,795,651 | 2,680,854 |

Besides the above, drugs to the value of 64,314*l*. were imported, as against 72,130*l*. in February 1897.

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 13,775 | 14,154 | 6,728 | 9,124 |
| Bark (tanners', &c.) ,, | 16,290 | 22,835 | 5,660 | 6,332 |
| Brimstone ........ ,, | 40,392 | 25,050 | 10,088 | 5,634 |
| Chemicals...... Value £ | .. | .. | 94,318 | 103,774 |
| Cochineal ......... Cwt. | 119 | 317 | 680 | 2,074 |
| Cutch and gambier Tons | 1,863 | 1,214 | 26,634 | 18,294 |
| Dyes :— | | | | |
| Alizarin ........Value£ | .. | .. | 21,587 | 16,366 |
| Anilin and other ,, | .. | .. | 41,761 | 41,158 |
| Indigo ............ Cwt. | 17,249 | 12,877 | 307,649 | 217,145 |
| Borax ............ ,, | .. | 1,111 | .. | 830 |
| Nitrate of potash . ,, | 29,706 | 10,220 | 22,648 | 8,371 |
| Valonia........... Tons | 2,803 | 2,374 | 28,993 | 21,887 |
| Other articles... Value £ | .. | .. | 106,725 | 99,726 |
| Total value of chemicals | .. | .. | 673,474 | 550,715 |

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 8,508 | 8,664 | 33,660 | 37,703 |
| Copper :— | | | | |
| Unwrought.... ,, | 28,813 | 25,592 | 76,055 | 65,260 |
| Wrought ...... ,, | 17,943 | 19,582 | 57,768 | 61,352 |
| Mixed metal... ,, | 15,044 | 12,118 | 37,006 | 29,867 |
| Hardware and cutlery. Value £ | .. | .. | 148,276 | 140,515 |
| Implements ..... ,, | .. | .. | 103,988 | 91,860 |
| Iron and steel ... Tons | 261,960 | 230,468 | 1,881,971 | 1,686,421 |
| Lead ............ ,, | 2,876 | 2,279 | 39,074 | 33,729 |
| Plated wares ... Value £ | .. | .. | 25,577 | 26,391 |
| Telegraph wires . ,, | .. | .. | 43,710 | 75,276 |
| Tin ............ Cwt. | 7,179 | 7,001 | 23,624 | 23,427 |
| Zinc............. ,, | 8,941 | 7,703 | 7,402 | 7,715 |
| Other articles .. Value £ | .. | .. | 64,351 | 76,735 |
| Total value ....... | .. | .. | 2,542,282 | 2,306,351 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 696,600 | 512,900 | 16,951 | 11,801 |
| Military stores.. Value £ | .. | .. | 78,974 | 80,284 |
| Candles.......... Lb. | 2,013,000 | 1,815,100 | 28,516 | 24,279 |
| Caoutchouc ..... Value £ | .. | .. | 91,078 | 86,929 |
| Cement.......... Tons | 29,150 | 27,904 | 46,533 | 48,903 |
| Products of coal Value £ | .. | .. | 116,062 | 121,960 |
| Earthenware ... ,, | .. | .. | 123,947 | 120,820 |
| Stoneware ...... ,, | .. | .. | 13,207 | 11,359 |
| Glass :— | | | | |
| Plate........... Sq. Ft. | 118,703 | 146,722 | 6,881 | 8,836 |
| Flint........... Cwt. | 7,034 | 7,759 | 16,812 | 16,535 |
| Bottles......... ,, | 53,015 | 56,238 | 24,381 | 25,549 |
| Other kinds.... ,, | 13,899 | 21,069 | 10,600 | 17,589 |
| Leather :— | | | | |
| Unwrought .... ,, | 12,451 | 13,709 | 111,622 | 114,638 |
| Wrought ..... Value £ | .. | .. | 27,815 | 30,515 |
| Seed oil.......... Tons | 4,519 | 4,036 | 78,137 | 66,570 |
| Floorcloth ..... Sq. Yds. | 2,318,400 | 2,416,600 | 98,238 | 97,320 |
| Painters' materials Val. £ | .. | .. | 119,578 | 135,399 |
| Paper ........... Cwt. | 80,748 | 76,160 | 122,489 | 119,846 |
| Rags............ Tons | 2,681 | 4,582 | 15,135 | 21,486 |
| Soap............. Cwt. | 54,297 | 59,788 | 58,935 | 59,130 |
| Total value ..... .... | .. | .. | 2,114,665 | 2,481,014 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 28TH FEBRUARY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 329,668 | 284,113 | 83,516 | 74,658 |
| Bleaching materials „ | 73,441 | 80,371 | 23,721 | 23,849 |
| Sulphate of copper Tons | 8,737 | 8,175 | 151,452 | 131,715 |
| Chemical manures „ | 34,207 | 31,115 | 178,938 | 182,374 |
| Medicines....... Value £ | .. | .. | 87,312 | 76,535 |
| Other articles... „ | .. | .. | 220,941 | 213,009 |
| Total value .......... | .. | .. | 745,880 | 702,140 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

3690. H. McPhail. An improved method of and means for drying wood and other vegetable substances. Complete Specification. Feb. 14.

3910. M. Daege. Improvements in distilling liquids and in apparatus therefor. Feb. 16.

3926. E. C. Segundo. Improvements in apparatus for sterilising milk and other nutritious substances. Complete Specification. Feb. 16.

3934. W. R. Green. Improvements in air or gas-compressing apparatus. Feb. 17.

4059. J. Prestwich. Improved apparatus for heating, cooling, or condensing. Feb. 18.

4117. E. Roberts and H. R. Dixson. An improved pipe or conduit for fermented or corrosive liquids. Complete Specification. Feb. 18.

4229. A. G. Hoffmann and A. Schwarz. An improved process of and means for heating and evaporating liquids; also applicable to the heating of vapours and gases. Feb. 19.

4342. A. Hanemann.—From — Thomsen, Uruguay. A new or improved composition for use as a non-conductor of heat. Feb. 22.

4421. E. Batault. Improvements in the methods of closing receivers charged with compressed or liquefied gases. Feb. 22.

4459. L. Rümelin and H. Auer. An improved receptacle for the storage of calcium carbide. Complete Specification. Feb. 22

4512. E. Bacault. Apparatus for transfusing the compressed or liquefied gases enclosed in a receiver into another recipient. Feb. 23.

4518. B. H. Scheibler. An improved apparatus for carbonising rags. Complete Specification. Feb. 23.

4860. J. Schumann. Improved heating apparatus. Feb. 28.

4925. C. W. J. Thomsen. Improvements in drying apparatus. Feb. 28.

4966. A. Webster. Improvements in or connected with distilling apparatus. March 1.

5097. W. A. Hull. Improvements in filters. March 1.

5038. H. K. Bamber. A special heating apparatus, with regulated draught for drying gypsum and certain other products at a regulated temperature. March 1.

5325. La Société Anonyme Du Temple and H. Brillié. Improvements in boilers, evaporators, or heaters. Complete Specification. March 4.

5333. L. Sterne. Improvements in condensing apparatus. March 4.

5682. A. Slucki. Improvements in apparatus for heating or cooling liquids, gases, and the like. March 8.

5884. J. Williamson. Improvements in and relating to filter cloths or other similar filtering media. March 10.

6037. H. H. Schou. Improvements in valves for regulating the flow of gases or liquids. Complete Specification. March 11.

6047. G. Dupont. Improvements in carburetting apparatus. March 11.

6076. J. McCulloch. Apparatus for drying organic and inorganic substances. Mar. 12.

### COMPLETE SPECIFICATIONS ACCEPTED.*

1897.

1494. S. Chatwood and S. R. Chatwood. Apparatus for super-heating steam. March 2.

4221. A. W. T. Hülssner and P. Röhrig. Cleaning or clarifying and filtering apparatus. Feb. 23.

4690. J. R. Wright. Improved means or apparatus for carbonating liquids. Feb. 23.

6157. L. Maiche. Apparatus for heating and cooling fluids by fluids. March 9.

7133. J. Gwynne. Method and machinery or apparatus for cooling liquids. March 2.

7547. H. H. Schou. Refrigerating apparatus. Feb. 23.

8596. T. C. Palmer. Improvements in and connected with condensers. Mar. 9.

9345. A. Forrett. An incandescent furnace for heating gas retorts. March 9.

9522. E. Edwards.—From C. Schaaf. Improvements in muffle furnaces. Feb. 23.

10,988. F. J. Collin. Apparatus for cooling and washing gases. March 9.

11,803. J. Williamson. Filter presses and similar filtering apparatus. March 16.

12,752. G. Watson. Improvements in furnaces for burning towns' and other refuse; applicable also to furnaces for general purposes. Feb. 28.

21,285. J. L. Collier. *See* Class XVI.

28,368. J. Leich and S. Delporte. An improved apparatus for compressing and mixing gas and air, more particularly for lighting purposes; also applicable for air compressing. March 9.

1898.

3039. J. Q. Rayner. Improvements in reverberatory furnaces. March 16.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

3643. E. Hopkins. Improvements in or relating to apparatus for generating acetylene gas. Feb. 14.

3684. W. G. Potter. Improvements in and connected with regenerative burners for calcium gas, coal gas, and oil gases. Feb. 14.

3689. F. Küchler. Improvements in or relating to acetylene gas generators. Feb. 14.

3722. C. J. Bailey and J. H. Nicklin. Improvements in apparatus for generating acetylene gas. Feb. 15.

3745. J. H. Ross. Improved acetylene gas generator. Feb. 15.

3770. A. M. Plaissetty. Improvements in or relating to the manufacture of mantles for incandescent gas and other lighting. Feb. 15.

3833. G. Fothergill and J. Fothergill. Improved means of obtaining heat from fuel and abating smoke. Feb. 16.

3834. L. M. Miller. Improvements in the manufacture of acetylene gas. Feb. 16.

3914. L. Etaix. Process for the purification of acetylene gas. Filed Feb. 16. Date applied for Aug. 11, 1897, being date of application in France.

3915. J. F. W. Featherstonhaugh. Improvements in gas-lighting and accessory apparatus. Feb. 16.

4074. A. J. Clitheroe. An apparatus for the generation of acetylene gas. Feb. 18.

4113. H. Berger. Improvements in apparatus for generating acetylene. Complete Specification. Feb. 18.

4129. W. A. Coulson and H. de Thiersant. A new or improved apparatus for the generation of acetylene gas. Feb. 18.

4194. H. Wolpert. Improvements in air carburetting apparatus. Complete Specification. Feb. 19.

4213. L. Rümelin and H. Auer. Improvements in and relating to generators of acetylene gas.

4341. H. O. Oddy. Improvements in acetylene gas generators. Feb. 22.

4352. H. Walker. An improved process and apparatus for the production and purification of acetylene gas. Complete Specification. Feb. 22.

4359. H. Dant. Improvements in apparatus for generating acetylene gas. Feb. 22.

4372. J. H. Ross. Improved acetylene gas generator. Feb. 22.

4410. W. J. Farmer and J. M. Somerville. Improvements in the extraction of cyanogen compounds from coal and coke oven gas. Feb. 22.

4446. P. M. Justice.—From The Allgemeine Carbid-und Acetylen-Gesellschaft mit beschränkter Haftung, Germany. Improvements in apparatus for the manufacture and treatment of acetylene gas. Feb. 22.

4529. J. B. Bourseau. A cartouche or receiver to store gas under pressure. Complete Specification. Feb. 23.

4608. W. G. Forster. Improvements in the extraction of cynogen compounds in the manufacture of coal gas. Feb. 24.

4619. C. W. Smith. Improvements in apparatus for producing artificial fuel. Complete Specification.. Feb. 24.

4717. E. A. Mitchell. Improved apparatus for generating, storing, and enriching hydrogen gas. Feb. 25.

4739. B. von Scheidt. Improvements in apparatus for generating acetylene gas. Feb. 25.

4801. R. J. Moss. Improvements in acetylene gas generators. Feb. 26.

4809. F. Lamplough. Improvements in gas-making apparatus. Feb. 26.

4837. J. St. C. Legge and A. S. Cooper. Improved generator to generate acetylene gas and the like without pressure. Feb. 28.

4856. J. Jacquot. Automatic apparatus for producing acetylene gas. Feb. 28.

5020. F. Bauer and V. J. Woschnagg. Improvements in apparatus for producing acetylene gas. March 1.

5040. A. E. L. Drechsel and C. R. Gunther. Improvements in fabric for incandescent mantles. March 1.

5180. E. G. Gearing. Improvements in or relating to acetylene generating and storing apparatus. March 2.

5317. E. Bauer, F. Freid, and J. Bredel. Process and apparatus for manufacturing acetylene gas by means of carbite. March 4.

5407. H. Kirkham. Improvements in apparatus for the abstraction of impurities from coal and other gases. March 5.

5479. W. Faludi. Improved acetylene apparatus. March 5.

5497. L. P. Hvüd. A new or improved process of producing acetylene gas. Complete Specification. March 5.

5498. L. P. Hvüd. New or improved apparatus for generating and utilising acetylene. Complete Specification. March 5.

5538. L. J. Dedecker. Improvements in apparatus for the automatic production of acetylene gas. March 7.

5578. G. F. Dinsmore. A process of and apparatus for manufacturing gas. Complete Specification. Filed March 7. Date applied for Aug. 9, 1897, being date of application in Belgium.

5585. A. M. Clark.—From Suddeutsche Glükörper Fabrik F. Sauer, Germany. Improvements in the manufacture of incandescent mantles and the like. March 7.

5594. C. H. P. Schlüter and C. L. F. Lüdemann. Improved apparatus for generating acetylene. Complete Specification. March 7.

5667. H. J. Cantley, J. R. Davies, and G. Davies. Improvements in incandescent light. March 8.

5701. R. Clayton and H. B. Steward. Improvements in generators for the manufacture of acetylene and other gases. March 8.

5760. J. Main Improvements in apparatus for the generation of acetylene gas. March 9.

5798. W. Elworthy. Improved apparatus for generating ozone. March 9.

5840. W. Rowbotham. Improvements in apparatus for generating acetylene gas. March 10.

5908. R. Vezin and A. Oudry. Improvements in or relating to apparatus for use in manufacturing acetylene gas. March 10.

5926. O. Lindemann.—From E. Korting, Germany. Apparatus for spraying or atomising combustible liquids and mixing the same with air. Complete Specification. March 10.

5941. F. H. Smith. Improvements in apparatus for the production of illuminating gas. March 10.

5945. F. B. Grundy. An improvement in burners for incandescent gas lights. March 10.

5956. A. Hutchinson and F. A. Hutchinson. Automatic absorptive acetylene gas generator. March 11.

5973. G. F. Thompson. Improvements in portable generators for acetylene gas. March 11.

6006. J. Percival. Improvements in gas generators for acetylene, hydrogen, and such like gases. March 11.

6097. R. Paterson. Improvements in apparatus for the manufacture of acetylene gas. March 12.

6154. C. Crop. Improvements in and relating to incandescent gas and oil illumination. March 12.

### Complete Specifications Accepted.

### 1897.

4709. F. H. Smith. An acetylene gas lamp. Feb. 23.

5445. G. Baldwin and C. Crastin. Apparatus for the manufacture of acetylene gas. March 2.

5817. Count de Hamel de Manin. Manufacture of incandescence bodies for illuminating purposes. March 9.

6464. P. Muschamp and S. Burridge. Manufacture of coke and apparatus and appliances connected therewith. Feb. 23.

6658. W. W. Strode and G. H. White. Manufacture of acetylene. Feb. 23.

7172. Comte Albert Dillon de Micheroux. New process to desulphurate coals, to destroy their smoke and bad smell, to increase the number of their calories, &c. Feb. 23.

7918. J. G. A. Kitchen. Improvements in or relating to acetylene generators. Feb. 23.

7929. F. H. Smith. An acetylene gas lamp or generator. Feb. 23.

8551. J. H. Exley. Apparatus for manufacturing acetylene gas. Feb. 23.

9045. C. O. Rusling. Improvements in protecting and sustaining the mantles of "Welsbach" and similar lights. March 16.

9656. W. A. G. v. Heidenstam. Manufacture of charcoal. March 16.

9763. J. G. A. Kitcher. Improvements in portable generators for producing acetylene from calcium carbide.

10,796. H. J. Barbier. Means for the storage of compressed gas. March 15.

15,317. M. Graetz. Improvements in oil burners for the production of non-luminous flames, especially for heating incandescent bodies.

15,754. F. H. Smith. Improvements in acetylene gas generators. March 9.

20,051. F. Rhind. Improvements in gas generating. Mar. 2.

21,150. J. Carter. The Carter incandescent gas light mantle and chimney protector. March 16.

27,652. M. Bernstein. Proprietor of the Firm of J. F. Wallmann and Co. Process for the manufacture of automatic igniters for illuminating gas. March 16.

28,368. J. Leich and S. Delporte. *See* Class I.

29,796. M. Bernstein. A process for manufacturing incandescent bodies. March 16.

30,637. J. Mücke and J. Mücke. Improvements in acetylene gas generators. Feb. 23.

30,689. G. W. Lee, F. Elliott, and G. Tatham. Process and apparatus for the manufacture of inflammable gas, and composition to be used therein. March 9.

1898.

49. B. Puchmüller. An improved fluid and process of manufacturing the same, for impregnating bodies or suitable fabric, threads, fibres, or the like, for incandescent lighting purposes. Feb. 9.

1013. E. Bournonville. Improvements in acetylene gas generators. March 9.

1665. R. Quatannens-Moens and E. Carreer-Dilger. Machine for producing acetylene gas for private and public lighting. March 16.

3493. W. MacKean. A burner for burning off incandescence mantles. March 16.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Complete Specification Accepted.

1411. V. T. Ragosin. A method of distilling, fractioning, and cracking mineral oils, and obtaining various products therefrom, and apparatus for that purpose. March 16.

## IV.—COLOURING MATTERS and DYES.

### Applications.

3671. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of acetylisoeugenol. Feb. 14.

3680. J. Hauff. Method of oxydation of ortho-toluolsulphamide to saccharine by avoiding the formation of ortho-toluol-sulpho acid or ortho-sulpho-benzoic acid. Feb. 14.

4045. A. G. Green, R. Levy, and The Clayton Aniline Company, Ltd. Manufacture and production of intermediate products, and of colouring-matters therefrom. Feb 17.

4226. H. R. Vidal. Improvements in the preparation of paramidophenol. Feb. 19.

4534. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the production of quinonoxime colouring matters (nitroso-phenols) upon the fibre. Feb. 23.

4545. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of pyridine colouring matters. Feb. 23.

4585. A. Ashworth and J. Bürger. Improvements in the production of colouring matters. Feb. 24.

5257. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of colouring matters for cotton. March 3.

5456. F. Valentiner. Improvements in the production of acetophenonphenetidide. Complete Specification. March 5.

5593. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of a new colouring matter. March 7.

5746. W. E. Heys.—From The Chemical Works, formerly Sandoz, Switzerland. The manufacture or production of brown polyazo colouring matters. March 9.

5915. H. R. Vidal. Improvements in the manufacture of colouring matters. March 10.

5916. H. R. Vidal. Improvements in the manufacture of colouring matters. March 10.

5949. G. W. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or production of xanthines and their derivatives. Complete Specification. March 10.

6055. J. Imray.—From La Société L. Durand, Huguenin, et Cie., France. Manufacture of new colouring matters from oxazines. Complete Specification. March 11.

6056. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Method of producing the tannin-antimony and the chromium compounds of the quinoneimide dyestuffs from their components on the fibre, prepared with a pheool, and on the fibre not so prepared in printing. March 11.

6057. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Method for the manufacture of sulphur trioxide (sulphuric anhydride). March 11.

### Complete Specifications Accepted.

1897.

5697. H. R. Vidal. Preparation of paramidophenol and the like. March 9.

6913. H. R. Vidal. Manufacture of colouring matters. March 2.

8235. G. W. Johnson.—From Kalle and Co. Manufacture and production of azo dyestuffs or colouring matters suitable for dyeing cotton. March 2.

9268. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of 1-phenyl 2-alkylpyrazolones. March 9.

## V.—TEXTILES : COTTON, WOOL, SILK, Etc.

### Applications.

3621. A. Hoegger. Improvements in and connected with the production of lustrous effects on double vegetable threads. Feb. 14.

3685. J. Eddison. An improvement in the manufacture of wadding. Feb. 14.

3898. H. C. Woltereck. Improvements in and in the manufacture of artificial silk. Complete Specification. Feb. 16.

5368. S. H. Sharp. Producing silk or satin stripes or design effects upon cotton and other fabrics. March 4.

### Complete Specifications Accepted.

1897.

9056. E. Crepy. Method of and means for "mercerising" vegetable fibres, fabrics, and the like. March 2.

11,313. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in mercerising vegetable tissues. March 9.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

3604. J. T. Reid and H. Thorp. Improvements in dyeing textile fibres, yarns, and fabrics. Feb. 14.

3672. R. C. W. A. Sieverts. Treatment of rattan, bamboo, and other plants having a silicious outer coating for the bleaching and dyeing thereof. Feb. 14.

3775. T. F. Stimpson. Improved method of printing on fabrics. Complete Specification. Feb. 15.

4199. R. Hutchison. Improvements in apparatus for use in stretching, scouring, washing, preparing, and dyeing tops, yarns, and fabrics. Feb. 23.

4613. T. Cadgène. Improvements in or relating to the dyeing of textiles and the like, and apparatus therefor. Feb. 24.

4967. F. S. Sandeman. Improvements in treating or bleaching jute. March 1.

5063. W. P. Thompson.—From J. Ratcliffe and M. Goldavsky, Russia. Improved method and apparatus for preparing laps of textile materials for dyeing purposes. March 1.

5087. J. S. Lord and G. S. Lord. Improvements in apparatus for dyeing, scouring, and washing hanks. March 2.

5475. J. Bündgens and L. Bündgens. Improvements in or relating to dyeing vats. Complete Specification. March 5.

5714. W. Mather and R. H. Haworth. A process and apparatus for continuous bleaching of fabrics or yarns in open condition. March 8.

### Complete Specifications Accepted.

1897.

8114. O. H. Stone. Process of colouring papers. March 9.

10,901. J. Imray.—From La Société Anonyme A. Roudillon et Cie. Process for fixing interference colours on paper or other surface. March 2.

27,270. C. Delescluse. Improvements in bleaching cotton, linen, and other vegetable textile materials. March 2.

1898.

1634. F. Erban and L. Pick. Improved method of bleaching, dyeing, washing, or treating fabrics and other materials with liquid, and apparatus therefor. March 16.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

1985a G. Bower.—From J. E. Hewes, United States. Improvements in the process for the manufacture of carbide of calcium, and apparatus therefor. Filed March 4. Date claimed Jan. 25, 1898.

3801. J. Y. Johnson.—From H. Dercum, United States. Improvements in the manufacture or regeneration of chromates or bichromates from waste battery or other liquors, or from chrome alum or sesqui salts of chromium. Complete Specification. Feb. 15.

4033. J. Landin. An improved method of producing carbides. Complete Specification. Feb. 17.

4298. P. Jensen.—From J. von Orlowsky, Russia. Process for rendering carbide non-hygroscopic. Complete Specification. Feb. 21.

4567. E. J. C. Christie. A method for rendering nitrate of ammonia anhydrous and non-deliquescent. Feb. 24.

4861. C. Diesler and F. Dieckmann. A process for the fabrication of metallic carbides. Feb. 28.

4862. C. Diesler and F. Dieckmann. Process for manufacturing metallic carbides. Feb. 28.

4863. C. Diesler and F. Dieckmann. Process for manufacturing metallic carbides. Feb. 28.

4866. G. Bower.—From J. E. Hewes, United States. Improvements in the process of making calcium carbide. Feb. 28.

4873. B. Crowther and B. Crowther, jun. A new or improved method of separating tannic acid from tannin solution so as to render the tannic acid applicable for dyeing and other purposes. Feb. 28.

5004. L. O. Helmers. Improvements in the production of metallic salts and salts of the alkaline earths. March 1.

5009. H. H. Lake.—From La Société Coignet et Cie., France. Improvements in the manufacture of nitrophosphates. March 1.

5293. P. Naef. Process of and apparatus for the manufacture of chlorine. Filed March 3, date applied for Aug. 7, 1897, being date of application in United States.

5481. W. Coleman. A process for extracting acetic acid from liquids containing it. March 5.

5496. W. W. McLauchlan. Improved method of charging, turning, and discharging bleaching powder chambers and the like. March 5.

6037. H. H. Seon. Improvements in valves for regulating the flow of gases or liquids. Complete Specification. March 11.

### Complete Specifications Accepted.

1897.

6648. T. Keene. Furnaces for the manufacture of calcium carbide or the like. Feb. 23.

7066. W. Garroway. Manufacture of ammonium nitrate and other salts. March 16.

8335. W. Wyld and J. H. Cox. Means or method of manufacturing ferric sulphate. Feb. 23.

9099. T. Savage. Production of nickel sulphate. March 2.

9215. H. R. Vidal. Process for manufacturing alkaline cyanides and other cyanated products. March 9.

10,361. C. Masson and C. Tilliere. Improvements in the preparation or manufacture of borax. March 16.

10,509. T. T. Sill. *See* Class X.

17,609. I. L. Roberts. Process of and apparatus for the manufacture of metallic carbides. March 9.

29,746. P. Jensen.—From L. Rohrmann and H. H. Niedenführ, Germany. Process and apparatus for obtaining nitric acid from low-graded nitro-oxygen compounds. Feb. 24.

1898.

32. J. Leede. Improvements in the art of producing calcium carbide. Feb. 23.

## VIII.—GLASS, POTTERY, and ENAMELS.

### Applications.

4708. G. M. Henley. Improvements in and relating to china-clay kilns. Feb. 25.

5073. E. J. Lutwyche. Improvements in and relating to ornamenting glass. Complete Specification. March 1.

6087. J. Bladon. Novel or improved glaze for pottery ware. March 12.

### Complete Specifications Accepted.

1897.

6840. A. D. Brogan, A. M. Malloch, and J. Craig. Leers or kilns for annealing glass. March 2.

11,343. R. F. Ayre and C. Dunnill and Co., Ltd. Improvements in pottery-kilns or ovens. March 9.

30,598. W. B. Johnson.—From G. W. Blair and H. I. Hays, United States. Improvements in the method of and apparatus for making hollow glass articles. March 9.

1898.

2112. N. Browne.—From The Glashüttenwerke Alderhütten Actien Gesellschaft, Germany. Improved hollow glass blocks for the construction of walls pervious to light. March 16.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

### Applications.

3657. A. E. E. Cantley and E. Springbom. A new composition for the manufacture of moulded articles. Feb. 14.

3793. W. S. Wilkinson. Improvements in the refining of asphalt. Complete Specification. Feb. 15.

3900. F. Grumbacher. An improved process for preserving, colouring, and hardening wood. Complete Specification. Feb. 16.

4260. J. Hutcheson and J. F. Service. An improvement in fireclay bricks and all other kinds of mortar. Feb. 21.

4343. J. J. Lish. Improvements in the manufacture of slabs and blocks for pavements, steps for stairs, and for other purposes. Feb. 22.

4513. C. Shoenfelder. Improvements in and in the manufacture of artificial stone, and in furnaces for burning the same. Feb. 23.

4594. F. J. Bergmann. Improvements in the manufacture of cement and building blocks. Complete Specification. Feb. 24.

4709. H. V. Simpson. Improvements in or relating to the impregnation of wood with fireproofing, waterproofing, preserving, or colouring materials. Feb. 25.

5351. P. A. Winkler. New method for the production of hard stones, especially lithographic stones. Complete Specification. March 4.

5812. W. P. Thompson.—From A. Domer, France. Improvements in the preparation of cork and cork waste refuse and powder with a view to its utilisation as a resistant against damp and heat for building and other purposes. March 9.

6063. E. Burton. An improved stone paving for streets and roads. March 12.

### Complete Specifications Accepted.

1897.

7798. T. Parker.—From G. W. Parker. Improvements in the art, method, or process of treating gypsum rock for the production of artificial marble and the like. Feb. 23.

9030. T. C. Hutchinson. Treatment of blast-furnace slag or scoria for the manufacture therefrom of hydraulic or similar cement, or for other purposes requiring use of dry granulated slag. March 2.

9072. D. Wilson. Cement or concrete. March 2.

9073. D. Wilson. Manufacture of slabs for the beds of billiard tables and other purposes, steps and blocks for building and other purposes, pillars, columns, posts, poles, chimney pots, pipes or tubes, baths, sinks, tanks, buckets, casks, drums or barrels, and other hollow articles and vessels. March 9.

20,670. T. Holden and C. Major. Improvements in the manufacture of cement.

1898.

2142. N. Browne.—From Glashüttenwerke Alderhütten Actien Gesellschaft, Germany. *See* Class VIII.

## X.—METALLURGY, MINING, Etc.

### Applications.

10,829A. J. Swinburne. Improvements in treatment of sulphide ores. Filed Feb. 25. Date claimed May 1, 1897.

3653. W. S. Rock and J. Sinel. Improvements in extracting metals from sea water. Feb. 14.

3654. W. S. Rock. Improvements in extracting metals from sea water. Feb. 14.

3668. H. W. Stillwell. An improved method of soldering aluminium joints. Complete Specification. Feb. 14.

3795. F. B. Aspinall and E. C. Ekströmer. Improvements relating to the extraction of precious metals from their ores. Feb. 15.

3807. G. F. Smith. Improvements in apparatus for extracting precious metals by chlorination. Complete Specification. Feb. 15.

3810. W. P. Thompson.—From B. Talbot, United States. Process of refining metals. Feb. 15.

3830. S. O. Cowper-Coles. Improvements in the reduction of tin oxides. Feb. 15.

4012. A. Reynolds. An improvement in the treatment of iron and steel in open-hearth furnaces, and appliances therefor. Feb. 17.

4296. J. Jones. Improved process for the recovery of zinc from refractory zinc-bearing ores. Complete Specification. Feb. 21.

4422. L. Liebmann. Improvements in the production or obtainment of metallic beryllium from materials containing it. Feb. 22.

4456. H. J. Kirkman. Improvements in apparatus for extracting gold from ores and the like. Feb. 22.

4483. F. Pich. Improved process and means for hard-soldering cast-iron. Feb. 23.

4603. A. Gutensohn and R. W. W. Rising. Treatment of sea sand and the like for recovery of precious metal. Feb. 24.

4911. H. E. Fry and R. Addie. Improvements in the recovery of zinc from zinciferous slags or residues. Feb. 28.

5347. J. B. de Alzugaray. Improvements in and connected with the metallurgical treatment of sulphide ores, sulphurets, mattes, and with the extraction and separation of metals therefrom. March 4.

5568. P. E. Secrétan. An improved aluminium nickel bronze. March 7.

5719. E. B. Parnell. Improvements in or relating to apparatus for the treatment of metalliferous ore and the like. March 8.

5722. E. J. Ball. Improvements in the metallurgical treatment of metallic ores and other metalliferous substances. March 8.

5843. G. M. Jackson. Improvements in appliances for dealing with molten iron from the blast furnace for use in the production of castings. March 10.

5943. S. O. Cowper-Coles. Improvements in the treatment of zinc-bearing ores for obtaining zinc and other metals therefrom. March 10.

### Complete Specifications Accepted.

1897.

3926. H. Niewerth. Manufacture of iron and steel. Feb. 23.

4857. W. A. Koneman and W. A. Hartley. Treatment of ores. Feb. 23.

6489. J. Davies and A. Sanders. Fluid furnaces for heating or smelting or refining metals. Feb. 23.

7555. H. R. Cassel and B. C. Hinman. Process of and apparatus for extracting gold from its ores and compounds. March 16.

10,509. T. T. Sill. Manufacture of red oxide from the waste liquors from galvanising works, tin-plate works, wet copper process, or other residual liquors containing iron in the form of chloride. March 16.

10,715. J. L. Dobell. Manufacture of cupels and crucibles to be employed in the refining of metals. March 16.

30,297. J. L. Stevenson and J. Evans. Cast-steel water-cooled tuyère and tuyère arch for blast furnaces. Feb. 23.

1898.

142. A. Pigott. Treatment of lead ores. March 9.

224. A. J. Boult.—From H. L. Hartenstein and G. A. Weber, United States. Improvements in or relating to the utilisation of waste products from metallurgical furnaces and the like. March 9.

976. C. E. Greene. Improvements in plates for collecting or separating gold. March 16.

2118. A. H. Pehrson and E. V. Pehrson. Improvements in moulded materials for use in the manufacture of steel castings, and in the method of preparing the same. March 16.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### Applications.

3749. J. G. A. Rhodin. A method of oxidation and bleaching by means of electrolysis. Feb. 15.

3838. A. Webb. Improvements in electric accumulators. Feb. 16.

4075. H. Maxim. Improvements in methods and apparatus for electro-thermally treating materials, more particularly for the manufacture of calcium carbide and other carbides, and the reduction of ores. Feb. 18.

4150. T. W. Allan, and Allan and Adamson, Ltd. Improvements in electrical accumulators or storage batteries. Feb. 19.

4306. P. W. Northey. Improvements in electrodes for secondary batteries. Feb. 21.

1514. V. I. Feeny.—From H. Becker, France. Improvements in and relating to cathodes. Feb. 23.

1624. J. Heibling. The electrolytic manufacture of alkaline bichromates. Feb. 24.

4903. J. Ensign Fuller. Improvements in primary batteries. Complete Specification. Feb. 28.

4910. C. Koenig. Improvements in and relating to primary batteries. Complete Specification. Feb. 28.

5186. R. Haddan.—From H. Dolter, France. Improvements in or relating to secondary batteries. Complete Specification. March 2.

5260. E. A. Mitchell. An improved primary battery. March 3.

5703. H. Pieper, Fils. Improvements in electrodes for secondary batteries or accumulators. March 8.

5704. H. Pieper, Fils. Improvements in electrodes for secondary batteries or accumulators. March 8.

5705. H. Pieper, Fils. Improvements in the manufacture and production of electrodes for secondary batteries. Mar. 8.

5796 Electric Reduction Company, Ltd.—From W. T. Gibbs, Canada. A method of and apparatus for obtaining phosphorus from materials containing it. March 9.

5912. G. Laura. Improvements in electrical galvanic batteries. Complete Specification. March 10.

6013. J. Hargreaves. Improvements in the manufacture of carbon. March 11.

6022. P. Lafargue and E. Drouet. Improvements in primary voltaic or galvanic batteries. March 11.

6028. J. Holloway. Improvements in the method of and apparatus for the electro-deposition of metals. March 11.

6044. A. Werner. Improvements in secondary batteries. March 11.

6145. R. von Barby. Process for hardening and rendering tenacious the active mass of electric accumulators. Complete Specification. March 12.

6147. H. Riecken. Improvements in the electrolytical treatment of ores and slimes for the recovery of precious metals therefrom, and apparatus therefor. March 12.

### Complete Specifications Accepted.

### 1897.

2272. J. L. Dobell. Improvements in or connected with electric batteries. March 9.

5826. A. Schanschieff. Active material of secondary batteries. March 9.

6929. F. Taylor, A. Cooke, and R. W. D. Montgomery. Diaphragm for separating the gases in electrolysis of sodium chloride and other salts, and other improvements. March 9.

7027. A. J. Marquand. Secondary batteries or accumulators, and the manufacture thereof. March 9.

13,100. C. N. Gauzentés. Improvements in or connected with primary or galvanic electric batteries. March 2.

26,919. O. C. Strecker and H. H. Strecker. Improvements in or relating to the application of electrodes in electrolytic processes. March 9.

26,921. O. C. Strecker and H. H. Strecker. Improved process for preparing insoluble or hardly soluble salts from metallic anodes by electrolysis of aqueous solutions containing two salts. March 9.

26,923. O. C. Strecker and H. H. Strecker. Improvements in or relating to the production of protoxides and oxides or of hydroprotoxides and hydroxides from metallic anodes by electrolysis of alkaline salt solutions. March 9.

29,710. D. Browne and P. Kamperdyk. Improvements in accumulators. Feb. 23.

### 1898.

1194. G. Philippart. Improvements in secondary batteries. March 16.

## XII.—FATS, OILS, and SOAP.

### Applications.

4153. E. G. Hughes. Improvements in the manufacture and composition of soap for toilet and laundry purposes. Feb. 19.

4577. L. Grote. Improvements in the manufacture of soap. Feb. 24.

4634. P. Candelot and Z. Candelot. An improved cleaning fluid. Feb. 24.

4741. H. H. Lake.—From E. Twitchell, United States. Improvements relating to the separation of fats or oils into glycerine and fatty acids, and to compounds for use therein. Complete Specification. Feb. 25.

4773. S. V. Kay and Kay Bros., Ltd. Improvement in soap. Feb. 26.

4916. W. P. Thompson.—From Anatole Des Cressonnières and Ernest Des Cressonnières, Belgium. Improvements in the manufacture of soap. Feb. 28.

5167. W. P. Thompson.—From Anatole Des Cressonnières and Ernest Des Cressonières, Belgium. Improvements in crushing or kneading and mixing apparatus for soap and other like material. March 2.

### Complete Specification Accepted.

### 1897.

8796. E. G. Scott. Improvements in the saponification of fatty matters and in the manufacture of glycerine. Feb. 23.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

3909. R. H. Bitfen and E. W. Howard. Improvements connected with the treatment of india-rubber, gutta-percha, and other gums. Feb. 16.

5046. C. H. Gray. Improved manufacture of ebonite or the like. March 1.

5080. C. Williams. The lacquer ship's bottom preservative and anti-fouling composition. March 2.

5373. J. F. Dell. An improvement in or applicable to anti-fouling compositions and the like. March 4.

5914. S. O. Cowper-Coles. A process for the direct manufacture of white lead from lead-bearing ores. March 10.

6043. C. Heinzerling. Improvements in the treatment of old or waste vulcanised rubber to render it capable of re-use. March 11.

### Complete Specifications Accepted.

### 1897.

9538. F. King. Improvements in or connected with the manufacture of articles wholly or partly of rubber or other gum and the like dissolved in a solvent. March 9.

9775. J. S. MacArthur. Improvements in making white lead. March 9.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

3678. A. MacFarlane and H. W. Stanbury. Improvements in the process of treating gelatine and the production therefrom of imitations of various substances. Feb. 14.

3959. J. Young. Improvements in apparatus for tanning, dressing, or otherwise treating hides or skins and leather. Feb. 17.

3994. J. B. Scammell, E. A. Muskett, and J. H. Smith. Improvements in or relating to the manufacture of japanned or patent leather. Feb. 17.

4262. L. Partl and C. Binzegger. Improvements in the process of and means for tanning. Feb. 21.

4763. J. K. Tullis. Improvements in treating hides and skins. Feb. 26.

4761. N. P. Andersen, J. Westengaard, and H. Zerener. Improved process and apparatus for tanning by the aid of electricity. Feb. 26.

4814. C. Smith. Improvements relating to apparatus for tanning hides and skins. Feb. 26.

4873. B. Crowther and B. Crowther, jun. *See* Class VII.

5003. G. Fettweis. Improvements relating to the tanning of skins and the like and to apparatus therefor. March 1.

5164. A. Stephan. An apparatus for the continuous treatment of size by boiling. Complete Specification. March 2.

5619. J. Williamson. Improvements in and relating to the refining of animal substances and the like. March 8.

5826. E. Heyl-Dia. Improvements in the manufacture of artificial leather. March 9.

6144. E. S. Cook. Improved method of preparing raw and other hides for the manufacture of travelling trunks, portmanteaus, and the like therefrom. March 12.

### Complete Specifications Accepted.

1897.

8042. A. Mitscherlich. Process for the manufacture of glutinous substances. March 16.

8391. A. Zimmermann. Treatment of gelatinous substances, skins, and the like. March 9.

12,326. S. Herzberg. A new or improved vegetable glue and a process for manufacturing the same. March 9.

12,450. P. G. Stanford. Improvements in the treatment of tanning liquids for clarifying, bleaching, or decolorising them. March 2.

## XV.—AGRICULTURE AND MANURES.

### Application.

5511. W. Wardle. *See* Class XVII.

### Complete Specification Accepted.

1897.

9369. A. Gladstone. Improvements in artificial manures or fertilisers. March 2.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

3659. E. R. Dinsmore. An improved starch mixture. Complete Specification. Feb. 14.

4532. W. Painter. Improvements in industrial compounds involving the use of gluten. Complete Specification. Feb. 23.

5183. J. E. Dunn. Improved method of and means for heating the juice or saccharine for defecating and other purposes in the manufacture of sugar. March 2.

5803. E. Shaw. Improvements in the method of and apparatus for preparing syrup consisting of sugar or glucose, or a mixture thereof, for use in the manufacture of sweetmeats. March 9.

### Complete Specifications Accepted.

1897.

1155. A. Colette, jun., and A. Boidin. Process for utilising moulds or microscopic fungi for extracting from residues, particularly those arising from the treatment of amylaceous and sugary substances, alcohol, and mucidines capable of being used in panification, distillation, saccharification, and the like. Feb. 23.

21,285. J. L. Collier. Improvements in steam pans for boiling sugar and other substances. March 16.

1898.

1285. C. D. Abel.—From E. L. Erben, Germany. Process for the refining of raw sugar. March 16.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

3674. E. S. Beaven. Improvements relating to the preparation of grain for malting. Feb. 14.

4686. R. D. Bailey. Combined mashing, wort-heating, and wort-cooling vessels for brewers' and distillers' use. Feb. 25.

4943. A. F. Watson. A process for recovering beer, ale, and porter from vat bottoms, vat sweepings, or vat deposits generally. March 1.

5511. W. Wardle. For the drying of barm or yeast for food or manure for future use. March 7.

5899. W. J. Hanlon. An economic receptacle for brewers when racking malt liquor. March 10.

### Complete Specifications Accepted.

1897.

5953. J. R. Swift. The beer preserver. Feb. 23.

8223. J. G. Hathaway. Method of and apparatus for purifying and maturing alcoholic liquors. March 9.

11,600. A. C. Briet. Diffusion apparatus for the manufacture of cider. March 9.

30,658. R. M. de Tangry. An improved process and apparatus for accelerating the ageing of alcoholic liquors. March 9.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

*A.—Foods.*

3819. E. F. Okell. An improved coffee substitute. Feb. 15.

4604. D. R. S. Galbraith. Improvements relating to the preservation of butter. Feb. 24.

4917. E. Stein. Improvements in the sterilisation and purification of water; also applicable to beverages and alimentary substances. Feb. 28.

5814. F. Dieckmann. Improvements in or relating to the preservation from decomposition of organic substances. March 9.

5965. A. Classen. Manufacture of new non-coagulable albuminous bodies from meat and blood, and of meat extracts which contain all the constituents of meat in an unaltered form, and processes therefor. Complete Specification. March 11.

6123. R. T. Hewlett. An improved mode of and apparatus for scalding, pasteurising, or sterilising milk. March 12.

*B.—Sanitation.*

4994. W. D. Scott-Moncrieff. Improvements in or relating to the bacteriological purification of sewage. March 1.

5603. J. J. Deery. Improvement in purification process. Complete Specification. March 8.

6061. H. H. Leigh.—From La Société Anonyme pour les Applications Industrielles de l'Ozne, France. Apparatus for the sterilisation or the disinfection of waters of all kinds, treatment of sweetened syrups and other analogous applications by means of ozone, air, or other gases. Complete Specification. March 11.

*C.—Disinfectants.*

3923. E. Copeland. Manufacture of a composition for preserving milk and its products. Complete Specification. Feb. 16.

### Complete Specifications Accepted.

*A.—Foods.*

1897.

4695. J. Hogarth. Treatment of alimentary substances. Feb. 23.

*B.—Sanitation.*

1896.

25,708. B. H. Scheibler. New or improved apparatus for carbonising rags. Feb. 23.

1897.

4040. F. P. Candy. Purification of sewage and polluted water, and apparatus therefor. March 2.

5007. L. Maiche. Apparatus for sterilising water. Feb. 23.

*C.—Disinfectants.*

1897.

6823. J. L. Mortimer and J. H. Williams. A new and improved disinfecting and antiseptic block. March 16.

1898.

2407. R. Ehrhardt. Manufacture of antiseptic soaps containing mercury albuminate. March 16.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

3633. F. A. Rouse. Improvements in blotting paper. Feb. 14.

3657. A. E. E. Cantley and E. Springbom. *See* Class IX.

4945. W. Curtis. Improvements in settling and recovering apparatus for paper and pulp manufacture. March 1.

### Complete Specification Accepted.

1897.

6054. J. S. Rigby, H. Sweetapple, and J. Clarke. Apparatus or machinery for the manufacture of vegetable parchment paper. March 9.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

4005. G. B. Ellis.—From Sedan and Hyppolite Fraissinet, France. Improvements in the production of formaldehyde vapours. Feb. 17.

4211. C. D. Abel.—From Anilinöl Fabrik. A. Wülfing, Germany. Manufacture of preparation of iron and albumen. Feb. 19.

4227. H. R. Vidal. Preparation of hydrocyanic acid and of hydrocyanic ethers. Feb. 19.

4280. J. C. Mewburn.—From Chemische Fabrik von Heyden, Gesellschaft mit Beschraenkter Haftung, Germany. Improvements relating to the employment and administration of vanillin. Complete Specification. Feb. 21.

4736. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of derivatives of purine containing sulphur. Complete Specification. Feb. 25.

4779. J. Y. Johnson.—From A. Verley, France. Improvements in the manufacture and production of perfumes. Feb. 26.

5177. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. A process for obtaining strong tuberculosis toxins from tubercle bacilli. March 5.

5747. R. Mackill. Improvements in the manufacture or obtainment of nicotine. March 9.

5947. J. W. Mackenzie.—From C. Schmid, Belgium. Improvements in artificial perfumes. March 10.

5960. J. W. Mackenzie.—From C. Schmid, Belgium. Production of a civet-like perfume. March 11.

### Complete Specifications Accepted.

1897.

6790. E. H. C. Durkopf. Manufacture of products of condensation from formaldehyde and tannin. March 9.

8915. J. Y. Johnson.—From C. F. Boehringer and Soehne. Method of preparing alkylated dichloroxypurines. March 2.

10,594. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier. Preparations for producing local anæsthesia. March 9.

14,328. J. E. T. Woods. Further improvements in the manufacture of substitutes for camphor, called camphorete, and products made therefrom. March 16.

## XXI.—PHOTOGRAPHY.

### Applications.

3961. C. A. Burghardt. Improvements in the method of and apparatus for producing photographic pictures and images with relief or stereoscopic effects. Feb. 17.

4044. J. Joly. Improvements in or relating to the art of producing photographs in natural colours. Feb. 17.

4622. H. C. Rheinlander and A. Wrench. Improvements in or connected with films for cinematographs. Feb. 24.

5721. C. Pietzner. Process for the production of relief photographs. March 8.

5988. P. E. B. Jourdain. Improvements in processes for photography in natural colours. March 11.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

3863. G. H. Hosie. Improvements relating to explosives and their use. Feb. 16.

3976. B. Schratzenthaller. Process for the manufacture of smokeless powder. Complete Specification. Feb. 17.

4034. H. Boyd. Improvements in explosives. Feb. 17.

4192. J. Regensburger. Improvements in the manufacture of explosives from molasses. Feb. 19.

5286. A. Luck and C. F. Cross. An improvement in the treatment of nitro cellulose for the manufacture of explosives and other products. March 3.

5533. W. A. Malson and S. R. Malson. Improvements in electric high and low tension fuse heads for blasting purposes. March 7.

### Complete Specifications Accepted.

1897.

6772. F. Müller, S. Oberlander, V. H. Fuchs, and S. Gomperz. Improved blasting compound, and process for its manufacture. Feb. 23.

6995. B. E. Dickinson and Kynoch, Ltd. Improvements in fog signals. Feb. 23.

9111. W. Greaves and E. M. Hann. Improvements in or relating to explosives. Feb. 23.

9535. T. Tevlev. Improvements in explosives. Feb. 23.

9970. E. A. G. Street. Improvements in or relating to explosives. March 9.

12,701. F. Render. Improvements in explosive cartridges. March 9.

15,693. F. G. du Pont. Improvements in and relating to process and apparatus for beating guncotton and other purposes. March 2.

29,064. F. A. Debize and L. F. Chasseigne. Improvements in machinery for the continuous manufacture of wooden matches. Feb. 23.

1898.

228. W. Salter. Improvements in matches and flaming fusees. March 16.

1007. W. L. Gordon. Improvements in lucifer matches. March 16.

## PATENTS UNCLASSIFIABLE.

### Applications.

4628. A. Imschenetzky. Process for the manufacture of a new refractory material. Feb. 24.

4927. E. Callait. An improved chemical product Feb. 28.

5271. W. H. Akester. An improved composition of materials applicable for general use where acid-proof, fire-proof, and waterproof qualities are required. March 3.

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THE JOURNAL

OF THE

# Society of Chemical Industry.

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No. 4.—Vol. XVII.] APRIL 30, 1898. [Non-Members 30/- per annum; Members 21/- per Set of extra or back numbers; Single Copies (Members only) 2/6.

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The names in *italics* are those of Members of Committee who retire at the end of the current session.

# NOTICES.

## Annual General Meeting.

The Annual General Meeting will be held in Nottingham on the 13th, 14th, and 15th July next. A Programme is in preparation. Tickets of Membership will be issued in time for the meeting.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. George Beilby has been nominated to the office of President; Prof. F. Clowes, D.Sc., has been nominated Vice-President under Rule 11; and Mr. George E. Davis, Mr. John Heron, and Dr. Chas. A. Kohn have been nominated Vice-Presidents under Rule 8.

Sir Robert Pullar has been nominated an Ordinary Member of Council under Rule 24, and the Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council. Forms for this purpose can be obtained from the General Secretary on application.

*Extract from Rule* 18:—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

## Alteration of Byelaws.

Notice is hereby given, in accordance with Rule 54 of the Bye-laws, that the Council will propose to the forthcoming Annual General Meeting the amendment of the Society's Bye-laws as follows:—

1. Rule 7. To add rule 19 to the list of exceptions.
2. Rule 19. To add "and no ballot shall be taken. If four persons only be nominated, no ballot shall be taken."
3. Rule 20. To add at beginning "If the number of persons nominated, as provided by Bye law 18, be more than four—"
4. Rule 56. To replace the words "be at the discretion" by the words "subject to the sanction."

## Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

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|---|---|---|
| To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List. | Each copy | 10*s.* |
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Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

## Telegrams.

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Warden, Dr. C. J. H., l/o Calcutta; c/o Grindlay and Co., Parliament Street, Westminster, S.W.

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# Liverpool Section.

*Meeting held Wednesday, March 30th, 1898.*

DR. C. A. KOHN IN THE CHAIR.

## JAPANESE WOOD OIL.

BY ROWLAND WILLIAMS.

Although wood oil ("tung oil") (this Journal, 1897, 684) can hardly be regarded as a commercial article so far as this country is concerned, I understand that it has been imported to a certain extent during the last year or so. A few analyses of the oil have been published from time to time, but, to the best of my knowledge, these refer only to single samples. About a year ago Mr. J. H. B. Jenkins read an interesting paper on this subject (dealing, however, with only one sample of oil) before the London Section of our Society (1897, 195). I happen to have had the opportunity of examining a dozen samples of Japanese wood oil, representing some fairly large consignments. These were received at considerable intervals of time, and, I believe, from several different sources. Most of the samples were quite clear and of a pale or golden yellow colour, but a few were much darker and somewhat turbid in appearance. My results are recorded in the accompanying table, in which I have also included Mr. Jenkins' figures for the purpose of comparison. It will be observed that all my sp. gr. figures are distinctly higher than his, exceeding 0·940 in every case.

I determined the iodine absorption of four of the samples, and found the results to agree moderately well with Mr. Jenkins'; as, on the whole, do my KOH and free fatty acid numbers.

My bromine-thermal (Hehner) values are, however, some degrees higher than the figures which I have calculated from Mr. Jenkins' bromine-thermal-iodine value $\frac{133\cdot4}{5\cdot7} = 23\cdot4$ C. I think this must be due to some difference between our conditions of working, as all my figures are remarkably concordant. One gramme of oil was used in my determinations, in accordance with Mr. Hehner's original directions ("The Analyst," Vol. XX., page 146). Mr. Leonard Archbutt, in a paper dealing with this new and valuable method of testing oils and fats (this Journal, 1895, 310), suggests that only 0·5 grm. should be employed in the case of linseed oil and (presumably) of other oils, which give a considerable rise of temperature with bromine, but I myself have not found this modification to be necessary with linseed and wood oils. What I have termed the "oxygen absorption" figures were obtained by heating about 0·5 grm. of each sample on a watchglass for several hours at 100° C., with free access of air. When heated in this way "drying" takes place with extraordinary rapidity and with considerable increase in weight, but the resulting film compares unfavourably, in every point except colour, with the residue obtained from relatively slow-drying linseed oil. I may say, in passing, that wood oil "dries" more quickly than any other oil with which I am acquainted, and this property may possibly constitute an actual source of danger, especially if the oil be used *per se*.

I determined the unsaponifiable matter in three samples and found the proportion to be very small. It may be interesting to mention that, without taking any special precautions, I isolated about 8 per cent. of what appeared to be fairly pure glycerine from each of these three samples.

| —— | | 1. | 2. | 3. | 4. | 5. | 6. |
|---|---|---|---|---|---|---|---|
| Sp. gr. at 15·5° C. .................... | .. | 0·9426 | 0·9413 | 0·9420 | 0·9418 | 0·9425 | 0·9431 |
| KOH absorption...................... | Per Cent. | 19·28 | 19·07 | 19·36 | 19·25 | 19·61 | 19·36 |
| Free fatty acids (as oleic) ............ | „ | 5·05 | 3·55 | 5·30 | 4·80 | 2·95 | 3·05 |
| Bromine Thermal value (Hehner) .... | °C. | 27·1 | 26·7 | 27·3 | 27·6 | 26·9 | 26·8 |
| Iodine absorption (Hübl) ............ | Per Cent. | .. | 157·36 | 155·37 | .. | .. | .. |
| Bromine „ (gravimetric) ...... | „ | 93·57 | 93·14 | 96·10 | 104·30 | 98·44 | 100·86 |
| Oxygen „ „ ...... | „ | 7·17 | 5·57 | 6·48 | 6·06 | 4·71 | 5·66 |
| Unsaponifiable matter................ | „ | 0·52 | 0·49 | 0·69 | .. | .. | .. |
| Insoluble fatty acids.................. | „ | 96·66 | 96·30 | .. | .. | .. | .. |
| „ „ ............. Melting Point. | | 40° C. | 49·4° C. | .. | .. | .. | .. |
| „ „ .......... Solidifying Point. | | 37·2° C. | 37·1° C. | .. | .. | .. | .. |
| „ „ ........ Combining Weight. | | 333 | 327 | .. | .. | .. | .. |

| —— | | 7. | 8. | 9. | 10. | 11. | 12. | Jenkins. |
|---|---|---|---|---|---|---|---|---|
| Sp. gr. at 15·5° C. .................... | .. | 0·9432 | 0·9416 | 0·9425 | 0·9426 | 0·9423 | 0·9424 | 0·9385 |
| KOH absorption...................... | Per Cent. | 19·38 | 19·13 | 19·15 | 19·47 | 19·19 | 19·37 | 19·40 |
| Free fatty acids (as oleic) ............ | „ | 2·95 | 2·85 | 3·90 | 3·85 | 2·70 | 2·95 | 3·84 |
| Bromine Thermal value (Hehner) .... | °C. | 26·8 | 27·2 | 27·1 | 27·1 | 27·4 | 27·5 | 23·4 |
| Iodine absorption (Hubl.) ............ | Per Cent. | .. | .. | .. | 164·80 | 165·60 | .. | 165·70 |
| Bromine „ (gravimetric) ...... | „ | 100·73 | 100·70 | 97·80 | 98·60 | 96·58 | 93·87 | .. |
| Oxygen „ „ ...... | „ | 6·45 | 4·24 | 6·76 | 5·79 | 4·92 | 5·19 | .. |
| Unsaponifiable matter................ | „ | .. | .. | .. | .. | .. | .. | 0·44 |
| Insoluble fatty acids.................. | „ | .. | .. | .. | .. | .. | .. | 96·40 |
| „ „ ............. Melting Point. | | .. | .. | .. | .. | .. | .. | 37° C. |
| „ „ .......... Solidifying Point. | | .. | .. | .. | .. | .. | .. | 34° C. |
| „ „ ........ Combining Weight. | | .. | .. | .. | .. | .. | .. | .. |

## THE PROPORTION OF UNSAPONIFIABLE MATTER PRESENT IN BOILED LINSEED OIL.

BY ROWLAND WILLIAMS.

A SHORT time ago a well-known chemist sent me some figures which he had obtained in the analysis of a sample of boiled linseed oil, with a request that I would express my opinion as to whether they indicated adulteration with mineral oil.

He mentioned, *inter alia*, that the sample in question contained 12·50 per cent. of unsaponifiable matter (sp. gr. 0·898), which alone was sufficient to convince me that it must be sophisticated, most probably with mineral oil.

As he informed me that he had been unable to find any published figures bearing on this particular point, and as I also have not been able to meet with any such information in the works of reference which I have consulted, it occurred to me that it might perhaps prove useful to others if I were to state the results of my own experience in this direction.

The amount of unsaponifiable matter in *raw* linseed oil varies from about 0·8 to 1·3 per cent., being, according to my experience, generally rather more than one per cent. Thomson and Ballantyne (this Journal, 1891, 236) found from 1·06 to 1·23 per cent. in raw linseed oil from various sources.

The proportion of unsaponifiable matter in *boiled* linseed oil is, I believe, almost always decidedly higher than in *raw* oil. As the result of a large number of determinations in boiled linseed oil of different consistencies, I have found the amount of unsaponifiable matter to vary from about 1·3 to 2·3 per cent., being usually about two per cent., or, say, twice as much as is normally present in raw oil. Consequently, I am of opinion that a boiled linseed oil yielding more than 2·5 per cent. of unsaponifiable matter should be regarded with grave suspicion; in fact, personally, I should have no hesitation in condemning such a sample.

In the following table, in addition to the unsaponifiable matter, I have given a number of other figures, which I hope will be found useful by chemists who are only occasionally called upon to make analyses of boiled linseed oil, and who probably have not the same facilities as myself for obtaining reliable samples for comparisons.

The oils given in the table constitute a fairly complete series, ranging from the thinnest possible, through increasing stages of viscosity, to the solid oil used in the manufacture of linoleum.

| Sample No. | Consistency. | Unsaponifiable Matter. | KOH Absorption. | Bromine Absorption. | Free Fatty Acids (as Oleic). | Sp. Gr. at 15·5° C. |
|---|---|---|---|---|---|---|
| | | Per Cent. | Per Cent. | Per Cent. | Per Cent. | |
| 1 | Very thin | 2·34 | 18·22 | 100·00 | 4·45 | 0·947 |
| 2 | Thin | 1·27 | 18·09 | 97·55 | 3·55 | 0·948 |
| 3 | Thin | 2·11 | 17·95 | 94·92 | 6·35 | 0·961 |
| 4 | Stout | 2·01 | 18·93 | 62·28 | 9·90 | 0·972 |
| 5 | Stout | 2·04 | 18·56 | 67·24 | 10·50 | 0·982 |
| 6 | Very stout | 2·14 | 18·30 | 61·90 | 12·55 | 0·983 |
| 7 | Solid | 2·08 | 19·39 | 71·36 | 7·05 | Solid |

There is some essential point of difference between all the oils, such as duration or temperature of boiling, nature or proportion of "driers" used, &c.

It is worthy of note that the sp. gr. and the Koettstorfer figures are influenced to a considerable extent both by the amount of heating to which an oil is subjected, and also by the nature and the amount of "driers" employed, *e.g.*, a boiled oil, to which a very large quantity of lead oxide has been added, will usually have a *higher* sp. gr. and a *lower* KOH absorption than an oil containing only a small proportion of lead compounds.

It may be well to mention that the bromine absorption determinations were made by the gravimetric method described in "The Analyst," Vol. XX, page 50.

## A FRENCH VIEW OF GERMAN INDUSTRIES.

BY PROF. J. CAMPBELL BROWN.

A RECENT writer in a French magazine* attributes the prodigious industrial development of Germany to several causes. First, to the great increase in her population—from under 40 millions in 1870 to 52 millions to-day—which has made the country, once chiefly agricultural, one of the most powerful industrial communities in the world; this excess of population requires more support than the soil can furnish, and at the same time provides the labour necessary to exploit the mineral treasures of the country, manufacture products, and sell them abroad. At the last census, that of 1895, the number of persons employed in Prussia in chemical industries had increased 72 per cent., in engineering industries 57 per cent., and in constructive industries 80 per cent., while the agricultural population of Germany had decreased to 36 per cent., from 42 per cent. in 1882.

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* *Abstract of an article in the "Revue des Deux Mondes," February* 1898.—The information, and warning, and exhortations addressed to Frenchmen are equally important and applicable to Englishmen, who, although they have all the education and materials at their own doors, are too much in the habit of cutting short their education and neglecting to apply capital in painstaking investigations by skilled employees, and in the exploitation of many small items of manufacture, whose aggregate would be very great.

A second cause is political unity, now more complete than in the days of the Customs Union.

A potent influence has been the war of 1870 and its results.

The progress of industry has ameliorated the condition of the working classes, and has reacted in the direction of effecting some improvement in agricultural prospects, and by increasing material prosperity has lowered the menacing tone of the socialist democracy.

Industry is the scene of the triumph of science. It is difficult to find a more complete demonstration of this truth than is afforded by the chemical industries of Germany. Proceeding first from the laboratories of such savants as Liebig and Hofmann, it continues to prosper, thanks to the incessant flow from the universities of hundreds of chemists annually, some of whom enter the service of special industries, others follow a purely theoretical and scientific career, in constant communication with the former.

It was Liebig who initiated the new method. Not content with working with the aid of one or two assistants, as did the great French chemists, such as Gay-Lussac, Dumas, and others, he wished to found laboratories where a great many young people should work under his direction and form a school destined to spread over the country. The results of this system have been so apparent that there has been no need to plead with any German Government to develop it. Germany is covered with laboratories costing often 25,000*l*. to 50,000*l*. each, while maintenance costs annually an aggregate of 50,000*l*. Not only Prussia but States of much less importance and wealth vie with each other in furnishing with liberality the means whereby students may instruct themselves, and professors may multiply researches and demonstrations. For electrotechnics Professor Kittler at Darmstadt has asked for a special laboratory, which has been granted at a cost of 130,000*l*. Similarly at Berlin, Aix-la-Chapelle, and Hanover. There are at once schools which turn out young electricians and centres of scientific work in communication with the workers destined to apply their discoveries. Regular communication is kept up between the chemical works and the universities. Managers of works are always ready to place their appliances at the service of a savant who seeks to make an experiment, who on his part never refuses to examine a problem which they submit to him. This close collaboration between service and industry has given the most brilliant results. A single works at Ludwigshafen employs to-day more chemical engineers than all England has for the same branch. England has no school of chemical industry. France has not enough.

The Germans have, in their works, replaced foremen by doctors of science, who add to their technical knowledge a guarantee which has its value. Instead of blindly following routine, their science gives these foremen authority, and the workmen, who, in spite of socialism, have brought back from their regiment habits of discipline, look upon them as officers and the inspectors as subalterns. Many of the workmen belong to soldier clubs, which maintain amongst them ties of comradeship.

Laboratories of chemical works are centres of uninterrupted labour which follow various lines, often unforeseen and almost always fruitful. A crowd of young chemists, who have at their disposal in their laboratories incomparable appliances, pursue researches in a multitude of directions, which have led to great discoveries. Science solves new problems as fast as industry presents them.

Formerly, one depended upon the season or the state of the weather. At the present day, operations are carried on at any temperature required, and at pressures which may be as high as 150 atmospheres, by means of autoclaves. This method, one of prime importance, has been applied for the first time in France at the establishment of Poirrier at St. Denis. Everyone knows that modern industry has converted tar and oil into colours, perfumes, saccharine, powerful explosives, febrifuges, and the like. One might say that every day a new body is discovered; and such is the perfection of manufacturing processes that industry no longer fears the publicity which is involved in taking out a patent. "The true secret of successful manufacture," said Mr. Caro, an eminent contemporary German chemist, "consists of scientific system, a constant communication between the research laboratory and the works, a rational division of labour, a harmonious collaboration of all, from the highest to the lowest in the scale.

Since the first discovery of aniline colours in England in 1856, the mere enumeration of the colours, the drugs and other products manufactured from oil, would be a table of contents of a text-book on chemistry. More than 800 patents for tar products alone were taken out in Germany between 1877 and 1890.

Looking next at the means by which these vast manufactories enter into relations with customers, we see that it is not sufficient to send out commercial travellers to offer their products for sale; it is essential that the manufacturer should come into direct contact with those who buy, dyers and other trades. Men capable of directing their customers, understanding their desires, and teaching them how to employ the products, are an indispensable link. A mutual collaboration is established between them and the customers whom they advise, and who in their turn explain their constantly-changing requirements. About the middle of the century the colour industry seemed destined to be concentrated in England and in France, in which countries the early discoveries were made, but it is the Germans who have profited by these discoveries. The annual production of tar products in Europe at the present day represents a total of 5,000,000*l*. sterling, of which Germany produces 3,600,000*l*., Switzerland 640,000*l*., France 400,000*l*., and England 360,000*l*. Notwithstanding the supremacy which Germany has established for herself in the manufacture of colours, she does not go to sleep in the confidence which this pre-eminence might inspire. She labours incessantly to attain new improvements, knowing that patents soon become public property, and that industry will concentrate its attention upon a small number of essential colours. But she is aware of the strength which she derives from a powerful organisation, alike industrial and scientific, abundance of appliances, and capital. She knows that the soda industry is not so well developed in Germany as the colour industry; it is England who has vast plant and an established organisation, against which it is most difficult to contend; nevertheless, during the last 25 years she has made remarkable progress even in the soda industry, a progress attributed by Mr. Robert Hasenclever, in a report to the German Chemical Society in 1895, to a more rational system of work, a more stable Customs policy, the new patent law, and the co-operation of engineers. The law of 1884, establishing accident insurance for workmen, obliges each branch of industry to form an association with the special view of insurance, to divide the risks, and diminish the burden of expense. The result of these incorporated unions has been to develop, side by side with the union imposed by law, a voluntary association composed of the same members for the purpose of watching over the interests of their own branch of industry, collating statistics, and expressing their collective opinion regarding Customs, legislation, treaties of commerce, and similar laws. Groups of these industrial associations are combined under the name of "The Central Alliance of German Industries for the Development of National Labour." This alliance is a very powerful body.

The central bureau of each industry knows the details of the smallest manufactory of its speciality, and is able to publish precise statistics without revealing names, to supervise works and prescribe methods and rules for diminishing the risk of accidents which are rigidly enforced and loyally obeyed by those interested, because they are framed by their own delegates and not by the Government.

As a result of this double organisation we have side by side with the report of the official corporation, showing the increase in the number of persons insured, and stating the percentage of them who have been indemnified for accidents, another report, that of the voluntary association, showing the increase in the number of workshops engaged in chemical industries in 1895; 34 millions of days worked in place of 33 in the preceding year; workmen employed, 114,000 in place of 110,000; an increase from 98 millions of marks to 103 millions paid in wages, 927,000 tons exported of a value of 339 millions of marks against 280 millions of marks imported. The mean dividend of 95 works belonging to joint stock companies was 12·71 per cent. The manufacture of "heavy chemicals" alkaloids and acids was less prosperous as a whole, though that of soda and sulphuric acid gave very good returns. Pharmaceutical and photographic products have suffered by competition, while alcoholic preparations have benefited by new legislation. Organic acids have fallen in price by one-fifth owing to increased production. The manufacture of stearine has been maintained, that of soap and perfumes has increased especially for export. Tar colours show steady progress, and the mean dividend has grown from 10 per cent. in 1893 to 23½ per cent. in 1895. The manufacture of explosives continues to give brilliant results. The price of artificial lubricants has fallen so low that the return on the capital employed is below 4 per cent. The Alliance secured the inclusion of measures for the protection of manufacturing secrets in the law against unfair competition; it has taken an active part in legislation for the protection of industrial prosperity, and negotiated at great length with the imperial patent office on the subject of the trade-marks law. It has been concerned with the customs law of Spain, Italian imports, railway tariffs, and everything that affects the defence of chemical industry. Its congress has been occupied with the Paris Exhibition of 1900, and has unanimously pronounced against the sending of a commission of experts to develop commercial relations with China and Japan, but it has, on the other hand, nominated a commission for obtaining from the Prussian government the largest subsidies possible for the teaching of chemistry in the higher schools. The more we study the development of German industry the more convinced we become that the determining factor of its success has been not specially favourable conditions, but the method of scientific training which has raised up in that country a veritable army of chemists labouring without ceasing for the continual improvement of processes, in the search for new ideas, and in the perfecting alike of details and of the industry as a whole. It is to the fountain which fertilises this vast field that we must go if we wish to understand what is taking place amongst our neighbours, and draw therefrom the necessary conclusions.

Mr. Lauth, reporter on the section of chemical products in the Exposition Universelle de Paris, has recently demonstrated by unanswerable arguments that our safety lies in the reorganisation of the teaching of chemistry; in the erection of laboratories where our savants shall be occupied not only in the pursuit of their admirable discoveries, but in exploring their applications and training pupils whose minds will be directed towards this end. Let the universities give to French industry 600 young chemists every year, prepared as they are in Germany, and a transformation will be accomplished. Science, we cannot too often repeat, has been a source of incomparable riches. It was a pupil of Bunsen, Auer, who, by laboratory experiments and reasoning, arrived at the idea of the burner which bears his name, and which has made his own fortune and that of numerous companies.

The last revolution in metallurgy dates from Thomas's invention, which enables steel to be extracted from phosphatic iron ores, which the Bessemer process, an acid method, did not render available. The only considerable bed of phosphatic iron minerals in Europe is in Lorraine, with an extension into Luxemburg; the treaty of Frankfort has, unhappily, deprived us of part, but we still possess sufficient to feed our factories. The most southern part of the ore-bearing basin is at Neuves-Maisons, 20 miles south of Nancy, where the Châtillon Commentry Co. has erected large steel works. We are as well situated, and have as great advantages and equal chances compared with the Germans; why, then, have we only four great steel works, while the Germans have a score, several of them producing 400,000 tons of steel per annum each, against hardly one-fourth of that amount as a maximum in one of ours. It is necessary to recognise here as elsewhere that it is not material conditions which condemn us to a state of inferiority, but the want of initiative on the part of our manufacturers. The Germans do not wait till a want is expressed: they create the demand. For example, their production of iron beams reaches 800,000 tons per annum, of which 150,000 tons are exported; the French production is 230,000 tons, of which 30,000 tons are exported. It is evident that the difference in extent and population of the two countries does not account for the difference. In 1896 Germany exported 1,700,000 tons of iron, equal to the total French production of iron and steel.

Among modern industries the applications of electricity appears to be the one most full of promise. The Germans have come to the front with remarkable energy. It is quite surprising to see how a certain number of great houses or societies have, without making any essential discovery, profited by foreign discoveries and developed in 15 years an industry which is cited as a model for the whole world. Is it not strange to think that at home in Paris it is a Berlin company that has bought the Edison patents, and that we have not the courage to raise appliances similar to those which we cannot but admire at Berlin and elsewhere, and which our engineers are certainly capable of raising and directing with equal success. A list of German electrotechnical works, exclusive of urban electric supply companies and constructors of accumulators, shows a total share capital of 267 million marks (13,350,000*l.*) and loan capital of 89½ million marks (4,475,000*l.*). One of these companies employs 6,711 hands; it has constructed 4,000 dynamos and electro-motors, with a total power of 50 millions of watts. A smaller company with a capital of 22,500,000 marks has orders on its books for 62,000,000 marks, of which 36 millions are for Germany and 26 millions for abroad.

Let our financiers, then, set themselves to work; the *personnel* will not be wanting. It is for them to form companies with sufficient capital to inspire confidence in public authorities or in the manufacturers with whom they have to deal. Our neighbours have shown us by what combination of qualities they have made their undertakings prosperous; it is for us to adopt their principles and follow their example.

A visitor to the Berlin establishments would be surprised to see the appearance of health and strength in the majority of the workmen; comfortably installed in vast halls, cool in summer, well warmed in winter, where nothing has been spared to place each one in such conditions as to give the maximum effect to his labour. Once again the American adage is verified, "the highest salaries yield the cheapest work."

In other establishments in Germany, along with material progress, and the multiplication of appliances, and of works, there is a continual improvement of the lot of the workman. In five years the total salaries paid has risen from 106,000,000*l.* sterling to 134,800,000*l.*, an increase of 25 per cent.

The necessity of selling beyond the frontier part of what is produced on national territory is a source of disquietude to a certain school which deplores the evolution which makes Germany more industrial than agricultural and dependent on the foreigner for part of her food, for which she pays in machines, rails, textile, cloths, in short the produce of her industry. "Along with certain advantages, grave inconveniences attend the increase of the industrial population relatively to the total population, and the tendency of capital, always seeking to find its most remunerative employment, to draw hands from the country in ever-increasing numbers, produces a kind of progress which is nothing but a change in the things consumed, a kind of progress which is deplorable; the whole rests on an uncertain base. It is bad for a country to depend for its sustenance on foreign countries which could at any moment refuse to supply its needs."

Professor A. Wagner thinks that statistics show that the exportation of English products is decreasing in value and sometimes in total quantity. He is certain that in Germany the exportation of manufactured products is in several cases stationary or even retrograde, and suffers from the competition of Japan and the United States. It is easy to answer these fears by showing that even if there were not a compensating increase in the demand for every kind of labour, the change has enriched Germany much more rapidly than a simple development of agriculture would have done, and that the latter has proceeded side by side with industrial progress, and one ought not to be disquieted by that industrial extension which is a law of modern progress. Such anxiety would be justified, only if agriculture were imperilled in consequence of desertion of the country, and if there were actually insufficient hands for agricultural and forest labour. (Such desertion of the country has begun in some districts of Great Britain, and anxiety for the agricultural industry of Britain would not be unjustifiable.)

What would the growing population of Germany do if the national industry were to become stationary or recede? It certainly could not find relief by occupying itself in the fields. Iron, steel, and textile fabrics are hardly less indispensable to man than bread; the community which produces them in abundance is not inferior to that which purchases them. The economic ideal of a people is to be like the United States of North America, at once the purveyor to the rest of the world of cereals, petroleum, certain of the metals, and the producer of the majority of manufactured articles which it needs. But nations less favoured by nature may yet find in a just equilibrium between the two orders of production, agricultural and industrial, the means of securing a prosperous existence. This is the spectacle which contemporaneous Germany offers to the eyes of an impartial observer.

# London Section.

*Meeting held on Monday, March 14th*, 1898.

DR. R. MESSEL IN THE CHAIR.

The Chairman referred to the great loss which the Society had suffered by the death of Dr. F. Hurter, and moved the following resolution of condolence:—

"That the Members of the London Section of the Society of Chemical Industry, assembled at their monthly meeting, desire hereby to express their condolence and sympathy with Mrs. Hurter and family in the decease of the late Dr. Ferdinand Hurter, of whose ability as a man of science, and whose personal worth, this Section has a high appreciation."

Mr. David Howard, in seconding the resolution, said that in dealing with Dr. Hurter's memory it was difficult to speak with absolute impartiality, to eliminate the feeling of personal loss of a valued friend. But, even making allowance for that personal feeling, one might say with perfect confidence that the Society had lost one of the most eminent, one of the most thorough chemists it had been their fortune to know; one who brought the highest scientific knowledge to bear on his work, and applied it to practical problems with the most admirable skill.

The resolution was then put, and was carried unanimously.

## ALUMINIUM AND OTHER ELECTRO-CHEMICAL INDUSTRIES AT FOYERS.

BY R. W. WALLACE, Q.C.

Electrolysis is more within the scope of the chemist than the electrician, and in a few years electrical energy will be, if not predominant in the production of chemicals, enormously extended.

Let us commence by considering the arrangements for the production of electricity at Foyers.

Foyers was selected as the first power to be developed. It is the only installation of any considerable magnitude in the United Kingdom. It was carried out without an Act of Parliament, and with the entire agreement of the local authorities of the neighbourhood. The property and water rights were acquired privately. The operations necessary involved the deviations of old, and the making of a few new roads and bridges.

The extent of country over which the British Aluminium Company have acquired the water rights amounts to somewhat over 100 square miles.

It is situated on the south-east side of Loch Ness, on the Caledonian Canal, and sea vessels of large size can be berthed alongside the Company's wharf. The greater portion of the land is at a height from 600 to over 2,000 feet above Loch Ness, parallel to it, and some three miles inland, and at a height of some 700 feet runs a long valley containing Loch Garth and Loch Faraline. The waters from these lochs, as well as from other smaller ones, and from the several rivers, all converge in one large river, discharging over the Falls of Foyers; and as the annual rainfall, especially on the high ground, is very heavy, the power available is a large one. Our original scheme to utilise the great natural advantages of this district has since been carried out almost in its entirety; this was to intercept the water before it reached the upper falls, some three-quarters of a mile from Loch Ness, bore a tunnel through the solid rock to bring the water at a high level to the side of the hill which slopes to the shore of Loch Ness, and convey it down to the turbines which are situated on a low-lying level of land of considerable extent, which seems almost to have been provided by nature for the purpose. This alone would have only allowed the use of the bulk of the water in an intermittent way, which would have been most unsuitable for the purpose of making aluminium on a large scale by the electrolytic process, where it is of the first importance that the supply of electricity should be always very nearly the same. This was provided for by the construction of a very large reservoir in the valley already referred to. A stone-built dam was thrown across this valley at the southern end of Loch Garth, which forms a reservoir that is capable of holding about 1,000,000,000 gallons of water. This reservoir is some five miles long by three-quarters wide, and forms the two lochs already mentioned into one grand loch by covering the intermediate low ground. Proper penstocks are provided in the dam, so that when the water from the hills is insufficient these penstocks can be opened and the water from the reservoir used to any desired extent. As regards the further development, the nature of the ground offers many sites for storing the water at different levels, so that it can, as it were, be stepped down and made to give out its power at each step, doing duty many times over before it reaches the lock.

The company bought the freehold estate of Lower Foyers and Gorthlick, which includes the lochs already named and an area of some 12 square miles. Operations were commenced in 1895, the company doing their own work; and in the course of 12 months the tunnel was completed, the intake formed above the lower falls, six lines of cast-iron pipes laid from the end of the tunnel to the turbines below, a factory built, turbines and dynamos erected, an additional pier made at the side of the loch with a short line of railway to the works, and the necessary machinery, which had already been ordered, set to work. Since then the manufacture of aluminium has been carried on without interruption. The dam at the end of Loch Garth was not completed till last Christmas. Of course, under those circumstances, the full power of the five turbines provided could not be fully utilised last year, owing to the necessarily intermittent character of the water supply.

The present installation for electric power at the factory consists of five large turbines and dynamos, each of about 700 electrical horse-power. The turbines and dynamos are rotating on the same vertical shaft respectively at about 150 revolutions per minute; of course, a direct current is used, the process being an electrolytic one; each dynamo gives over 8,000 ampères, and therefore very large copper cables have to be used. Two more large turbines and dynamos of the same power are now being erected. There are also two other smaller turbines and dynamos for lighting and for working motors.

The result of these operations has been to produce electrical energy, including all costs and interest on capital, at less than one-third than the same could be produced by coal and steam. The first uses to which this energy has been applied is to the production of aluminium and carbide of calcium.

The true history of aluminium may be said to date from February 6th, 1854, when Henri St. Clair Deville read his first paper before the French Academy. His process was originally the same as that employed by Wöhler, but discovering that potassium was both dangerous to handle and extremely expensive to use—costing as it did about 20*s.* per ounce—he was led by Bunsen's good fortune in reducing magnesium chloride with the aid of the battery, to study the action of electricity in the case of aluminium. Bunsen himself also attacked the problem with considerable success, and by May, 1854, Deville was in a position to send five or six grammes of the metal to Liebig for examination and report. When it is considered that Deville was working with primary batteries, the only wonder is that he was able to prepare any metal at all. Deville, in fact, quickly saw that there was no hope of succeeding commercially by means of the then electrical power, and once more he returned to the use of an alkali metal. But he was compelled first to elaborate some reasonable method of obtaining his reducing agent; and, as the atomic weight of sodium is lower than that of potassium—that is to say, capable of separating a larger proportion of aluminium—he turned his attention to the production of sodium. By the beginning of 1855, Deville was ready to commence the regular manufacture of aluminium, which he did at a chemical factory at Javel, near Rouen. In 1856, proper works were erected at Glacière, a suburb of Paris; in the next year the plant had to be removed to Nanterre; and later on it was transferred to Salindres, where the operation has been conducted until within the last few years.

Deville's process may be divided into three stages: the preparation of metallic sodium; the conversion of the natural French bauxite into a double chloride of aluminium and sodium; and the final reduction to the metallic state. Within a space of two or three years—starting with sodium, which then cost 48*s.* per ounce, and with a metal which had hitherto only been seen in single grammes—Deville was able to put aluminium of respectable purity on the French market at 8*s.* per ounce.

In this country, at the commencement of 1855, Dr. Percy suggested that the mineral cryolite would be a very suitable ore for this metal, and that it might be employed without the tedious process necessary to purify bauxite and to convert it into chloride. Messrs. Dick and Smith accordingly tried a number of experiments, and at the Friday evening meeting of the Royal Institution held on March 30, 1855, Faraday exhibited the first specimen of aluminium actually reduced in England. About the end of 1858, F. W. Gerhard opened the first English aluminium works at Battersea, and by February, 1859, he was able to accept an offer to sell 500 ounces at 3*s.* 9*d.* per ounce. Unfortunately, although some trouble has been taken to clear up this matter of history, nothing can be learnt about Mr. Gerhard except that he made his own sodium, used the cryolite process, and ceased to manufacture about the middle of 1863. Messrs. Bell Brothers manufactured aluminium at Washington, near Newcastle-on-Tyne, from this date till 1874.

From 1874 to 1882, Messrs. A. R. Pechiney and Co., the proprietors of the Salindres works, had a monopoly. Webster, who in 1862 had made improvements in the manufacture of the double chloride of aluminium and sodium, organised the Aluminium Crown Metal Company at Birmingham in 1882. In 1886 and 1887 the Aluminium Company was formed to take over both the Webster and the Castner patents, and the works at Oldbury were started in July 1888. Mr. Castner's process, which has often been described, was a great improvement on that of Deville, and consisted in the substitution of a sort of carbide of iron for the charcoal used to split up the sodium salt. From this date it is almost impossible to sketch the history of aluminium in brief, new processes and patents appearing on every hand with almost inconceivable rapidity. In 1885 the Cowles patents were taken out. In 1886 those of Heroult and Hall were applied for; and Kleiner's electrical process was unsuccessfully inaugurated at Tyldesley, near Manchester. In 1887, Minet's electrical process and Netto's chemical method appeared on the scene. This process I had the opportunity of seeing at work at the excellent factory of the Alliance Aluminium Company at Newcastle on the occasion of a patent lawsuit between this and the Oldbury Company. In 1889 an attempt was made to start the electrolytic manufacture of aluminium at Patricroft with the aid of steam power, but the turbines of the Swiss and French factories soon rendered it abortive. Gradually the inferior processes succumbed, and at the present day the electrolytic method owned by the British Aluminium Company in this country stands absolutely without a rival.

There have been many statements made as to the production of aluminium by electrolysis in an aqueous solution; but after many experiments which I have made, I think I may say that those who made such statements were mistaken, as only a film or an alloy of the metal can be obtained. The cost is, of course, much greater than in the fused bath.

Only three compounds of aluminium are practically available for reduction to the metallic state—the oxide, the chloride, and the fluoride (*i.e.* cryolite)—for the sulphide, in spite of Bucherer's and Blackmore's endeavours, is still apparently far more troublesome to prepare than is warranted by its subsequent greater ease of decomposition. These compounds are capable of reduction in three different ways—by the action of sodium at a moderate temperature, by carbon at a very high one, and by electrolysis. Of all oxides except that of magnesium, and possibly calcium, alumina is the most difficult to break up. Baille and Féry's number for the heat of formation of 1 molecule of $Al_2O_3$ being 391,600 calories, while that of $Na_2O$ is only 99,760. This fact explains why some early experiments on the action of sodium upon aluminium oxide were not successful, and distinctly points to the utter impossibility of the process under any practicable conditions.

The following table of chemical equations, showing when calories are absorbed or evolved, will show this more clearly:—

Table I.

$Al_2Cl_6 + 6Na = Al_2 + 6NaCl$ evolves 264,180 cals.
$Al_2F_6 + 6Na = Al_2 + 6NaF$ evolves 106,200 cals.
$Al_2O_3 + 3C = Al_2 + 3CO$ absorbs 304,600 cals.
$Al_2O_3 + 6Na = Al_2 + 3Na_2O$ absorbs 92,320 cals.

It will be seen that, in the case of fluorides or chlorides, the addition of sodium will liberate aluminium with the evolution of considerable heat, whereas in the case of the oxides heat is absorbed.

The Deville-Castner process, accordingly, was very easy to carry out without external heat. When once the reaction between the sodium and the aluminium chloride had been started it proceeded smoothly enough, and the yield calculated upon the weight of the alkali metal taken was to all intents and purposes up to the theoretical. But it suffered under two fundamental disadvantages. For physical reasons the aluminium salt actually employed was the double chloride of sodium and aluminium—a body containing only 14 per cent. of metal—so that, apart from the expense of preparing it, a very large mass of unprofitable material had to be dealt with in the furnaces. Secondly, the bulk of the energy was derived from the sodium, an element which is always costly to produce, and which regarded as a source of energy alone is some six hundred times more expensive than coal. In fact, it has been shown that at Salindres in 1872 the cost of the coal and sodium amounted to nearly 50 per cent. of the total cost of the metal, which was then sold at about 60*s.* per lb. As aluminium is a trivalent metal, three molecules of sodium are needed to replace one of the former, or about two and a half times by weight, so that the expense for sodium alone in the Deville-Castner process was about 1*s.* 10*d.* per lb. of aluminium, or considerably more than the selling price of the metal in 1898. And as no further information has yet been vouchsafed about the method of reducing aluminium sulphide with molten iron outlined by Blackmore (this Journal, 219, 1897), it may be asserted that sodium is the only reducing agent which is available without the assistance of electricity in some form or other. Whence it follows that unless the alkali metal could be manufactured at roughly ½*d.* per lb. (which is absurd), no purely chemical method of winning aluminium yet foreshadowed has any hope of success.

A still further objection to the sodium process is based on the circumstances that during any reduction of aluminium in the presence of large amounts of sodium salts (except the fluoride, which is the most stable of those now being dealt with), the metal is liable to be contaminated with sodium, and this of course is the most deadly impurity it can possibly contain.

The actual decomposition of alumina by carbon alone has never yet been effected on a manufacturing scale. It seems quite impossible of achievement by chemical heat, that is to say, by the heat of combustion; and even in the Cowles furnace it was found advisable, if not necessary, to add some copper to the charge, in order that the heat, evolved by the union of the reduced aluminium with the excess of copper in the formation of the alloy aluminium bronze, might assist the electrical current. Nevertheless, the Cowles process was not, strictly speaking, an electrical one; it depended simply on the chemical decomposition of aluminium oxide by means of carbon, and the current of electricity was used solely as a source of heat; in a word, it was a method of electric smelting. Its leading disadvantage was that only alloys of aluminium were produced, and, moreover, there was no way of controlling the proportion between the metals they contained, so that each batch required to be analysed, then bulked with a number of other products of known composition, and the whole melted down once more.

Aluminium fluoride is the only compound that is suitable for electrolytic reduction in the fused state without the aid of a flux, but as it is not a natural mineral it would have to be specially prepared. Cryolite, even supposing it occurred free from all impurities, is not satisfactory, because the difference between the heats of formation of its two ingredients is too small to ensure the decomposition of aluminium unmixed with sodium. The theoretical voltage required for the decomposition of aluminium fluoride is 4·0, that of sodium fluoride 4·7, and a very small jump in the current would therefore throw down the alkali. But the theoretical voltage for decomposing alumina is only 2·8, so that it is not worth while to prepare a pure fluoride when the oxide, with the aid of a flux, is not only easier to reduce, but contains a larger percentage of metal. At first sight it might appear that the sulphide could be used with advantage, as the voltage required for its decomposition is only 0·9, but so far this does not appear to be the case. In fact, the economy in electricity, especially when this latter is produced by water-power, is more than counterbalanced by the less quantity of metal contained in the sulphide compared with the oxide, and by the extra cost and difficulty in the preparation of the aluminium sulphide.

Alumina itself is practically infusible, but it dissolves readily in several salts which melt at a reasonably low temperature. Of these only the fluorides of aluminium and sodium are of industrial value, and although the salt $Al_2F_6 2NaF$ has a slightly lower specific gravity in the molten state, thus allowing the reduced metal to sink rapidly to the bottom of the bath, $Al_2F_6 6NaF$ (cryolite) dissolves more alumina, and so is more suitable for employment. The high voltage required to decompose aluminium fluoride, to which reference has already been made, is manifestly an advantage when it is used only as a solvent. So long as the alumina itself is sufficiently pure for reduction the cryolite can be added in its natural condition, and as it suffers no decomposition there is little or no loss.

The Heroult cell, as used by the British Aluminium Company, consists of a carbon-lined iron box, itself forming the cathode; the anode is a bundle of carbon rods suspended within it, and reaching nearly to the bottom. The bath containing the molten cryolite is constantly fed with alumina as the aluminium is deposited, the reaction going on smoothly and continuously. The temperature of the bath has been measured to be between 750° C. and 850° C. A tension of 3 to 5 volts is ample to maintain the working temperature as well as to effect electrolysis. The current density is about 700 ampères per square foot of cathode surface, or about 8,000 ampères per cell. The practical yield is 1 lb. of metal per 12 E.H.P.-hours.

I shall now proceed to discuss the materials used at Foyers. The alumina is made at the British Aluminium Company's factory at Larne, and is obtained from bauxite won from the Company's mines.

A description of the Irish deposits may be found in a "Sketch of the Geology of Co. Antrim," by A. McHenry, published in the Proceedings of the Geologists' Association (**14**, 129), August 1895. The author writes: "Almost the whole surface of co. Antrim is covered by the sheets of basaltic lava that were poured out from numerous necks, vents, and fissures in tertiary times. These basaltic outflows have been mapped out into two distinct periods of volcanic activity—upper and lower. They are separated by a well-defined horizon of bole, lithomarge, bauxite, and pisolitic iron-ore deposits. . . . The zone of the iron-ore and bauxite can be traced in many places over the entire county."

The average composition of the ore is shown in the accompanying table; but it should be remarked that as its appearance is no guide to its purity, while the proportion of foreign matter is subject to very wide variations, samples have to be fully analysed before any new batch is taken into use.

Table II.

*Composition of Irish Bauxite after drying it at* 100° *C.*

$Al_2O_3$ from 59·6 to 33·2
$Fe_2O_3$ „ 2·0 „ 30·0
$SiO_2$ „ 7·0 „ 24·4

The balance being titanic acid and water of combination.

By glancing at a map, the great advantages possessed by the British Aluminium Company in the position of its several works will be instantly apparent. The Swiss factories have to obtain their ore from the south of France, the American from Georgia or Alabama; in both cases a long distance inland. The Company's Irish mines at Glenravel are close to the Belfast and Northern Counties Railway, only 35 miles distant from their alumina works at Larne Harbour, and a comparatively short direct sea passage brings the purified alumina without transhipments through the Caledonian Canal to the reducing plant at Foyers on Loch Ness.

The process originally employed by Deville for obtaining pure alumina from bauxite consisted in heating the mineral with sodium carbonate in a furnace till the reaction was complete, and the mass no longer effervesced on addition of acid. The residue was extracted with water, the solution filtered from the insoluble oxide of iron, and finally decomposed by a current of carbonic acid gas. Later, on in 1889, Dr. Bayer, who still at that time fused the bauxite with the sodium carbonate, discovered that it was possible to precipitate a considerable proportion of the alumina contained in a solution of sodium aluminate by agitating it with a small quantity of undissolved oxide, the result of a previous operation. In this manner some 70 per cent. of the alumina combined with the alkali is recovered, corresponding to a change in constitution of the aluminate from $Al_2O_3Na_2O$, or $Al_2O_32Na_2O$, to $Al_2O_36Na_2O$. Further experiments soon showed that the ore could be attacked without the assistance of furnaces, simply by heating it with caustic soda solution (specific gravity 1·45) under a pressure of 70 or 80 lb. per square inch, and then the advantages of Bayer's method of spontaneous precipitation became more evident, for instead of having to evaporate all the waste liquors to dryness before they could be causticised, and thus rendered available for use over again, it was only necessary to concentrate them to their original density, because they already contained sufficient free alkali to attack fresh bauxite. The whole process (which has been fully described by Mr. Sutherland, our manager at Larne, before the Institution of Mechanical Engineers, and abstracted in your Journal for October 31st, 1896), is therefore continuous; and the only by-product is a certain quantity of red residue containing chiefly the ferric oxide and silica of the natural ore. The washed precipitate of alumina is dried in a gas-fired furnace, and shipped to Foyers in iron drums.

The cheap production of pure alumina is a matter of great importance, and I suggest to chemists the matter as a subject worthy of investigation. As an example of what may be done I may mention a process which has been proposed by my old friend Mr. Claus, with whom I worked many years ago.

The first stage of this process is a modification of Hargreaves' method of producing sulphate of soda. Claus mixes bauxite and common salt in suitable proportions, and subjects this mixture, in the form of hard balls, to the action of sulphurous acid air and steam. The conversion of the salt into sulphate of soda, with evolution of hydrochloric acid, is facilitated by the bauxite, which renders the balls more porous and permits the gases to act on the salt more rapidly than when salt alone is used. When this reaction is complete the balls are exposed in the same furnace to the action of water-gas, which reduces the sulphate with formation of sulphuretted hydrogen and aluminate of soda. The silica in the bauxite forms an insoluble compound with some of the soda and alumina. The soluble aluminate of soda is decomposed by carbonic acid into alumina and carbonate of soda. Thus hydrochloric acid and carbonate of soda are obtained as by-products, the sulphuretted hydrogen of course being burned to sulphurous acid for decomposing fresh quantities of the salt or converted into free sulphur.

The next material used at Foyers is carbon. Theoretically, two parts of carbon are consumed for every three parts of aluminium deposited, but in practice the anodes are used up at an equal rate, and as all the impurities they contain must fall into the bath it is necessary that the material shall be exceptionally pure. It is equally indispensable that they shall not disintegrate under the dense current (about 35 ampères per square inch of anode area) employed, or else short-circuiting, and considerable waste of energy, will ensue; and seeing that such qualities of carbon as are perfectly suitable for lighting electrodes are not *ipso facto* adapted for commercial electrolysis, the British Aluminium Company has erected its own plant for the manufacture of carbons at Greenock. Just as the company's works at Foyers for the manufacture of aluminium is the first instance of water-power being made use of on the large scale within the United Kingdom, so this factory at Greenock is the first established in Great Britain for the preparation of large electrolytic carbons; and besides those needed to supply its own requirements the company is making electrodes for the Cyanide Company and the Acetylene Illuminating Company, for the manufacture of calcium carbide, while experiments are now being tried at Mr. Castner's request with the view of making such as are wanted in the Castner-Kellner sodium process, which at present have to be imported from abroad.

The only other material at present used at this factory is cryolite, which comes from Greenland, but arrangements are now being made for manufacturing, at the Larne factory, an artificial fluoride to be used as solvent.

The aluminium produced at Foyers is carried to Milton, in Staffordshire, to be worked up into rods, bars, &c. A new works is also about to be erected on the Clyde, at Greenock, for similar purposes.

The aluminium of commerce is practically pure, but many consumers prefer a lower grade than this, *viz.*, one with 98·5 to 99·0 per cent. of Al, as they find that this 1·0 per cent. of impurity, which consists mostly of iron, makes the metal appreciably tougher. When chemically pure aluminium has a perfectly silver-white colour without the bluish or violet tinge characteristic of the ordinary product. After exposure to damp for a certain time, a thin, almost invisible film of oxide forms on the surface, which, however, perceptibly darkens the colour of the metal, while affording an absolute protection from further attack.

Pure aluminium is somewhat harder than tin, or about 2·5 on the usual scale, but after rolling it becomes sensibly harder still. The specific gravity of cast aluminium is 2·583 at 4° C., but the commercial metal is generally a trifle heavier; it is increased by rolling or hammering to 2·688. The density of most of the alloys is higher than the figures calculated from their composition, which proves that much contraction takes place during their formation, and that they are, to some extent at least, chemical compounds. In fact, unless this proposition be admitted, it is impossible to explain how the admixture of two such comparatively weak metals as aluminium and copper should produce alloys which are comparable for hardness and strength with the best quality steels, as the annexed table shows.

TABLE III.

*Tests of Aluminium Bronzes.*

| Mark. | Breaking Load. | Elongation. |
|---|---|---|
| | Tons per sq. in. | Per Cent. |
| R. 4. Maximum | 43·7 | 13·00 |
| Minimum | 39·6 | 6·50 |
| Mean | 41·8 | 9·75 |
| R. 3. Maximum | 36·6 | 35·00 |
| Minimum | 30·8 | 15·50 |
| Mean | 34·0 | 23·30 |

The melting point of aluminium, as determined by Heycock and Neville with the aid of a platinum-resistance pyrometer (Trans. Chem. Soc. 1895, **67**, 187), or, more strictly speaking, its freezing point, is 654·5° C. Cowles states that it volatilises slightly in the electric furnace.

Aluminium is very ductile and malleable, standing third on the list for malleability, and sixth for the former property; sheets of $\frac{1}{40000}$ inch thickness having been prepared, while wire can be drawn down to $\frac{1}{250}$ inch in diameter. Its tensile strength, when cast, may be taken at from 6 to 8 tons per square inch, increased by rolling, &c., to 10, 12, or even occasionally 16 tons; many of its light alloys testing as high as 19 or 22 tons per square inch, with an elongation of 5 or 10 per cent. Weight for weight aluminium may be said to be equal to steel of 35 tons tensile strength, while the alloys are of course much stronger.

Lord Kelvin's latest results for the electrical resistance of aluminium (99·69 per cent. pure) show $6\cdot997 \times 10^{-6}$ Board of Trade ohms per gramme-length (*i.e.*, for a length of wire weighing 1 gramme) at 0° C.; or 2·58 microhms per cubic centimetre. Taking Dewar's value for pure copper ($1\cdot562 \times 10^{-6}$ Board of Trade ohms at 0° C.), the relative resistance of the aluminium becomes 165·3 per cent.; that is to say, its conductivity volume for volume is 60·5 per cent., and weight for weight double that of copper.

From the chemical point of view aluminium is specially valuable owing to its resistance to air, moist or dry, water, and most acids except hydrochloric. Alkalis, however, attack it readily. It is not blackened by sulphuretted hydrogen, which gives it an advantage over silver for many purposes. Organic acids attack it to an almost inappreciable extent, the presence of common salt, as in most cooking operations, slightly increasing their action; but after a saucepan of other appliance has been in use for some time it becomes covered with a hard insoluble skin which prevents any further dissolution of the metal. Moreover, the compounds of aluminium are perfectly harmless to the animal system, so that the metal is better suited than any other for the manufacture of culinary utensils. It is also used with much success in surgery; tubes of the pure metal having been inserted in the human body for long periods without perceptible corrosion.

To show in what a marvellous way the aluminium industry has developed within a short period, it is almost enough to say that the total output of the whole world for the year 1886 is estimated to have been 5 tons.

The following table shows at a glance the remarkable fall in price and the consequent increase of consumption during the last seven years.

TABLE IV.

*Annual make of Aluminium, and Price per Ton.*

| Year. | Tons per Annum. | Price per Ton. |
|---|---|---|
| | | £ |
| 1890 | 40 | 1,083 |
| 1891 | 200 | 504 |
| 1892 | 300 | 308 |
| 1893 | 530 | 298 |
| 1894 | 1,200 | 186 |
| 1895 | 1.800 | 160 |
| 1896 | 2,000 | 155 |
| 1897 | 2,500 | 148 |

The selling price is now cheaper, bulk for bulk, than that of copper or tin.

The chief industrial uses of aluminium fall into three general classes: it may be employed alone, as an ingredient in alloys, and as a metallurgical reagent in the purification of other metals. From the fact that alumina is the second or third most difficult oxide to reduce, it follows that aluminium itself is capable of extracting oxygen from almost all other oxides. In the purely scientific direction, Moissan's recently published work is a brilliant example of this. As is well known, he has succeeded, by the use of aluminium filings at a moderate temperature, in splitting up many of the most refractory oxides, and in recovering several of the metallic elements which had previously been considered extremely rare. He has been able to produce alloys of aluminium with nickel, molybdenum, tungsten, uranium, and titanium; one in particular containing 75 per cent. of tungsten being formed without difficulty, although it was so infusible that it only remained liquid by reason of the enormous amount of heat liberated by the reaction. Aluminium is very serviceable in the manufacture of ordinary brass or bronze, when used in such small quantities (1 per cent. or even less), that it does not appreciably affect the mechanical or chemical properties of the finished alloys, and hardly appears in their analysis, yet it greatly improves them, removing the traces of oxygen from the copper and yielding sounder and stronger castings. But aluminium is still more advantageously adopted in foundry work and in making steel. By the addition of a "pill," in the proportion of one part per 20,000 or 25,000 to the molten steel, the suspended or dissolved oxide of iron, which prevents the metal from being perfectly fluid and thus hinders the evolution of the gases, is instantly reduced; alumina, which immediately rises to the surface, being formed, so that when the steel is run into the moulds it stops boiling within a few seconds, and the ingots turn out absolutely solid to the top. The saving in wasters or spongy castings, both of iron and steel, is very great. In all cases it more than compensates for the relatively minute cost of the aluminium, and in some large foundries the scrap has been reduced by about 90 per cent.

It is not intended on this occasion to deal fully with the question of aluminium alloys. This is a subject which, I hope, will be taken up by metallurgical chemists. Knowledge on this subject is still far from perfect, yet even now the matter deserves more voluminous treatment than can be undertaken in the present paper. It has been found, however, among binary alloys especially, that as the proportion of the two ingredients becomes more and more equal, the products lose their valuable qualities, and therefore the industrial alloys may be sharply separated into two kinds; one containing 90 to 99 per cent. of aluminium with 10 to 1 per cent., or even less, of some other metal or metals, the other containing 1 to 10 per cent. of aluminium and 99 to 90 per cent. of the other components. In this manner they can be regarded either as pure aluminium strengthened by the addition of some other substance, or as one of the older metals improved physically and chemically by the introduction of aluminium.

As an individual metal the use of aluminium is growing day by day. A large amount of the metal is being consumed in the construction of apparatus for chemical works; amongst others, in the glue, confectionery, printing, pharmaceutical, oil, rubber, and varnish trades, aluminium is found very valuable for boiling pans, storage vessels, &c.

In most cases, where brass or copper is at present used, there aluminium may be substituted, with the double advantage of a reduction of two-thirds in the weight of the finished article, and the total absence of tarnishing and corrosion.

When dead weight has to be moved about, in cycles and carriages, in military appliances, in chemical plant and cooking apparatus, in ships' fittings, in launches and in machinery, aluminium or one of its alloys will be found speedily to supplant the heavier metals.

In a word, this remarkable quality of lightness places aluminium in a quite exceptional position to other metals; and now that it is brought down to a price comparable with copper or brass, it will take its permanent place with other metals in the arts.

The production of a new metal and its manufacture in a commercial way is not the only triumph of electro-chemistry. Carbide of calcium is also now made at Foyers. I had intended to go fully into this matter to-night, but the exhaustive paper read by Mr. Henry Fowler at the Institute of Civil Engineers last Tuesday makes it unnecessary for me to say anything beyond the fact that the first carbide made in this country was made with steam power by the Acetylene Illuminating Company, who soon found that it would be impossible to produce it commercially in this manner. Accordingly the company came to us for water power, and during the last year they have produced carbide of calcium at a paying price.

These are the only electro-chemical processes carried on at present at Foyers, but there are many other processes that could be equally well carried on in the same way as they are at present done in other countries. I might name the production of cyanides, chlorate of potash, sodium, caustic soda, and carborundum, not to mention phosphorus, the manufacture of which has left this country to go to Niagara. These and other processes require for their commercial success the use of cheap electrical energy. The cost of the power to produce this energy is the all-important factor, and our experience, gained from the development of the water power at Foyers, proves to us that this power costs but a fraction of that produced from coal or any other known source.

### Discussion.

Mr. Claude Vautin had listened with the utmost interest to the paper, and would be glad if the author would supplement it by stating what proportion the cost of electricity bore to the total cost of producing aluminium at Foyers. He trusted that the answer would enable him to show foreign inquirers that the metal could be manufactured as cheaply in England as in the United States. He had lately inspected a process based on the production of pure alumina from felspar, at a cost of from 3*l*. to 4*l*. per ton. Referring to that portion of the paper which reviewed the various aluminium processes, he confirmed the author's view that the only chemical process which was at all likely to compete with the electrolytic process was Grabau's process of reducing aluminium fluoride by means of metallic sodium. The reaction in this instance went on very smoothly without the application of any external heat, and the aluminium produced was practically free from metallic sodium, which was not the case when the chloride was reduced by the same reagent. Of course the success of such a process depended upon the cheapness of metallic sodium, and he understood that by a new process sodium would in all probability be produced at a cost not exceeding 1*d*. per lb. The great advantage of such a chemical process lay in the fact that fluoride of aluminium could be produced at a much less cost than pure alumina. Referring to the remarks of the author as to the production of calcic oxide, he could state that metallic aluminium would reduce calcic oxide; and, if he might be allowed to refer to his own work on the subject, he would remind the meeting that some three or four years ago he had demonstrated that such oxides as those of chromium, tungsten, vanadium, &c., could be reduced by means of metallic aluminium, and that at the present moment considerable quantities of metallic aluminium were used for the production of pure chromium in Germany. He was sure that there was a considerable field for the use of metallic aluminium in reduction processes. He would also like to know whether the company had any horse-power to spare at Foyers, as, if so, one or two industries might be retained in England which at present seemed likely to drift to the other side of the Atlantic.

Mr. C. C. Hutchinson agreed with the view expressed by the author that the field for the use of aluminium in the arts was a very wide one, and not the narrowest part of that field would be the production of the many alloys of the metal which possessed the remarkable properties pointed out. He did not, however, share the author's view that the production of these alloys, *e.g.*, the alloy of copper and aluminium, was the result of chemical combination of the two metals. Personally, he regarded the alloy of aluminium, with copper, and in a lesser degree that with silver, as one of the most difficult things to obtain as an alloy. The popular idea that such an alloy could be obtained by merely mixing and melting the metals was quite a fallacy. There was always a large amount of segregation, and the upper and lower portions of such alloys would differ greatly, not only in composition, but in the degree of crystallisation, and very materially in strength and physical properties. This difficulty was usually attempted to be overcome in a rough-and-ready way by re-melting and re-casting the alloy over and over again, and ten or twelve times was the usual number of these meltings to obtain a strong alloy. His own observation of this crude method of working showed that the segregation and want of combination was due to the two metals not being brought into sufficiently intimate contact during the melting. His own method of working to correct this was to granulate the metals in the well-known form of "bean-shot copper" by pouring them in a molten state into water. If in that state they were then mixed well together, and melted well together, in a carbon or plumbago crucible, the result was, that three or four meltings yielded a stronger and better alloy than ten meltings in the ordinary way, and he found from extensive experience that the difficulty of obtaining a sound alloy of even composition and strength was greatly overcome. Mr. Wallace had referred to the use of aluminium in the production of steel; he would mention another large and important industry in which its value was not sufficiently recognised, the production of ordinary iron castings. When he was engaged in that industry he found it highly advantageous and economical to use aluminium, even at a price as high as 7*s.* 6*d.* per lb., as it enabled a founder to produce much finer and better castings than otherwise was the case. The ordinary iron-founder, however, had much to learn before he would be able to benefit from the use of aluminium. The applications of aluminium to the production of articles of everyday use might, however, be carried too far in certain directions. For instance, it was obvious that it was not a suitable material for articles where mass was a prime feature, as it obviously lacked the weight necessary to give the required result. In one instance he had found out to his cost, when he attempted to construct the valves of fast-running air-compressing engines of an alloy of aluminium and silver, an alloy which, by the way, deserved attention for many purposes. The result was that the valves were so light that they failed to close upon their seats. He believed, however, that the more the industrial applications of aluminium were brought before those who could employ it the wider would the field of application become, and when more intelligence was brought to bear on the solution of these problems the metal would be used to a very large extent, and the figures given by the author in Table IV., the results of which he could amply confirm, would be thrown entirely into the shade.

Mr. O. Guttmann, referring to the author's statement that the Irish bauxite was cheaper because of the smaller distance it had to be carried, wished to inquire whether other well-known European deposits did not contain bauxite richer in alumina and poorer in silica than those of Ireland. In the diagrams of the Larne Factory he noticed some cellulose filters. He would be glad to know if the cellulose was such as was used in the manufacture of paper, or wood shavings. He would also like to know the cost of plant per horse-power developed, so as to compare it with the cost of plant for steam and water power. At present they had only Mr. Wallace's statement that the cost of power was only a fraction of that for producing it from coal. It had been stated at a meeting of the Institute of Civil Engineers that at the present price of coal in this country it was possible near the pits to produce power cheaper than by water. He would like to know whether the works at Foyers had been so designed and worked as to upset this view. To the long list of industrial uses for aluminium given by the author he would like to add some of special interest to chemical industry, which he had come across lately, namely, for the construction of vessels for nitrating cellulose, tubes for pumping nitric acid of as low as 1·4 sp. gr., and vessels for storage of mixed acids or strong nitric acid.

Mr. Otto Hehner wished to utter a word of warning against the use of aluminium vessels for the storing of nitric acid. Recent careful experiments by Stillman (J. Amer. Chem. Soc., Vol. 19, p. 711) had shown that under some circumstances aluminium is rapidly and powerfully attacked by nitric acid. Possibly the question of absolute purity might have something to do with it; but in the case he referred to the metal was pure commercial aluminium, containing 99·6 per cent. of aluminium.

Mr. L. Gaster thought the most interesting question suggested by the paper was the relative cost per horse-power produced as compared with the cost of producing the same power from coal. Mr. Steiger had recently read a paper in which he showed that in dealing with this natural source of power it was not necessary to have a very great fall of water. He thought that more advantage might be taken of the natural resources of the country to produce electricity and send it to other parts where these stores of power did not exist.

Mr. T. L. Cabot said that it might be interesting to the meeting to know that the Pittsburg Company obtained coal slack at 65 cents per ton, and that it took about 200 lb. of slack for every lb. of alumina made, which would be equal to about 6 cents per pound of alumina. He would be glad to know what the figures R 4 and R 3 on the diagram No. 3 meant.

Dr. O. J. Steinhart thought it curious and interesting to note that the aluminium works at Neuhausen were increasing the production of calcium carbide at the cost of their aluminium production for the present. On the other hand, it was to be noted that as far as chemical electrolysis was concerned, processes based upon thermo-electrical principles were the leading processes. There were several instances where other processes dealing with electrolytes in solution had succeeded, but on the whole, methods in which fused electrolytes were employed were found to be better. The manufacture of the carbons was a most important matter, especially from the point of view of the life of the carbon; the use of electric-light carbons being out of the question. He would be glad of any further information the author could give as to the proportion which the carbon consumption bore to the production of aluminium; and also as to the method of manufacturing them. He would also like to know whether the heat used in their manufacture was applied by a muffle furnace, or if the fuel gases passed direct over the carbons.

Mr. R. W. Wallace, in replying, said that he was glad Mr. Vautin had taken part in the discussion, because he wanted to hear from him whether or not he had done anything more as to the reduction of aluminium sulphide. If he had not, he could only express the hope that he would continue his researches on that subject in the near future. The process at Krupp's works to which reference had been made was a very interesting one. The great difficulty was to start the reaction. He understood that they had altered their procedure and now started it with a barium fuze which set the whole process at work, and that the liquefaction then proceeded rapidly without application of external heat. As to iron and steel founders, he quite agreed with Mr. Hutchinson, and he could only understand their continuing to employ the imperfect methods now in use on the ground that they had much capital invested in them. Unless more intelligence were brought to bear on arts and manufactures in this country trade would gradually drift from us to Germany and elsewhere. He hoped that chemists would secure more influence than they had hitherto done over the other arts. As to the cost of horse-power at Foyers, roughly speaking, the cost per horse-power was about 30*s.* per annum, exclusive of interest on capital. They had power to spare both there and in other parts of the country, and would be glad to negotiate with customers having processes which were not objectionable. Mr. Guttmann had inquired as to the purity of the Irish bauxite compared with those of France. He considered that the French bauxites were more regular and contained less silica than the Irish; but by careful selection of the samples good results could be got from the Irish products. Mr. Vautin's statement that alumina could be produced at 3*l.* or 4*l.* per ton was very interesting to him, seeing that his company's production cost about 12*l.* per ton, which implied a cost of 24*l.* per ton for raw material for aluminium. In reply to Mr. Guttmann's question about the cellulose filters, he was sorry to say they were of not much use for the purpose owing to their getting clogged; they therefore preferred to rely on precipitation and the ordinary filter presses. Mr. Gaster had referred to the cost of producing electrical energy from coal and steam power. Prof. Kennedy (a most accurate tester) had put the lowest price for producing it in Scotland (where coal cost 3*s.* 6*d.* per ton) at 9*l.* per horse power per annum. He doubted if it could be done at the price, all things considered. Referring to Mr. Hehner's remarks, his own experience in working with aluminium was that nitric acid did not act upon it at all; in fact he had hitherto regarded the action of nitric acid on a sample as a proof that it was not pure aluminium. He feared therefore that Mr. Hehner had experimented with something that was not pure aluminium. He spoke with reserve because Mr. Hehner had said "under certain conditions," which conditions were not stated. With regard to the consumption of carbon he had stated that the theoretical consumption was two tons of carbon to three tons of aluminium; but in practice the carbon used was about equal to the aluminium. The excess of carbon went chiefly into the cryolite, was skimmed off and re-used. Of course it was very important that the carbons used should be as pure as possible. Under the present system of working the gases entered into the furnace, and that method answered perfectly well. The terms R 4 and R 3 on Table 3 had no cabalistic significance, but simply indicated two classes of alloys. The alloys on Table III. varied from 90 of copper to 99 of copper and 1 of aluminum to 10 of aluminium; and the elongation varied very much in different samples. The ratios had not been calculated out, and it would be very difficult to do so.

A Member inquired if any fluorine were given off.

Mr. Wallace replied that none should be given off; and if the voltage were kept down to three to five volts none would be. If the voltage were increased so as to get near the decomposing point of the fluoride, some fluoride would come off.

*Meeting held on Monday, April 4th, 1898.*

DR. R. MESSEL IN THE CHAIR.

## A NEW SYNTHESIS OF GUAIACOL.

BY WATSON SMITH.

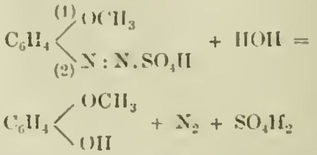
Guaiacol is an important constituent of wood-tar creosote in which it occurs inextricably mixed up with other substances of a phenolic nature. From these it has been found impossible to separate it in the pure state.

For pharmaceutical purposes when required pure, it has been prepared by the method of Gorup-Besanez, by heating together equivalent weights of pyrocatechol, potassium methyl sulphate, and caustic potash—

$$C_6H_4(OH)_2(1.2) + SO_2(OK)(OCH_3) + KHO =$$
$$C_6H_4(OH)(OCH_3)(1:2) + K_2SO_4 + H_2O.$$

Guaiacol may be thus obtained as a colourless, crystalline substance melting between 28 and 33° C. In this state of purity according to the Extra Pharmacopœia of 1895, it is to be preferred for medicinal use to any of the liquid guaiacols, which contain phenolic homologues. I am not prepared to say what the yield by the pyrocatechol process, is, nor what the expense may be, but the process I wish to bring before you looks very promising as regards yield, and certainly, to say the least of it, does not stand behind the pyrocatechol process, in point of cost. The new synthetic process was invented by an old German member of our Society, Dr. W. Kalle, of Biebrich-on-the-Rhine, and he has sent me this beautiful specimen of his synthetic guaiacol to exhibit to you. The raw material Dr. Kalle employs is o-anisidine, $C_6H_4(OCH_3)(NH_2)$, and this substance is largely used in the preparation of anisidine dyestuffs. You will find an abstract of Kalle's patent (Eng. Pat. 7233 of 1897) in this Journal, 1898, 269.

The interesting point is, that to prepare guaiacol from *o*-anisidine, the inventor makes use of the diazo-reaction, which is one not always remarkable for giving good yields as Kalle uses it. He overcomes the difficulty involved, in the tendency of the reaction to run into side-issues, in the following interesting manner. The ortho-anisidine is diazotised in presence of an excess of dilute sulphuric acid, after which the diazo solution is run into a still, containing sulphuric acid of greater strength, to which a considerable quantity of anhydrous sulphate of soda (salt-cake), has been added, and which is heated up to a temperature of 135° to 140° C. Thus as soon as the guaiacol is formed by the decomposing action of the hot sulphuric acid on the diazotised *o*-anisidine, it is vaporised and carried over by the steam, to the receiver. The reaction is—



$$C_6H_4\begin{cases}^{(1)}OCH_3\\ _{(2)}N:N.SO_4H\end{cases} + HOH =$$
$$C_6H_4\begin{cases}OCH_3\\ OH\end{cases} + N_2 + SO_4H_2$$

To the possible inquiry as to what technological importance attaches to guaiacol, I may briefly state that it is of extreme value in the treatment of pulmonary tuberculosis. It also forms the raw material for the preparation of guaiacol carbonate $CO(OC_6H_4OCH_3)_2$, which in its physiological action is said to resemble salol, and also for guaicol carboxylic acid $C_6H_3(OCH_3)(OH)CO.OH$, which is used as an anti-rheumatic. Its sodium salt is used as a substitute for sodium salicylate. In the most recent issue of the *J. Pharm. et de Chim.* 7, [7], 324, André describes a sulphonated guaiacol obtained by treating guaiacol with pure concentrated sulphuric acid (equal weights of each material), avoiding sudden rises of temperature, and letting the mixture stand for 18 hours, so that the reaction may be completed. The sulphonic acid, $C_6H_3(OCH_3)(OH)(SO_2OH)$, thus formed is neutralised by calcium carbonate, and the purified calcium salt is termed "guaiacyl." It is said to have been found useful as a local anæsthetic, being in this respect nearly equal to cocaine.

### Discussion.

Mr. Otto Hehner inquired how the guaiacol was obtained in a state of purity from the distillate.

Mr. A. R. Ling considered that the subject of Mr. Watson Smith's communication was one of peculiar interest to the Society, as it furnished an instance of the technical applications of the modern developments of organic chemistry, of which there were so many brilliant examples in Germany, but which were apt to be neglected in this country. The diazo-reaction, discovered by Griess, was one which often figured in chemical patent literature, and it still presented numerous possibilities to the chemical technologist. It was, however, a very subtle reaction, and its applications required the exercise of high chemical skill and ingenuity in order to determine the necessary conditions to insure success.

Dr. S. Rideal was surprised to hear that the calcium sulphonate from guaiacol was suggested as an anaesthetic and he thought that it was important that one should have fuller information before accepting such a statement. Any preparation of pure guaiacol should command a ready sale in England, as it was now very generally used for the treatment of phthisis.

Dr. F. B. Power expected that the new product before them would replace the crude and impure guaiacol of commerce. He believed that the first specimen of synthetic guaiacol in crystalline form was shown at the Chicago Exposition in 1893, and was of French manufacture. Since that time it had come into general use for pulmonary tuberculosis. More recent experience, however, had shown that it was not so valuable for that complaint as the guaiacol carbonate, which is now consequently more generally used. He would point out that there was a marked peculiarity about the synthetically prepared guaiacol, namely, the difficulty of obtaining it in crystalline form after it had been once liquefied. If, however, a very small fragment of the solid guaiacol were added to the solution the whole would readily solidify to a crystalline mass.

The Chairman inquired what percentage of real guaiacol was contained in guaiacol capsules.

Mr. W. Lloyd Williams said that he was in a position to answer that question, having examined a number of samples, and he found that the percentage of guaiacol varied between 60 and 90.

Mr. Watson Smith, in reply to Mr. Hehner's question, said that according to the patent specification the guaiacol was separated from the aqueous distillate by the addition of common salt, in which solution guaiacol is insoluble, and subsequently shaking with ether. With regard to Dr. Power's remarks on the superfusion of guaiacol, he would point out that the action of this body in that respect closely resembled that of carbolic acid; and the manufacturers of pure phenol took advantage of the property to induce further growths of the crystallised product from "carbolic oils."

He could sympathise with Dr. Rideal's feeling of astonishment, that by sulphonating guaiacol and then forming the calcium sulphonate, a local anæsthetic, well-nigh equal to cocaïne, could be formed. But such was the declared result, and an abstract of this paper for the Journal was now in hand. (See May issue under XX )

## THE BACTERIAL TREATMENT OF SEWAGE CONTAINING MANUFACTURING REFUSE.

BY W. J. DIBDIN, F.I.C., F.C.S., AND G. THUDICHUM, F.C.S.

Some three years since, one of us presented to this Society the results of our experience with regard to the bacterial treatment of the sewage effluent at the Northern Outfall works at Barking Creek, carried out for the main drainage committee of the London County Council. The outcome of these experiments was the clear demonstration of the fact that the (principally) dissolved matters remaining in the sewage after treatment with lime and protosulphate of iron could be practically rendered innocuous by subjecting them to the attack of bacteria under suitable conditions; and these conditions were to a great extent ascertained and set forth in the communication above alluded to.

The system then adopted and applied to the purification of effluent from which the suspended solids had been removed by precipitation has since been shown to be capable of extension to a degree at that time unsuspected, since by the bacterial action alone the whole of the solid matters in the crude sewage may be destroyed and the effluent water rendered innocuous and unobjectionable. This has been shown to be the case by long-continued working at Sutton, Surrey, where experimental purification of the sewage as it arrives on the works, bacterial agencies alone being employed, has been in successful operation for some 18 months. These experiments have already been published, and need not therefore be now further referred to beyond a statement as to the general result of the work, which may be summed up in the following table.

| Average during Month. | Crude Sewage. | | | Coarse Bed. | | | Fine Bed. | | |
|---|---|---|---|---|---|---|---|---|---|
| | Oxygen absorbed in 4 Hours. | Albuminoid Ammonia. | Nitrogen as Nitrites and Nitrates. | O 4 Hours. | $NH_3$ Alb. | N as Nitrites, &c. | O 4 Hours. | $NH_3$ Alb. | N as Nitrites, &c. |
| | | | | Grains per Gallon. | | | | | |
| November 1896 .......... | 4·215 | .. | .. | 1·795 | .. | .. | 0·858 | .. | .. |
| December " .......... | 6·219 | .. | .. | 1·782 | .. | .. | 0·759 | .. | .. |
| January 1897............. | 5·223 | .. | .. | 1·924 | .. | .. | 0·634 | .. | .. |
| February " ............ | 4·464 | 0·278 | Trace | 1·378 | 0·189 | 0·566 | 0·542 | 0·147 | 1·649 |
| March " ............ | 5·044 | 1·108 | Nil | 2·057 | 0·381 | 0·740 | 0·614 | 0·173 | 1·313 |
| April " ............ | 3·111 | 0·893 | Nil | 0·789 | 0·185 | 0·936 | 0·434 | 0·119 | 1·549 |
| May " ............ | 3·873 | 0·706 | Nil | 1·949 | 0·486 | 0·207 | 0·759 | 0·222 | 0·192 |
| July " ............ | 2·757 | .. | Trace | 1·849 | .. | 0·148 | 1·185 | .. | 0·553 |
| September " ............ | 1·295 | 0·154 | Nil | 0·350 | 0·067 | 0·410 | 0·210 | 0·037 | 0·742 |
| January 1898 ............ | 3·00 | 0·78 | Nil | 0·670 | 0·120 | 0·600 | 0·360 | 0·080 | 1·530 |
| March " ............ | 2·58 | 0·25 | Nil | 0·750 | 0·120 | 0·098 | 0·320 | 0·040 | 2·250 |

It having been thus shown that the bacteriological action is fully equal to the task of purifying domestic sewage under ordinary conditions, it remained to be ascertained whether sewage containing relatively large quantities of refuse from various manufactories could be treated on the same lines with equal success. As we have had ample opportunity of testing this important point, we venture to submit for the information and criticism of the members of the Society the outcome of the work done in this direction.

We would first point out that London sewage, upon which the whole of our earlier experiments were carried out, contains very large quantities of manufacturing refuse of all kinds. It has been frequently stated that up to quite recently we had dealt with domestic sewage only, and that when the method was tried in manufacturing towns it would probably fail. There is, of course, in the sewage of London no such preponderance of any particular kind of trade refuse as is found in some other towns, as, for instance, the iron pickle liquor in Leeds and West Bromwich, the refuse from the leather dressers at Yeovil, or the dye liquors in some of the northern towns; but the members of this Society are probably better informed than the general public as to both the quantity and the nature of the various trade effluents which are passed into the metropolitan sewers. We may fairly say that, whilst the Sutton experiments were made with a purely domestic, although very strong sewage, the Barking Creek results were obtained from a sewage containing all manner of trade refuse, but with no special preponderance of any one kind.

Turning now to places in the case of which the special pollutions are strongly emphasised, it will be well to set out the more important of the various descriptions of refuse which have been dealt with, and then to discuss the results of the treatment of the sewage into which these are discharged in quantities to make their presence individually evident.

The special trade effluents examined are:—Tan liquor, brewery waste, distillery waste, leather dressers, acid iron pickle liquor, milk washings, gas liquor, soap works, paper mills, margarine factories.

### *Experiments at Leeds.*

As a type of sewage highly charged with refuse from various manufactories, and in which one special component is strongly marked, that of Leeds may be considered with advantage. By the courtesy of Colonel Harding, the Chairman of the Streets, &c. Committee of the Corporation of Leeds, we are enabled to place before you the results of the experiments which have been carried out at the Knostrop works at Leeds since the first of October last.

The bacteria beds employed are filled with coke in the manner elsewhere suggested, and consist of a coarse bed followed by a fine one. The depth of the first one is 4 ft. 6 ins., and that of the the second 5 ft. The quantity of sewage dealt with is about 200,000 gallons daily.

During the first three months the sewage was put on to the coarse bed directly, without any straining other than that which it received by passing through a screen having a 1¼-in. mesh before arriving at the pumps. This resulted in a quantity of fibrous matter, largely composed of wool refuse, accumulating upon the surface of the bed; but this defect has now been remedied, and so far there appears to be no reason to anticipate any renewal of the trouble thus caused, which, however, did not affect the satisfactory character of the final effluent, the chief symptom being a too great reduction in the original water capacity of the bed.

During the first three months the beds were used twice a day, with the result that they were gradually brought into thoroughly good working condition, as is shown by the following table of averages of the daily analysis which were made.

| Crude Sewage. | | Coarse Bed Effluent. | | Fine Bed Effluent. | |
|---|---|---|---|---|---|
| Oxygen absorbed, 4 hours. | Albuminoid Ammonia. | Oxygen absorbed, 4 hours. | Albuminoid Ammonia. | Oxygen absorbed, 4 hours. | Albuminoid Ammonia. |
| 3·94 | 0·372 | 2·42 | 0·212 | 0·84 | 0·115 |

It was noticed that nitrification was only developed to any extent at about the seventh week, and that it never attained the proportions found in dealing with the purely domestic sewage of Sutton. This is of course in part due to the Leeds sewage being much poorer in nitrogen, and partly also to the very large quantities of iron liquor frequently present. It was observed that whenever the incoming sewage was acid, it retained that reaction through both stages of the treatment. So powerful, however, was the oxidising action of the beds that not only was the organic matter acted upon but the iron salts in solution, which in the crude sewage were ferrous, were in many instances found in the final effluent to be completely changed into the ferric state. In the majority of the samples the iron separated out after a time and settled as ferric oxide in the bottom of the vessel, leaving an absolutely clear and brilliant effluent.

When the beds were filled three times per diem the results obtained were to all intents and purposes as good as before, but the trouble mentioned above, *viz.*, the collection of fibrous matter on the coarse bed, somewhat delayed the experiments in order to arrange for the fitting of suitable screens.

The following shows the average of the analyses made whilst the beds were thus working at the increased rate.

| Coarse Bed Effluent. | | Fine Bed Effluent. | |
|---|---|---|---|
| Oxygen absorbed, 4 Hours. | Albuminoid Ammonia. | Oxygen absorbed, 4 Hours. | Albuminoid Ammonia. |
| 1·31 | 0·15 | 0·23 | 0·066 |

The general character of the effluents is all that can be desired. They are (after the separation of the iron) bright and clear, they are practically without smell, a very slight earthy odour being occasionally noticeable; they are absolutely non-putrescible, and fish can live and thrive in them in the state in which they issue from the beds directly, as has been demonstrated by actual trial, extending over three months, in the course of the experiments under notice. It is thus evident that if such an effluent were allowed to flow into a river even of relatively very small volume there would be no cause for alarm as to its effect upon fish life, which may be taken as a crucial point in regard to the purification of sewage entering streams which are not afterwards used for the purpose of supplying potable water.

Having regard to the facts that these results have been obtained during the winter months we may reasonably hope that as the warm weather sets in the work done by the bacteria beds will be even more effectual.

The particular trade effluents entering the Leeds sewage are those derived from tanneries, galvanising works, copper works (wet process), with large quantities of shoddy waste.

### *Experiments with Maidstone Sewage.*

The sewage of Maidstone contains large quantities of brewery and tannery refuse, and is organically twice as strong as that of Leeds, the albuminoid ammonia averaging 0·64 grains per gallon as against 0·37 grains in the case of the last-mentioned towns. So far, experiments made with this sewage have been on a laboratory scale only, but satisfactory results have been obtained, as will be seen from a consideration of the average amount of oxygen absorbed by the crude sewage and coarse and fine bed effluents respectively, as follows:—

| —— | Crude Sewage. | Coarse Bed. | Fine Bed. |
|---|---|---|---|
| Oxygen absorbed from permanganate in four hours, at 80° F. grains per gallon. | 5·805 | 2·411 | 0·957 |

The albuminoid ammonia was, unfortunately, not estimated in all the effluents, but where done showed a reduction of 66·6 per cent. Since our almost invariable experience has been that work of this kind is always better when done on a large scale than when carried out in our small laboratory bacteria beds, we feel confident that the experiments about to be instituted at Maidstone will result in the production of a good effluent from a very foul sewage.

### *Experiments with West Bromwich Sewage.*

In this case the chief trade effluent which enters the sewers is one derived from the galvanising works. This pickle liquor is of a nature similar to that mentioned in connection with the treatment of the Leeds sewage, and the experimental filters at West Bromwich have also worked out with great success. We might here state, however, that if an effluent be intended to be finally used on land, the iron must first be removed, either by previous lime precipitation or by aeration and settlement of the fine bed effluent in which it still remains in solution. This is a question of the utmost importance in the case of some towns, as, for example, Wolverhampton. Although the action of bacteria beds is not seriously impeded by the presence of the iron, we are nevertheless of opinion that it would be better to remove it by preliminary treatment, where such can be carried out at a reasonable cost.

### *Experiments with Yeovil Sewage.*

The principal industry of Yeovil being the manufacture of glove leather, the chief trade refuse which enters the sewers comes from the yards of fellmongers and leather-dressers, and is of an especially obnoxious character. In addition to the organic impurities it contains arsenic, which is largely employed in the preparatory processes.

In this case the system of treatment adopted has been the now well-known septic tank, in which a large proportion of the solid matters are first brought into solution by anærobic agencies, and then oxidised by passing through fine bacteria beds. The results obtained are in all respects comparable with those given above.

In the cases which have so far been reviewed we have dealt only with sewage containing larger or smaller quantities of the specific effluents. It may be of interest to the members of the Society to instance some experiments made with bacteria beds upon some actual trade effluents in the condition in which they were discharged into the sewer. In each case the liquid was allowed to remain in a coarse bed for two hours, and following this in a fine bed for a similar period of time.

Special trials made on refuse liquor from tanneries, margarine works, galvanising works, gas liquor, milk washings, and yeast washings showed that in all cases observed a very considerable improvement was effected, excepting in the case of the gas liquor, which had to be somewhat diluted before being placed in the beds. The resultant effluents contained no suspended solids, and did not become putrid on keeping.

It will, of course, be understood that these samples are only given to show that the refuse liquors named are amenable to bacterial treatment; the effluent so produced must not in any way be taken as representative of what is required in the way of purification. It is, however, clear that if so much work can be done by passage through two beds, the purification can be extended by increasing the number of times of submission to microbial influences. In practice, concentrated chemical refuse liquors would have necessarily to receive special treatment in order to fit them for bacterial purification; excess of acid or alkali must be corrected; iron, if present in large quantities, removed; and so forth. The point which has been so far considered is as to the treatment of sewage containing manufacturing refuse; and, so far as our experience has gone, such sewages can be efficiently purified by the bacterial method equally with the domestic sewage from a purely residential neighbourhood.

## Discussion.

General **Sir Harry Prendergast** said that he spoke as an unscientific man, but he had taken great interest in the subject of the paper and knew that it was one which was constantly under the consideration of the Local Government Board. He had seen the process in operation, both at Exeter and at Yeovil, and at both these places it worked with perfect success. The sewage at Yeovil was known to be one of the foulest in the world. The results there had varied somewhat at first, because the filters were not properly constructed, but now that they had been reconstructed and refilled he had every hope that the process would prove quite successful.

Mr. **Wood-Smith** observed that however successful the process under notice might be in removing albuminoid material from sewage, that was not wholly sufficient, and it would be necessary to deal further with the removal of poisonous bacteria.

The **Chairman** asked what was meant by poisonous bacteria.

Mr. **Wood-Smith**, continuing, said he meant by that term organisms which were capable of causing death or disease. The process might break down bacteria to their lowest point, but at the same time it was possible for the germs of say, for instance, typhoid fever to pass unnoticed through the beds, as had been proved by several observers. If this happened and the bacteria passed into a slow-flowing river there might be a great collection of typhoid bacilli in the river, which at any time might lead to a serious outbreak of fever if the water were consumed in the lower portions of its course. He would suggest, therefore, that in addition to the present process for removing albuminoid matters from the sewage it should afterwards be treated with free chlorine for the destruction of disease bacilli.

Mr. S. **Rideal** considered that after the last speaker's remarks it would be well to bring the discussion back to the question of dealing with manufacturing refuses, as the broad question of bacteriological processess hardly came within the province of the present paper. The subject before them seemed to resolve itself into the simple question, Does the liquid produced from a sewer or manufactory possess properties which would be destructive to organisms? If the sewage or waste did not possess such properties then it could be satisfactorily dealt with by a bacterial process. He did not think that these questions need concern them as manufacturers, nor from the point of view of health of any towns concerned, because in practically all cases of sewage treatment the quantity of waste chemicals in the sewers was so small, compared to the volume of the sewage, that it could not have any injurious effect upon the life of these organisms. Perhaps free chlorine was the most dangerous substance to be feared, but it would have to exceed one-tenth of a grain per gallon—a very large quantity—to produce any marked retarding effect.

Mr. H. A. **Martin**, *àpropos* of Dr. Dupre's remarks on pathogenic bacteria, quoted from Dr. Angus Smith's presidential address of 15 years ago, in which he said that "it was clear that organic substances, germs, and disease microbes, and the smallest organisms themselves were all subject to that universal unsparing attack of putrefaction and oxidation." It had been proved years ago that the bacteria of decomposition and oxidation were the deadly foes of pathogenic bacteria. Referring to the subject of the paper, it was only right to say that the authors had rendered a public service by their work on this subject. He had attended many Local Government Board inquiries into applications to borrow money for works for sewage disposal, and in every instance the inspectors made a point of inquiring what were the prevalent manufactures in the districts. They evidently attached much importance to this question from the idea that bacterial processes which dealt with sewage of an ordinary character would be likely to fail in dealing with certain manufacturing residues. He therefore regarded the present paper as most timely and opportune, and believed that it would result in saving the country many millions of pounds. He had recently carried out some similar experiments on a small scale with the Yeovil sewage, with the result that a filter constructed similarly to those referred to in the paper as in use at Exeter had produced a greater amount of nitrification than was possible with the Exeter sewage, and that the oxygenation was less than that at Exeter. Moreover, a filter which worked well with Exeter sewage, when brought to bear on Yeovil sewage gave but indifferent results for the first few weeks, though it afterwards yielded very good results indeed. This seemed to indicate that a filter of this kind was a somewhat sensitive apparatus and was affected by different conditions. If so that was a point which would have a practical application and it would be interesting to know how far the Leeds filters had been subjected to varying kinds of sewage during the day, as that would probably provide an explanation of the variations in their working. At Yeovil they were not subject to such changes because the sewage passed first through a tank and the effluent was practically constant in its composition. The results obtained in dealing with manufacturing effluents and liquors unmixed with sewage were surprising and of great practical importance; and when the paper came under the notice of the Local Government Board, as doubtless it would, he hoped it would secure their approval of these bacterial agencies for dealing with all kinds of sewage and residues.

Mr. A. D. **White** thought it most important in connection with this work that one should have the microscopical results given, so that one might know the kind of microbes that were present in the effluent water. It was all very well to say that fish would live in the effluent water produced by this system, but the question was, How long would they live? and he thought that all who took an interest in the subject would feel much more satisfaction if they knew more of the results of the microscopical examination.

Dr. F. **Clowes** wished to add a word of congratulation to the authors on this valuable continuation of the work with which Mr. Dibdin had associated his name. He was sure that the Society generally would be glad to find that Mr. Dibdin was continuing that work in a special direction. Attention was drawn in the paper to the fact that iron present in the sewage underwent oxidation during the filtration, presumably by the action of the bacteria. If that were the cause probably this process of oxidation would be made use of industrially. But it occurred to him that the oxidation of the iron might in reality be due to the highly aërated filter and liquid; this oxidation of the iron was impossible so long as the organic substances were present, but when the bacteria had broken them up then the strongly aërated filter produced its action upon the iron.

He should be glad to hear that the action was due to the bacteria, but the other explanation seemed to him to be more probable.

Mr. A. E. Fletcher had had some experience of this matter as inspector under the Rivers Pollution Act in Scotland. The sewage from chemical works might be roughly divided into two classes, mineral and organic. The discharges from tin-plate and other works containing iron, &c. might usually be specially treated on the works, so that there remained for consideration little but the organic matter mixed with the sewage. It was extremely interesting to see how the septic tank system being carried out unawares throughout the country. Fifteen miles north and south of London there existed chalk formations which acted naturally in bringing about the results sought to be obtained by that system. He knew a case 20 miles north of the metropolis where an old chalk cesspool was dug out and no trace of sewage could be found beyond a few yards from it. The paper before them showed how the work of the septic tank was carried on by nature. At Caterham, lying among the chalk hills south of London, a fear had recently existed that the drainage in the district would get into the deep wells supplying the country round. It was thought that there might be fissures in the chalk: but, were this the case, such fissures would doubtless be filled with gravel, and they were not continuous. In the present cesspools the drainage from the houses was actually exposed to the conditions sought to be realised under the septic treatment. Then it was effectually filtered by the chalk strata. It had been proved by repeated analysis that the water which was pumped from the deep wells in the Caterham chalk beds was perfectly free from all taint of sewage impurity.

Mr. E. Grant Hooper wished to ask whether it was not a fact that not merely cesspools in the chalk but cesspools generally had been found to act in the manner referred to by the last speaker. He thought the authors of this process would admit that they were simply imitating what might be called a natural process which for a long period had been in use in the country. It had been found by everyone who had experience of cesspools that such a receptacle might be continued in use for a considerable time without requiring to be emptied. In porous strata there would be a filtration of the liquid portion of the sewage, but in many other districts it would be found that, having regard to the amount of matter entering the cesspool, the quantity of solid to be periodically removed was extremely small. That was also the experience under this system at Exeter; and it was really the hopefulness of the process that the removal of the sludge was actually effected for us. Of course in dealing with trade effluents the facts in each case must be recognised. If a poisonous liquid were run into the tanks and upon the filter beds that would bring their beneficial action to an end by destroying the organisms which effected the requisite decompositions, and such a state of things must naturally be corrected. To allude to the criticisms of a previous speaker, it could hardly be imagined that anyone dealing with sewage would propose or expect to use the effluent as a source of drinking water. Their chief object must be to bring it into such a condition that it could be run into rivers without causing a nuisance. With regard to the danger of the effluent from a bacterial treatment of sewage being found swarming with such a pathogenic organism as that of typhoid, there could be no doubt that the chance of this was remote, for it had been shown that pathogenic organisms were crowded out in the vast development of the sewage organisms.

Dr. W. C. C. Pakes considered the subject of the treatment of sewage from trade effluents as one of extreme importance to the country generally. He thought there was a misunderstanding in the minds of some members with regard to this process. Some had mentioned it in connection with the septic tank treatment, but the two things ought not to be confounded together. The process of which the authors took advantage was that which had gone on for thousands of years, namely, the nitrification of the matter by the nitrifying organisms present in the superficial layers of the soil. As the sewage passed through these layers the albuminoid matter became oxidised to nitrites and nitrates. The organic material was also oxidised, so that in the deeper strata the organic material had practically disappeared. One member had referred to the difficulty of dealing with the sludge, but it was one of the author's points that the sludge actually disappeared in the process. Other speakers had referred to the question of cesspools. It was quite true that under certain conditions sewage would become dead from sheer want of nourishment for the organisms therein, but this was not the process of the authors, which depended on the oxidation of the organic matter and with it the organisms in the sewage. He could not understand Mr. Wood-Smith's criticisms. That gentleman appeared to assume that the authors did not know what they were talking about. Mr. Wood-Smith surmised that pathogenic organisms would get through the filter beds; but if he read Mr. Dibdin's papers he would find that it was carefully provided for therein that the filter beds must be alternately dry and wet, and under those circumstances it would be impossible for any organisms to get through. Referring to the question of contamination of wells by drainage, he would point out that Dr. Vivian Poore had for years maintained a garden in the country systematically manured with human sewage, and in the centre of this garden was a well. The garden received the excreta from thirty cottages yet Dr. Poore stated that, having submitted the well water to four or five bacteriologists (among them being Dr. Klein), not one of them had been able to find a *bacillus coli communis*.

Mr. W. Balfour could testify to the success of the operations carried out at Leeds. He was there a fortnight ago and conferred with the engineer who had charge of the process and was assured that everything had been conducted rigidly and that the complete liquefaction of the solids treated had been effected. He had no interest in the process and had been perfectly satisfied with what he saw, not only at Leeds, but also at Sutton and Exeter. He hoped that the matter would soon be taken up by the Royal Commission and that the result would be that many places now paying a heavy penalty for the disposal of their sewage would find a relief, due to the success of this process.

Mr. George Thudichum said that owing to the kindly nature of the criticisms he had very little to reply to. There had been a general misunderstanding on one point. Mr. Dibdin and himself did not propose to read a paper on the general bacteriological aspect of the question, but solely on the application of the process to trade effluents. They had therefore left the wider question untouched. One speaker had said that they dealt with sewage by one process only—the Sutton process. He thought they had made it clear in the paper that they adopted any process that could be called a bacteriological one. The septic tank process had been examined and it had done excellent work on trade effluents. The question of pathogenic organisms had been discussed already that evening, and he, therefore, need not refer to it further than to say, in reply to Mr. Wood Smith's statement that pathogenic organisms might go through the filter beds, that the whole of his experience was in the opposite direction. The experiments made in Exeter by Dr. Picard pointed specially to this, and of the *bacillus typhosus* put in only an extremely small percentage came through. It was also common knowledge that in sewage generally the *bacillus typhosus* had a very poor chance of living. A few years back Messrs. Lawes and Andrews had made a research on the subject and failed to find this bacillus in London sewage at a greater distance than 50 yards from even a special fever hospital drain, and then only when the use of disinfectants was discontinued. He hoped that that would make it clear that they had not much to fear. What a large area of land would do in this respect the artificial bed would do as well. He had been asked to give an idea as to the quantity of sludge that would be produced in dealing with a sewage such as that of Leeds. The essence of both the septic tank process and the Sutton process consisted in the fact that there was *no* sludge production. The matter which at Leeds they screened from about 200,000 gallons per day was merely a few barrow loads per day, which could hardly be considered as sludge, since it consisted of fibrous woody matter readily dried and got rid of, and had none of the offensive properties of ordinary sewage sludge. One member had said that the processes which they had utilised were the natural processes of nitrification pure and simple. That was true to a certain extent, but a good deal of work had to be done before they came to nitrification, *viz.*, the peptonising of the suspended solids, and that was quite as important a matter as the other. With regard to the President's (Prof. Clowes) remarks, the authors did not wish to be thought to state that oxidation of the iron was effected by bacteria. This was done by residual oxygen after the bacteria had done their work upon the organic matters.

# Manchester Section.

*Meeting held on Friday, April 1st, 1898.*

MR. GEO. E. DAVIS IN THE CHAIR.

## SECTION 22 OF THE PATENTS ACT, 1883, AND ITS BEARING ON BRITISH INDUSTRY.

BY IVAN LEVINSTEIN.

In the year 1717, Germany established compulsory education; towards the end of the Napoleonic wars we find that not only considerable efforts were made to re-organise her military system, but also great attention was devoted to the development of secondary education, in her gymnasiums, and highest-grade scientific education in her universities. But, in spite of all her great achievements in the field of knowledge, her position and her industrial progress, even as late as 1870, were quite insignificant as compared with ours. Superior education alone, therefore, cannot account for the remarkable industrial progress of the German people. As far as education is concerned, and I have in my mind especially its bearing on our chemical industries, we are not now so much behind as is generally asserted. Our great College and the Victoria University are now turning out chemists quite as well trained as those who have studied an equal term at any German university. If the number of these highly-trained chemists is not yet large enough to supply the demand, there is no difficulty in getting first-class chemists from Switzerland and Germany, at reasonable salaries. What are, then, the reasons for the phenomenal expansion of the chemical industries in Germany? That the people of this country are by nature as gifted and as well adapted for the acquisition of scientific and technical knowledge as their German *confrères*, there can be no doubt; that they possess inventive faculties at least equal to them is confirmed by the fact that some of the greatest scientific and industrial discoveries have been made by Englishmen. It is obvious, therefore, that Germany must possess advantages over us, beyond her superior system of education, in the development and expansion of her chemical industries. That this is true I will endeavour to show.

The military system of Germany, with all its drawbacks, has created a highly-disciplined army of workers, men by no means better technically trained than our own, but both disciplined to obey and accustomed to command; systematically and methodically trained to order, cleanliness, punctuality, and subordination. The German Government fully appreciates the importance of the chemical industries; not only because they are now an important factor of her national wealth, but on account of the far-reaching influence which they already exercise on the development of the textile and other allied industries, and which they are destined to exercise in a still larger degree in the future. Any disadvantages under which their home industries may be labouring as compared with foreign competitors, if brought before the Government by her chambers of commerce, or other recognised authorities, are speedily amended. In addition, Germany possesses the following advantages:—

1. Cheaper labour.
2. Cheaper inland freights.
3. Cheap pure alcohol (ethyl and methyl).
4. Lower rates and taxes.
5. Patent laws enacted for the benefit and advantage of her native inventors and her home industries.

As far as cheaper labour comes into question it need not trouble us much, for (as I have stated on various occasions) the fiscal policy of Germany combined with the ever increasing taxation will soon bring wages to a level with ours; moreover, it is even questionable whether lower wages are really an economical advantage. Cheaper inland freights by rail and canal are however, a distinct advantage enjoyed by our rivals. A much more serious item, however, is the fact that German manufacturers have at their command both pure ethyl alcohol and pure methyl alcohol at a price so much lower than that which we have to pay in this country, that we are practically excluded from manufacturing a large number of medicinal preparations and colouring matters or other compounds in the production of which these alcohols are required. As to patent laws, I venture to express my absolute conviction that the chemical industry of Germany, especially that of coal-tar derivatives, would not be to-day in such a prominent position but for the German patent laws, which have been enacted and are executed for the benefit of German inventors and German industries.

Now, what are the advantages which we possess?

1. Free trade.
2. Cheap fuel.
3. Cheap primary raw material.
4. Favourable geographical position.

We possess, therefore, all natural factors wanted for the establishment of chemical industries to a larger extent than any other country, and, if we have not succeeded in retaining the supremacy once possessed and have allowed Germany to steal a march upon us, we have ourselves, or rather our legislators, to blame for this misfortune.

For years we have been, for example, agitating for an amendment of our patent laws, which are doing endless injury to our inventors, our trades and industries, in their want of conformity with those of our foreign rivals. Now, the following facts will show what, so far, has been the result of our agitation:—

In February 1897, Captain Phillpotts, M.P., called attention in Parliament to the want of conformity of our patent laws with those of other countries, particularly in regard to the non-requirement of the working of patents in this country. The President of the Board of Trade referred Captain Phillpotts to section 22 of the Patents, &c. Act of 1883, and stated that he was not satisfied that the law was not sufficient, as it now stood, to meet the cases referred to by Captain Phillpotts.

Again, on the 26th February last year, when Mr. Schwann, and later on Mr. Cawley, directed attention in Parliament to the same section, pointing out some of its shortcomings and asking for the nomination of a Select Committee to examine the question, and to report, with a view to the making of such changes in the patent law as might be shown to be equitable and practicable, Mr. Ritchie's reply to Mr. Schwann was similar to the one given to Captain Phillpotts: "Until it is proved by experience that section 22 of the Act is insufficient to compel the 'working' of patents taken out by foreigners in this country, I am not prepared to support a motion for a Select Committee."

On April 8th last year, a deputation from the Manchester, Blackburn, Belfast, and Macclesfield Chambers of Commerce, the Manchester Calico Printers' Association, the Bleachers, Dyers, and Finishers' Association of Lancashire, the Manchester Association of Engineers, and other important commercial and industrial bodies, attended at the Board of Trade to point out the enormous injury inflicted upon British industries owing to the ineffective character of section 22.

The deputation, which was introduced by Sir William Houldsworth, M.P., including Sir Henry Roscoe, Mr. F. Cawley, M.P., Mr. C. E. Schwann, M.P., Mr. F. Platt-Higgins, M.P., Mr. H. O. Arnold-Forster, M.P. (the Belfast Chamber of Commerce), Captain Phillpotts, M.P., Mr. W. H. Holland (President of the Manchester Chamber of Commerce), Mr. Ivan Levinstein, Mr. Le Neve Foster, Mr.

Grimshaw and Mr. E. Helm (also of the Manchester Chamber of Commerce), Mr. Henry Harrison (Blackburn), Mr. W. L. Cameron (Macclesfield), Mr. Neville Clegg, Mr. L. B. Lee, Mr. Joshua Knowles (Calico Printers' Association), Mr. F. Hardcastle (Bleachers' Association), Mr. Henry Allen and Mr. John Stanning (Dyers' Association), Sir W. H. Bailey (Manchester Association of Engineers), and Mr. John Christie (John Orr Ewing and Co., Glasgow).

In introducing the deputation which, in the unadvoidable absence of Mr. Ritchie was received by Sir Courtenay Boyle, C.B., Sir Wm. Houldsworth referred in particular to the ambiguity of the word "default" in section 22, which was understood to mean an absolute refusal on the part of the patentee to grant the license to the applicant; further, that the Act fixed no time within which the patentee was bound to "work" his patent; again, that the word "work" was also ambiguous in its meaning; finally, that the power to enforce by "mandamus" was insufficient, and he suggested that a patent should be declared null and void if the conditions prescribed by the Board of Trade were not fulfilled.

He also mentioned that the patent laws of France and Germany were much more in harmony with the suggestions which would be submitted for the amendment of this section than with the British law. Other members of the deputation pointed out that section 22 had been practically dormant for 15 years, since no petition had ever been proceeded with.

In reply to Mr. Levinstein's questions as to the meaning of the word "default," Sir Courtenay Boyle declined to admit that the Board would be unable to proceed if there was default but no definite refusal. Mr. Levinstein gave an illustration of a case of a foreign inventor of a machine for making flannellette, which machine the patentee did not make in this country but which was used here, and he asked Sir Courtenay Boyle whether the machine being worked in this country, the patent was "worked"; to which Sir Courtenay replied that he should say that the machine was worked in this country, but the patent was not.

Mr. Levinstein then drew attention to the immense injury done to British trade by the ambiguity of the Act, and pointed out that in the five years 1891 to 1895 no less than 600 patents were granted to foreigners for coal-tar products, not a single one of which was being "worked" in this country.

Sir Courtenay Boyle, in his final reply, stated that it would not be wise to disturb an Act of Parliament until its inefficiency was clearly shown, and until they had the opportunity and sufficient knowledge to enable them to replace it with a better one. He saw little prospect of legislation in this matter during that session, but between this and the next session it was desirable that carefully selected cases should be the subject of application to the Board of Trade.

There were many instances — Mr. Levinstein had mentioned several—in which it was clear that the default of the patentee to work his patent in the United Kingdom and supply the requirements of the public had been a public injury alike to the traders and the industrial interests in this country.

Surely cases might be found in which, taking advantage of sub section "A" (if the patent had not been worked in the United Kingdom) or sub-section "B" (that the reasonable requirements of the public could not be supplied) or both, the Board of Trade might be pressed, and pressed more closely than it has ever yet been, to take proceedings under the section, and to order the patentee to grant licenses on reasonable terms.

Of the willingness of the Board of Trade to do all that they could to carry out the intention and spirit and letter of the Patents and Trade Marks Act of 1883, he could give them the utmost possible assurance, because he felt that the interests of the patentees in this kingdom and the interest of the trade and industries, which was so largely affected by English and foreign patent legislation, had the greatest possible claim upon the attention, not only of that Department, but of Parliament. He hoped that they would watch the procedure of the next few months, if application were made to the Board, in order that during the next session, if necessary, a further application might be made to the Board.

It will be clear from these proceedings that the Board of Trade distinctly invited the deputation to apply to the Board with a few selected cases of applications for licenses of patents not "worked" in this country. It was in consequence of this invitation that Mr. Ivan Levinstein, of Messrs. Levinstein, Limited, at the request and with the approval of the Manchester Chamber of Commerce petitioned the Board of Trade, for a compulsory grant of a license to work two patents of a foreign patentee which were not up to that time "worked" in this country.

Had it not been for the pressure put upon the deputation to bring some test cases before the Board, this section of the Act would have continued to be dormant. No one, probably, would have petitioned the Board, because the opinion of the highest legal authorities in this country was that the section was altogether ineffective owing to its ambiguity. Even the legal authority appointed by the Board of Trade to adjudicate between the parties—entitled the Referee—stated on the 10th day of the proceedings (February 28th) that "whatever the intention of the people who framed this section was, they had not stated it in the clear and definite language in which an Act of Parliament ought to be drawn."

Before going further it will be desirable to give the words of the British law referring to the grant of licenses, and working of patents, in comparative form, with the corresponding laws of France and Germany, which will clearly show the want of conformity of our law with those of the two countries mentioned :—

### *British Law.*

*Section* 22 *of the Patents, &c. Act*, 1883.—"If on the petition of any person interested it is proved to the Board of Trade that by reason of the default of a patentee to grant licenses on reasonable terms (*a*) the patent is not being worked in the United Kingdom; or (*b*) the reasonable requirements of the public with respect to the invention cannot be supplied; or (*c*) any person is prevented from working or using to the best advantage an invention of which he is possessed, the Board may order the patentee to grant licenses on such terms as to amount of royalties, security for payment, or otherwise as the Board, having regard to the nature of the invention and the circumstances of the case, may deem just, and any such order may be enforced by mandamus (which will require application to the High Court of Justice)".

### *French Law.*

*Article* 32.—"The following shall be deprived of all their rights :—

(1.) The patentee who has not paid his annuity before the beginning of each year of the term of his patent.

(2.) The patentee who has not worked his discovery or invention in France within the terms of two years from the date of the signature of his patent, or who has ceased to work it during two years, unless in the one case or the other, he justifies himself as to the cause of his inaction. (*a*.) The patentee who has introduced into France articles of manufacture made abroad and similar to those which are protected by his patent."

### *German Law.*

*Paragraph* 11.—"The patent can be revoked after the expiration of three years. (1.) If the patentee fails to work his invention in Germany to an adequate extent, or at least to do everything that is necessary to ensure it being worked. (2.) Whenever the grant of a license to others to use the invention appears to be demanded in the public interest, and the patentee refuses to grant such license upon adequate compensation and good security."

It has been said by some that the laws of France and Germany in regard to the working of patents is not strictly carried out, that one may get round the Act in some way or other. This is not true, as is proved by the fact that the following firms of coal-tar colour makers have large establishments in France, in which they manufacture all articles patented by them in France on a scale sufficiently large to supply the whole demands of France. They do not import an ounce of such patented article, and if they did it would be at the risk of having their patent declared void :—

Badische Anilin-Soda Fabrik, Mannheim.

Farbwerke, Vormals Meister, Lucius, and Brüning, Hoechst-am-Main.

Cassella and Co., Mainkur.

The Berlin Actiensgesellschaft für Anilin Fabrikation.

Farbwerke Mühlheim, &c.

Farbenfabriken Elberfeld, &c.

As regards Germany, cases have been decided by the Court in which the patent, not having been worked within three years, has been declared null and void.

### *The Spirit of Section* 22.

If we look for a moment at the fundamental principle of our Patent Laws, it will be seen that clause A of section 22 (the section commencing *a.* "The patent is not being worked in the United Kingdom") was really acted upon long before the enactment of this section. This sub-section must have been adopted for the purpose of emphasising this principle. The Statute of Monopolies passed in the reign of James I. was intended to cut off all claims on the part of the Crown to the right of granting monopolies. That statute declared that all monopolies, grants, and letters patent were contrary to the law and void, but it expressly excepted from its operation all letters patent, and grants of privilege for the sole working or making of any manner of new manufactures to the true and first inventor of such manufacture, which others, at the time of making of such letters, patents, and grants, should not use "If they be not contrary to law, nor mischievous to the State by raising prices of commodities at home, or hurt of trade, or generally inconvenient."

This statute is law at the present time. It is unfortunate that section 22, instead of defining and extending it, has only created difficulties in carrying out the previous law, owing to its ambiguous and meaningless language. By the statute of James I. it is evident that a patent which was not worked in this country could not be upheld. The statute was intended, by creating these exceptions to the otherwise general abolition of monopolies, to grant a patent only to the inventor who had introduced a new industry; employing British labour, and benefiting British trade by the initiation of such new trade or industry.

### *The Petition.*

The petition to the Board of Trade in the matter of Letters Patent No. 9642 of 1889 and No. 15,176 of 1889, Levinstein, and Meister, Lucius, and Brüning, sets forth that the patentees (*inter alia*) are manufacturing chemists who carry on their manufactures at Hoechst-am-Main, in the German Empire; that the patentees have wholly failed to introduce the use of their patents or of any of them within the realm, and have not at any time attempted so to do; that it is, on the contrary, the settled commercial policy of the patentees to prevent the introduction of their patented process within this realm. Thus, by reason of the default of the patentees, the grants of privileges thus made to them are mischievous, are the cause of hurt of trade, and are prejudicial to Her Majesty's subjects in general.

The petitioners are manufacturing chemists carrying on their business on a large scale at Crumpsall Vale, Blackley, near Manchester. They have conducted numerous and costly experiments, as a result of which they have introduced from time to time many improvements in the manufacture of such dyestuffs. They have recently discovered processes by which valuable new dyestuffs can be manufactured. Other similar improved dyestuffs have been discovered, and these can only be manufactured by employing part of the respondents' patented process. The petitioners therefore apply to the patentees for a licence, and this the patentees refused to grant. It is necessary, to enable the petitioners to introduce the patented manufacture into use within this realm, and to work and use to the best advantage the said inventions of which they are possessed, that they should have this licence. The petitioners therefore pray that an order may be made by the Board of Trade under section 22 of the Patents, &c. Act, 1883, that the patentees shall grant to them a licence.

### *Proceedings.*

The referee appointed by the Board of Trade was Mr. R. W. Wallace, Q.C. The proceedings being altogether of a novel character, there was no fixed procedure, nor were there any precedents to follow. The referee settled the order of proceeding. The petition was heard in the Royal Courts of Justice, Queen's Bench, Court No. 6, London. It lasted eleven days. The total expenditure of all parties concerned amounts to about 4,000*l.* The expenditure of the Board of Trade will be about 700*l.* The petitioner was for about six days examined and cross-examined, and for several days at least, instead of being in the position of a plaintiff, occupied that of a defendant.

The petitioner relied not only upon clause (*a*), but he produced four patents for which he wanted the raw material patented by the respondents. This raw material was patented nine years ago. The respondents had never worked or produced their raw material in this country. On the other hand, this raw material was open and free to all German manufacturers to produce, the reason for this being that one of the patents of the respondents was refused in Germany when applied for, and the other was revoked by the German courts on the ground of its want of novelty.

For several days the respondents attacked the novelty of the patents granted to the petitioner unsuccessfully. The petitioners were forced to produce the whole of their manufacturing calculations, and to divulge private business affairs to the great prejudice of their interests. The petitioners' costs amounted to nearly 2,000*l.* If the decision of the Board of Trade is against the petitioner, after all his expenditure, risk, and trouble, he has no right of appeal. On the other hand, if the decision is in the petitioners' favour, the decision would not be valid until the case had been argued again before the High Court upon a plan for a writ of mandamus. After this, the respondent has a right of appeal to the House of Lords.

As between a financially weak applicant and a strong respondent, the arrangements could not possibly have been contrived better for the respondent to play the game of "heads I win, tails you lose." When these considerations are borne in mind, as well as the fact that the petitioners run the risk of their own invention being invalidated, it will be seen at once that section 22 is quite useless for any practical purpose, "*whatever the decision of the referee may be.*"

It appears that the whole onus of proof is thrown upon the petitioners. They have to prove everything. They have to prove that not only the respondent's patents are not worked in this country, but also that the invention of which they are possessed is one which would stand the test of hostile examination in open court. How different to all this is the procedure in Germany and France will be seen on consulting the parallel statutes of the three countries on page 320 which have been quoted.

In France, if the patent is not worked within two years—and the patentee has to prove that it is worked—the patent is revoked or may be declared void. In Germany the law is similar, with this exception, that the patentee, instead of two, has three years' time within which to work his invention. The expenditure on the part of a petitioner will not amount to as many shillings as here the costs are pounds.

## THE PRODUCTION OF METALLIC TANNATE LAKES OF BASIC COLOURING MATTERS ON COTTON CLOTH.

BY ROBERT J. FLINTOFF, F.C.S., ETC.

There are several methods in general use for precipitating on cotton cloth combinations consisting of a metallic oxide, tannic acid, and basic colouring matter. Salts of antimony are for the most part employed for this purpose, but tin compounds are also used to a limited extent. The colouring matter, dissolved in some suitable medium—say acetic acid—is mixed with a thickened acetic acid solution of tannic acid, and the resulting paste printed upon the cotton fabric. During the drying and steaming processes, the acetic acid is driven off, leaving the tannic acid lake precipitated on the cotton fabric. But as this compound is not fast to soaping, the cloth is passed through a hot solution of tartar emetic, when an antimony tannate lake is formed on the fibre and a greater degree of fastness obtained.

Now, in the case of a tin compound being used, the cloth is prepared before printing with a solution of stannate of soda, and then passed through a bath containing a very dilute solution of sulphuric acid, and finally well washed in cold water. A hydrated stannic oxide is precipitated on the cloth, and then the colouring matter is fixed during the steaming process by combination between the tannic acid and oxide of tin.

In some styles it is advantageous to prepare the cloth with tartar emetic before printing instead of passing it through a hot solution afterwards.

The objections to the use of antimony as a fixing agent are its high price and the unavoidable waste incurred during the passage of the cloth through the fixing beck.

This must always take place, because the cloth becomes saturated with the potassium antimony tartrate solution, and consequently removes from the bath in the large majority of cases a greater amount of the antimony compound than is required to actually precipitate the tannic acid. The quantity of fixing agent wasted during this process will depend upon the nature of the colours used in the printed design and the thickness of the cloth.

Suppose, for instance, cotton cloth has been printed with a six-colour design, and every colour is a basic dye fixed with tannic acid. Then, on its passage through the fixing solution, a definite amount of antimony will be removed—an amount not depending upon the tannic acid contained in the cloth, but upon the quantity of solution absorbed by the cloth. And if, in the pattern under consideration, only one small peg colour had been a basic dye, and the cotton cloth a greater thickness, then a very much larger amount of antimony would have been abstracted from the beck under these conditions, although they actually required less.

When a tin compound is employed, another process is introduced, and consequently the cost of production is increased. But should this be a matter of little or no importance, cleaner, brighter, and faster work is obtained. Now, since the efficiency of any process is represented by a factor expressing the ratio between the quality of the work produced and the cost of production, it appeared to me that the efficiency of the present process for fixing tannic acid colours might be materially increased by adopting a cheaper method, yet one giving equally good results.

The experiments I am about to describe were made with this object in view. And, although they have not been as successful as I could have wished, nevertheless interesting results have been obtained and several new ideas developed in connection with the fixing of basic colouring matters on cotton cloth. Firstly, it was necessary to find a metallic oxide to replace the antimony oxide, and then to ascertain the best possible way of applying it.

A metallic oxide is required that will give equally good, bright, and fast shades, both to light and soap, with any basic colouring matter as can be obtained by the older processes. Accordingly, a tannic acid printing paste containing 4 oz. of Auramine Yellow and 1 oz. of Brilliant Green to the gallon, and made as previously described, was printed upon oil-prepared cloth, dried, and steamed. The fent was now divided into 14 equal parts, and each part was separately passed into a solution of a metallic salt, well washed in cold water, soaped at 120° F. for 10 minutes in a bath containing 5 oz. of soap to the gallon, again well washed, and finally dried. It is not necessary to give the quantity of each precipitant actually used, as in every case the bath contained an excess of the metallic salt.

The following table gives the essential results of these experiments.

TABLE No. 1.

| No. | Precipitant. | Shade. | | Fastness. | |
|---|---|---|---|---|---|
| | | Intensity. | Colour. | Soap. | Light. |
| 1 | Aluminium acetate... | Same | Slightly greener. | All about the same. | 6 |
| 2 | " sulpho cyanide. | Less | " | | 5 |
| 3 | sulphate. | Less | " | | 4 |
| 4 | Glue.................. | Very much less. | " | | 11 |
| 5 | Lead acetate ........ | Greater | Browner | | 2 |
| 6 | Stannous chloride.... | Less | Greener | | 12 |
| 7 | Oxymuriate of tin.... | Less | " | | 13 |
| 8 | Chromium acetate ... | Greater | " | | 14 |
| 9 | " sulphate.. | Less | Slightly greener. | | 10 |
| 10 | Ferric acetate........ | Very dark myrtle | | | 1 |
| 11 | Calcium acetate...... | Same as No. 6 | | | 9 |
| 12 | Zinc sulphate ........ | Less | Greener | | 7 |
| 13 | " acetate ......... | Less | Greener | | 8 |
| 14 | Potassium antimony tartrate. | Standard for Comparison | | | 3 |

The first column is the precipitant used; the second gives the corresponding shade judged with respect to its intensity and colour; the third and fourth explain themselves. The affixed number represents the comparative fastness of each shade; hence No. 1 is the fastest, and No. 13 the most fugitive. In every case the antimony tannate lake was taken as the standard for comparison.

There are several points in this table worthy of notice. The iron tannate and the lead tannate compounds both withstand the action of light better than the antimony tannate lake. The alumina tannate is rather more fugitive. Then follow the zinc, calcium, chromium, and, lastly, tin lakes.

The acid eliminated during the fixing process would appear to exert a certain influence on the shade. For instance, in the case of the three alumina salts. The acetate gives the best result, and the sulphate the worst. This also applies to the chromium and zinc salts. In these cases a much stronger solution was taken than would be ordinarily employed for practical working, for the simple reason that the relative actions of the liberated acids might approximate to the actions obtained on the large scale. After the bath had been in operation some time, the metallic oxides might be expected to arrange themselves in a different order with different colouring matters, but from the results of experiments similar to the above made with several colouring matters I have found the facts stated to be substantially true for any basic colouring matter, always presupposing the conditions remain the same.

The shade obtained by using glue as the precipitating agent is exceedingly bright and transparent, but is somewhat fugitive to light. The aluminium and antimony lakes gave the best results in these preliminary trials, and therefore I decided to make further experiments with aluminium salts of organic acids in order to determine the acid least injurious to the colour lake. These salts were prepared in solution by dissolving alumina in an aqueous solution of the corresponding acids.

Comparative trials similar to the ones previously described were now made by fixing fents printed with Auramine Yellow, Brilliant Green, and Victoria Blue in solutions of oxalate, tartrate, and citrate of aluminium, with the results given in Table No. 2.

In that table the precipitants are arranged in accordance with the qualities of the shades given, and it will be noticed the results are fairly concordant. The oxalate gives the best shade in two cases out of the three, and in the other it is inferior to the one from potassium antimony tartrate. The tartrate gives in every case an inferior result to the oxalate, and the citrate is decidedly the worst. Soap affects each shade to the same extent. With regard to fastness to light, the aluminium oxalate is not quite equal to the antimony compound, but this is not a serious matter, as there is very little difference between the fastest and most fugitive colour lakes in each case. From these results it is obvious that oxalate of aluminium may replace potassium antimony tartrate with certain advantages respecting efficiency. But, as we have still to employ the method now in vogue of passing the printed cloth through the solution, there will still be considerable waste during an extended period of time. I fixed many fents printed with several colours in a bath of oxalate of alumina with very satisfactory results, and found in every case the work was as clean and bright as when fixed with potassium antimony tartrate. The oxalate of alumina bath brightens alizarin red and pink, and also gives good clear whites. In this particular it is a distinct improvement on the older method. It does not affect logwood black.

Table No. 2.

| — | — | Auramine Yellow. | Brilliant Green. | Victoria Blue. |
|---|---|---|---|---|
| Shade of colour.. | 1 | Al. Ox. | Pot. Ant. Ta. | Al. Ox. |
| | 2 | Al. Ta. | Al. Ox. | Al. Ta. |
| | 3 | Pot. Ant. Ta. | Al. Ta. | Pot. Ant. Ta. |
| | 4 | Al. Cit. | Al. Cit. | Al. Cit. |
| Fastness to soap .. | — | All about the same. | | |
| Fastness to light | 1 | Al Ox. | Pot. Ant. Ta. | Pot. Ant. Ta. |
| | 2 | Al. Ta. | Al. Ox. | Al. Ta. |
| | 3 | Al. Cit. | Al. Ta. | Al. Cit. |
| | 4 | Pot. Ant. Ta. | Al. Cit. | Al. Ox. |

No. 1 indicates the best result.
No. 4 indicates the worst result.

Although the oxalate of alumina would be rather cheaper than the antimony potassium tartrate as a fixing agent, yet the method of procedure has not been made more economical from a scientific point of view, for the reason previously stated; and, therefore, experiments were now made with the object of eliminating entirely the fixing of the colours by a passage through a solution of a metallic salt. I desired to substitute a method the function of which should be to regulate the quantity of metallic oxide used to the actual requirements; that is to say, under all conditions the quantity of fixing agent employed will always have a definite relation to the quantity of tannic acid lake to be fixed. Obviously, to obtain this result attention must be directed to the printing paste.

If any alumina compound could be found to mix with a tannic acid printing paste without precipitation taking place until the steaming process, when the formation of the alumina tannate lake would be brought about, then the lake so produced would be perfectly fixed, and it would only be necessary to wash and soap the goods.

The amount of fixing agent required to precipitate the tannic acid could be fairly accurately determined, and a slight excess always used. Hence the quantity wasted by this process would be exceedingly small.

This I have succeeded in doing by adding a little precipitated hydrate of aluminium, along with a small quantity of an organic acid, to the printing paste.

Tartaric acid is very good for this purpose, but a smaller quantity of oxalic acid will give equally good results.

The alumina pulp is made by precipitating a solution of alum with ammonia, washing the precipitate well, and allowing it to drain on a cloth filter until it contains about 20 per cent. of $Al_2O_3$. Then it may be kept any length of time in a moist condition, but it is advisable only to make a small supply at a time, as its preparation requires very little labour and entails no inconvenience.

Three comparative trials were made with fents printed with Auramine Yellow, Brilliant Green, and Victoria Blue, fixed with alumina pulp and potassium antimony tartrate.

They were all equally fast to soap, and, except in the case of Brilliant Green, the alumina compound was decidedly faster to light. I also consider the shades produced from the alumina were equal to the corresponding antimony ones.

In these experiments four parts of alumina pulp 20 per cent. were taken for every two parts tannic acid contained in the colour.

It now became necessary to determine the exact amount of alumina pulp required to combine with a given quantity of tannic acid, or in other words, the amount of pulp required to give the best shade with a colour paste containing a known amount of tannic acid.

After making many trials with varying quantities of 20 per cent. alumina pulp, I find one part of the latter will precipitate on the fibre seven parts of tannic acid, 70 per cent.

From the results of these laboratory experiments I immediately came to the conclusion the process just described had no small practical value, but on experimenting further on the large scale these hopes were not realised. The printing paste, when freshly prepared, gave excellent results, but as its age increased so did the quality of the colour produced from it decrease, and, therefore, the practical value of the process became somewhat small. It was now a matter of interest to ascertain the nature of the changes taking place in the composition of the printing paste owing to the prolonged action of the ingredients on each other.

As a result of this inquiry I found an aluminium compound of the tannic acid lake had already been formed, and consequently existed in the printing paste as a pigment. It is, therefore, clear the shade resulting from printing this mixture on the fabric could not possibly equal the shade produced by the direct precipitation of the same compound through the structure of the cotton fibre. Because the particles of colour are in much larger masses and aggregates in the former case than in the latter, and hence a very much less degree of superficial evenness results. Consequently the reflecting surface is inferior and, therefore, the quality of the colour is also inferior.

This alumina tannate lake is formed by the acetic acid combining with the alumina to form aluminium acetate, which reacts on the acetic acid solution of the tannic acid lake with the simultaneous formation of an alumina tannate lake.

For these reasons this particular application of alumina for the fixing of basic colouring matters cannot be employed on the large scale. However, the difficulty may be overcome by substituting for the alumina a metallic oxide, which is not acted upon by acetic acid. Antimony-trioxide answers the purpose admirably, and a printing paste containing this metallic oxide as the fixing agent may be kept without decomposition for a long time. This modification of the present method of fixing tannic acid colours with a solution of antimony salt obviates the passage of the cloth through a fixing bath after printing. Thus the process has been made more efficient, as no unnecessary waste is now incurred. But as the same metallic oxide is still employed as a fixing agent, the improvement on the present process depends solely upon the alteration in the method of application.

In order to find a substitute for antimony I have made experiments with several metallic oxides, and have found all of them to be more or less completely converted into soluble salts by the continued action of acetic acid.

The fixation of printed tannic acid colours during the steaming process by the introduction of antimony oxide into the printing paste eliminates a process—*viz.*, fixing after steaming. It is exceedingly simple and gives results equal to the older method in every respect.

The facts just enumerated relating to the fixing of printed basic colours exhibit such a marked contrast to the results obtained by the method of dyeing, that it seemed desirable to investigate this particular connection still further in order to explain the discrepancy.

To facilitate the inquiry a complete appreciation of the variations in the details of the processes is absolutely necessary.

In the case of the direct production of printed designs the basic colouring matter, tannic acid and metallic oxide are placed upon the fabric all at the same time; or the metallic oxide may be omitted, and the fixing performed in the usual manner. Whereas, in the case of dyeing, the metallic oxide is firstly placed upon the cotton fabric, then fixed by precipitation with a metallic salt, and finally dyed by immersion in a solution of the colouring matter.

These are the main and essential differences. And it is difficult to understand why there should be such wide divergencies in the properties of the colours produced by these respective processes, where everything remains a constant except for a reversion of the method of preparation. In each case we are supposed to have a combination pure and simple consisting of basic colouring matter and metallic tannate, but if this is the case the properties of the colours on the fabric should be similar. This is not so, and it is my object to investigate this matter in order to bring forward an adequate explanation.

Two simple experiments, relatively quantitative, illustrate the precise disagreements in the results obtained by the methods of dyeing and printing. Cotton cloth is prepared in a solution of tannic acid and dried. It is now divided into two parts. One is fixed by immersion in a solution of a salt of antimony, and the other in a solution of a salt of aluminium, and both are separately well washed in cold water. They are now dyed in the same bath of any basic colouring matter and dried. We now find a great difference in the respective shades. The antimony lake is far superior to the alumina lake. If, on the other hand, two printing pastes of the same basic dye are prepared and to the one hydrate of aluminium is added and to the other hydrated oxide of antimony; then on printing, steaming, soaping, &c., both colours under exactly similar conditions, practically, no difference can be detected between the two shades.

I shall have occasion to frequently use the phrase "modification of the absorption power of tannic acid," and I take this opportunity to explain its exact meaning. It refers to the changes in the property of tannic acid to extract a basic dye from its aqueous solution, resulting from the conversion of the free acid into metallic tannates. And as an illustration of the modification of the absorption power of tannic acid by combination with metallic oxides, I cite the following experiment:—

Cotton cloth known to contain a given quantity of tannic acid is divided into two parts. On one is precipitated antimony tannate, and on the other aluminium tannate. Each part is separately dyed in a solution of a basic dye of known strength. After dyeing, both solutions are made up to a given volume, and a definite portion of each compared colorimetrically. This may easily be done quantitatively in the usual way. It was found the antimony tannate lake had absorbed more of the basic dye than the aluminium tannate, and consequently I infer the absorption power of the tannic acid has been modified to a greater extent by the aluminium than by the antimony oxide.

Possibly useful results might be obtained by constructing a table showing numerically the relative influences of different metallic oxides on the absorption property of tannic acid for basic colouring matters.

Firstly, I propose to investigate the functions of the metallic oxide in particular in relation to the shade and fastness of the colouring matter, and then to apply the results obtained to explain the discrepancy.

To describe my experiments in detail would prolong this paper to an unnecessary extent, and therefore I will simply summarise the matter and state the results as briefly as possible.

It seemed advisable to examine each process separately, in order to isolate the better the most pertinent differences.

In the case of dyeing, the metallic oxide modifies the capability of the tannic acid to absorb or combine with the colouring matter. This may be shown by taking swatches of cotton cloth which contain exactly the same quantities of tannin but different metallic tannates, and dyeing in a basic dye solution. In every case a variation in the intensity of the shade is noticed. Further, the colour and transparency of the metallic tannate affect the shade of the colour lake. A notable example of the effect of the colour of a metallic tannate is the case of iron tannate.

The more transparent the metallic tannate, the purer is the shade of the colour lake, and yet the purity cannot be said to vary in direct proportion to the transparency of a metallic tannate, for the simple reason other factors exert a far greater influence on the colour. In one or two instances the colour of the metallic tannate chiefly affects the result, but more generally the specific cause is the influence exerted by the metallic oxide on the absorption power of the acid.

I did not observe any marked relationship to exist between the properties of a colour lake and the atomic weight of the metal it contained. The effect of the colour of the metallic tannate could always be predicted with certainty. The modification of the absorption property of the tannic acid was a matter for direct experiment. But, once ascertained for one colouring matter, its relative influence on any other colouring matter could be judged by analogy.

The variations in the transparencies of metallic tannates could only produce any visible effects on the purity of the shades when the metals formed white tannates. To ascertain the relative transparencies of these tannates the following method was adopted.

Into two Nessler tubes were placed 20 c.c. of a very dilute solution of tannic acid. To one was added an excess of a solution of a salt of the particular metal under examination, and to the other a slight excess of a solution of a salt of any metal whose tannate had been chosen as a standard of comparison. The slight excess of each solution was determined in a previous experiment. The contents of the tubes were now both made up to 50 c.c. with water and compared in the usual way for a colorimetric determination. By this method a coefficient of transparency may be determined for each metallic tannate, and hence the relative variations in the shades may be predicted owing to variations in the transparencies of the metallic tannates.

In the dyeing process the metallic oxide influences the shade produced from a basic colouring matter in the following ways:—

1. By a modification of the absorption property of the tannic acid.
2. By the colour of the metallic tannate itself.
3. By the transparency of the metallic tannate.

And the function of the fixing process is to produce an insoluble tannic acid compound on the fibre in order to prevent the tannic acid being washed away during the dyeing process.

In the printing process a thickened acetic acid solution of the tannic acid colour-lake is printed on the fibre and saturates the cotton fabric. On exposure to steam the acetic acid is driven off and the colour lake is precipitated through the structure of the cloth; so that the particles of fibre and colour-lake are now intimately mixed. The actual colour-producing compound is now on the fibre; and the subsequent conversion of this colour-lake into a triple compound containing a metallic base renders it faster to washing, soaping, &c., but does not materially affect the shade of colour.

It appeared to me an explanation of the similarity in colour between different metallic tannate lakes of the same colouring matter produced on the fibre by the above method might be given by supposing the function of the metallic oxide to be a means for the conversion of the excess of tannic acid into an insoluble compound, and hence to prevent its deleterious solvent action on the tannic acid colour-lake.

In the preparation of acetic acid solutions of these colour-lakes, there is always an excess of tannic acid used, to prevent the colour washing away during the soaping process to too great an extent.

If an excess of colouring matter were used, the soap bath would become coloured with the uncombined dye. The purity of lighter shades would consequently be much impaired. I was inclined to take the above view of the matter, because the tannate lake, being already precipitated in the cloth, might be expected to be tolerably fast to washing. Further, supposing the above speculations to be true, we should have in every case—no matter what metallic oxide had been employed as a fixing agent—the same colour compound on the fibre. Hence, we should expect to obtain similar colour results from different metallic oxides so long as their properties of colour, transparency, &c., fairly approximated.

There is still another point which might support this contention.

The colour tannate, being in an insoluble state, might not form a metallic compound, as it would when dissolved in some liquid medium.

Experiments were now made to test the accuracy of the above speculations. The tannate lake of tetraethyldiamidotriphenylcarbinol was prepared, and due precautions taken to insure the presence of an excess of the colouring matter. After being well washed with water a portion was digested in the cold in an aqueous solution of antimony potassium tartrate for 15 minutes, and then well washed. On this precipitate being examined it was found to consist of an antimony tannate lake insoluble in acetic acid. Swatches of cloth containing a tannate lake were treated with solutions of metallic salts, and after being well washed were boiled along with untreated ones in solutions of acetic acid of varying strengths. In every case a metallic tannate lake was found to exist on the fibre.

These results show the above explanation cannot be looked upon as wholly valid, although it cannot be denied one function of the fixing process is to precipitate the excess of tannic acid for the reasons previously given.

From the nature of the printing process it is obvious the fixing agent cannot influence the absorption power of the tannic acid, since the tannate lake—the colour-producing principle of the triple compound—is already on the fibre. Here is the essential difference between the two processes. In the printing process the colour-producing compound is on the fibre before fixing, and in the dyeing process the colouring matter is not assimilated by the tannic acid on the fibre until after fixing. The adequacy of this reasoning may be more easily appreciated by reducing the facts to an algebraic expression.

If we suppose the metallic tannate lake to be represented by the formula—

$a + b + c$, where
$a$ = the colouring matter,
$b$ = the tannic acid (mordant),
$c$ = the metallic oxide (fixing agent);

then the colour of the compound $a + b + c$ will depend upon the relative quantitative value of the factor $(a)$ to the whole molecule $a + b + c$.

Now in the dyeing method the numerical value of $(a)$ depends directly upon the value of $(b)$. This is obvious. But it is not easy to understand how variations in the values of $(c)$ could in themselves materially influence the colour effects. In my experiments $(b)$ remained a constant, the conditions were such that $(a)$ should be a constant, and $(c)$ was the variable quantity. Consequently the metallic tannate lakes possessing varying colour characteristics may be represented by the expressions—

$$a + b + c$$
$$a_1 + b + c_1$$
$$a_2 + b + c_2.$$

$c$, $c_1$, $c_2$ being the different metallic oxides and $a$, $a_1$, $a_2$ varying amounts of the same colouring matter. Now as these expressions represent my experimental results one is compelled to admit that the variations in colour are due to the variable factor $(c)$. And this must be owing to the modifying influence of $(c)$ on the absorption property of $(b)$ for $(a)$, resulting from the formation of the quantity $(b + c)$. Hence it is only necessary to take a given metallic oxide as a standard, and to determine by experiment for one colouring matter the variations in $a$, $a_1$, $a_2$ brought about by the introduction of the factors $c$, $c_1$, $c_2$ into the above expressions to obtain absorption coefficients for the combined factors—

$$(b + c),$$
$$(b + c_1),$$
$$(b + c_2), \text{ \&c.}$$

Then the relative values of $a$, $a_1$, $a_2$ for any colouring matter may be determined by calculation. Such results should be of interest and utility to the colourist.

It is important to notice in the dyeing process that the only constant factor as a necessary condition of experiment is $(b)$. The formation of the component $(b + c)$ follows, and the chromophorous factor $(a)$ is introduced last of all.

Now, in the printing method $(a + b)$ is always a constant as a necessary result of the modus operandi, and the only variable quantity is $(c)$; therefore we may represent the compounds as follows:—

$$a + b + c$$
$$a + b + c_1$$
$$a + b + c_2$$

But as the colour effects will depend almost entirely upon $(a + b)$, so long as the qualities of colour and transparency of $c$, $c_1$, $c_2$ practically approximate, the variations $c$, $c_1$, $c_2$ will not materially affect the results. Consequently all the above expressions will possess similar colour characteristics.

Therefore it is easy to understand how in the printing process the identities of $c$, $c_1$, $c_2$ are of little or no importance, whereas in the dyeing process they are the factors which determine the colour characteristics of the resulting colour lakes. As to what is the exact nature of the modification of the absorption property of tannic acid in the one case and not in the other, I am not prepared to express an opinion. The causes may be purely chemical or perhaps physical, but I should be rather inclined to surmise they would prove to be chemico-physical. However, it is certain this reversion of the method of production of metallic tannate lakes on the cotton fabric enables several metallic oxides to be employed as fixing agents in the one case and precludes their application in the other.

## New York Section.

*Meeting held at the Havemeyer Hall, Columbia University, on Friday, March 25th,* 1898.

PROF. CHAS. F. CHANDLER IN THE CHAIR.

Mr. C. E. **Tripler** gave some interesting experiments with liquid air. Liquid air boils fairly rapidly at ordinary temperatures, the nitrogen evaporating more readily than the oxygen, so that the liquid becomes more and more concentrated in oxygen.

Mr. W. H. **Birchmore**'s paper on the "Modern Development of Electrical Furnaces for the Laboratory" showed that when intense heat was employed the vapours of chromium were readily obtained so as to show the spectrum of this metal. A button of metallic chromium was produced.

### THE DETERMINATION OF TIN IN TIN PLATE AND ROOFING TIN.

BY ROBT. JOB.

In response to a number of inquiries, I submit the details of a method which we adopted several years ago for the determination of tin in roofing tin. By this method we are able to make a very accurate determination of tin in about 20 minutes. As will be seen, the method is simply an adaptation of the old titration with iodine in acid solution; but we have found that success or failure in this determination depends largely upon the care exercised in avoiding every possible chance of oxidation. The method has frequently been checked by gravimetric determinations, and has demonstrated its accuracy, provided that the details are carefully observed.

In our shipments, roofing tin is received in boxes containing 112 sheets, 20 × 28 ins., the black plate being evenly coated with not less than 16 lb. per box of a mixture composed of about one-third tin and two-thirds lead. The boxes when received are arranged in groups of five, and one sheet is taken at random to represent each group. A piece containing 4 sq. ins. is then cut from the centre of the sheet, and the tin determined as follows.

Add about 100 c.c. of C.P. hydrochloric acid, sp. gr. 1·20, to a 200-c.c. graduated flask; add then a piece of marble, about 5 grms.; cork with Bunsen valve, and let stand for about five minutes. Meantime, cut the 4 sq. in. strip of tin plate into pieces sufficiently small to pass easily into the flask. Remove the cork, drop in the tin, and replace the cork quickly. Heat until rapid action takes place, and then, when the plate is almost entirely dissolved, add as quickly as possible a small piece of marble and replace the valve. Then, the moment the plate is entirely in solution, while the effervescence from the marble still continues, cool down the flask as rapidly as possible until the solution is ice-cold and the salts have crystallised out. Remove the Bunsen valve from the flask, and fill to the mark with ice water, pouring carefully down the side to avoid air bubbles. Stopper the flask tightly, and mix thoroughly.

Take now a 50-c.c. pipette standardised against the flask, remove 50 c.c. of the solution into an 8-oz. Erlenmeyer flask, add a few drops of a fresh solution of starch, and titrate with standard iodine solution. Run in then into another flask this same amount of iodine required in the titration, remove another 50-c.c. portion of the stannous solution from the flask, place the end of the delivery tube just beneath the surface of the iodine solution, and let the greater part of the tin solution run into the iodine, raising the pipette just in time to catch the end drop. Add a few drops of starch, and finish the titration with the iodine. By this means oxidation by the air is completely avoided.

Calculate then from the iodine the number of grms. of tin per square inch of the roofing, and this number, multiplied by 0·98, gives the weight of tin in pounds per box of 112 sheets 20 × 28 inches.

In our practice the iodine solution is standardised at least once a week, and has approximately the following strength: 1 c.c. = 0·000560 grm. of tin.

# Scottish Section.

*Meeting held in Glasgow on Tuesday, April 5th, 1898.*

MR. G. T. BEILBY IN THE CHAIR.

## THE DEVELOPMENT OF OFFICIAL METHODS OF AGRICULTURAL ANALYSIS.

BY JAMES HENDRICK, B.SC., F.I.C.

If we look up a standard work on analytical chemistry of 50 years ago, we do not find much information on the analysis of manures. The first English edition of Fresenius, 1846, deals with the analysis of soils and of the ashes of plants, but manures are not mentioned. The fertiliser industry was then in its earliest infancy, and had not yet called for the development of special methods of analysis. Very soon after this date, however, we begin to find that the analysis of fertilisers is assuming importance, and textbooks on analysis begin to treat of it. In the third English edition of Fresenius, 1860, manure analysis is treated of at some length, and methods are given for the analysis of bones, guanos, and dissolved manures.

The methods too, though cumbrous, are substantially those which we use at the present day. We have altered and simplified details, but the main facts of nearly all our principal methods of determining nitrogen phosphates and potash in their various forms are already there. The only great method which has now received universal recognition, and which was not then known, even in principle, is the Kjeldahl method of determining nitrogen. In the case of phosphates the molybdenum method, the uranium method and the magnesium method are all described. But in order to obtain accurate results by any of these methods in the presence of such impurities as lime, magnesia, iron and aluminium, the manipulation is so cumbrous as to render them quite unsuitable for modern technical work.

Let us recapitulate the steps by which we have reached the stage of official agricultural analysts in this country.

At first all agricultural analysis was private work, and there were no special agricultural chemists. Then, as the use of artificial manures increased, and it became recognised that agriculturists were very open to fraud in the purchase of such substances, whose genuineness and quality they could not themselves judge, analysts were appointed by private associations of agriculturists to look after their interests. The great agricultural societies, for instance, appointed themselves official chemists, and analytical associations sprang up in various parts of the country. Finally, Government recognised that special legislation for the protection of purchasers of fertilisers and feeding stuffs was needed, and passed the Act of 1893, which provides for the appointment of a chief agricultural analyst by the Board of Agriculture, and of district agricultural analysts by local authorities, generally the County Councils. Thus we have evolved a whole hierarchy of official agricultural analysts, but with no organisation, no system, no uniformity.

Everyone proceeds by his own methods, good, bad, or indifferent, and it is not the business either of the central authority, or of the chief analyst, or of anybody else to give any guidance; neither is there any society or association in a position to do anything with authority.

Let us now glance for a moment at how official agricultural chemists have been evolved elsewhere. The two countries to which I shall particularly refer are Germany and the United States of America. Both of these are great manufacturing and commercial countries, and though agriculture is relatively more important with them than with us, especially in the case of the United States, yet they are peoples whose interests are not bound up in agriculture. In both these countries, as in nearly all other civilised states, special attempts have been made to bring science into relation to agriculture by the foundation of agricultural experiment stations. Some of these were, in their origin, private, or semi-private, institutions, but as they developed they have gradually become officially recognised, till now they are all more or less public institutions, largely supported by public money. While their original function was experimental research, it was very natural that the staffs of these stations should be made use of for purposes of control analysis. So in time it came about that all the official control analysis, corresponding to what is here done under the Fertilisers and Feeding Stuffs Act, and, to some extent at least, to that done under the Food and Drugs Acts also, was placed in the hands of the staffs of the experiment stations. In these stations there are skilled staffs consisting of chemists, bacteriologists, botanists, &c., whose primary work is agricultural experiment, but who also very fitly look after the composition and quality of all the artificial manures and feeding stuffs of their districts.

There is at present a considerable agitation in this country for the foundation of experiment stations corresponding to those abroad. It does not seem to be generally known to those who are promoting this laudable object that the work of foreign experiment stations is not generally confined to experimental research and instruction. The control work is also concentrated in them. This removes one of the great objections which is urged in certain quarters against our system; for those who carry out the control work are not engaged in ordinary commercial work, but merely in research and control. The laboratories of the research stations are thus comparable, as control laboratories, to the Government laboratory only, in this country. The appointment, not only of the director, but of every member of the staff, is subject to official control. The staffs are generally large, and specialisation is carried out to a high degree. The system not merely commands public confidence in a way which, unfortunately, ours does not seem to do, but tends to efficiency and economy.

The experiment stations soon felt the need of uniformity in their methods in carrying out their control work. Hence, in both America and Germany, associations were formed whose main object is to devise and test methods of analysis, so that certain methods of proved efficiency may be made official and adopted by all the members of the association. Such official methods, of course, obtain at once a certain position and authority even in the eyes of those who are not members of the association. Manufacturing chemists and commercial chemists, whose work is liable to be revised by the control chemists, naturally feel bound to take note of their methods. If our Government laboratory were to publish certain methods as its official methods, which it intended to make use of in its position as referee under the Adulteration Acts, those of us who are district analysts might not agree with those methods, or think them so good as our own, nor is it probable that we should be pleased to have these methods imposed on us by the authority of a single laboratory, but we would feel bound to take careful note of those methods, and even to use them in many cases. But when a powerful official association makes certain methods its official methods, and has them tested thoroughly, not in one, but in a number of different laboratories, till all the members of the association agree to the efficiency of the methods, and not only publishes the methods, but all the facts and figures concerning the tests by which their efficiency was proved, then such methods have not only to be accepted, but must be respected as well.

The American association, which is, I think, older than the German one, is the "Association of Official Agricultural Chemists." It meets annually, and its meetings are open to all analytical chemists, but only chemists of the central authority, the United States Department of Agriculture, and of the national and state experiment stations and agricultural colleges are eligible to membership, and only one representative, properly accredited, from each of these institutions is entitled to vote. The first object of the association is "to secure uniformity and accuracy of the methods, results, and modes of statement of analysis of fertilisers, soils, cattle foods, dairy products, and other materials connected with agricultural industry." No change can be made in any method of fertiliser analysis except by unanimous consent, till every official chemist having charge of fertiliser work shall have had opportunity to test the proposed change.

When it is determined to study any subject or method of analysis a "Reporter" and "Associate Reporter" on that subject are appointed. They secure the co-operation of as many official and other laboratories as possible for the work they have in hand. During recent years there have constantly been reporters on the following subjects: Phosphoric acid, nitrogen, potash, soils and ash, dairy products, foods and feeding stuffs, fermented and distilled liquors, sugar, and tannin. Each of these presents a report at the annual convention, which lasts three days, and at which all these subjects are discussed. The full proceedings of these conventions are published as an official bulletin of the United States Department of Agriculture. Everyone is thus enabled to see exactly how the association evolves its methods, and how thoroughly it tests them. Methods are changed very slowly, and only after very thorough discussion and examination in a number of independent laboratories. For instance, the reporters on potash have been engaged for several years in thoroughly testing their official "Lindo-Gladding" method and determining its exact limits of accuracy, and answering certain objections which had been made to it, chiefly in Germany. For the purposes of such work as this the reporters make up mixtures of known composition, containing all the impurities whose effect on the results they wish to test. Sealed bottles of these mixtures are sent out to all the laboratories which have undertaken to assist in the work, with exact instructions as to the method or methods of analysis to be followed. Usually two or more methods or modifications of methods are tested alongside of one another. The exact composition of the mixtures sent out is known only to the reporters. In some cases European as well as American laboratories join in the work. The results are all sent to the reporter together with a report as to any difficulties or points of interest noticed, or containing any suggestions as to improvements in details. From these the reporter prepares his general report, which is submitted, together with all the figures of all who have carried out the analysis, to the next meeting of the convention. If he thinks fit the reporter submits a motion suggesting improvements in the official method, or cancelling it altogether, or submitting another for it. This motion is discussed by the whole convention and, if necessary, voted on.

It is interesting to compare some of the results obtained in the early years of the association with those now obtained. There could be no better illustration of how far the association has attained its first object. In the early reports of the association the results obtained by different analysts of repute, working with methods well thought of and extensively used, often differed so widely as almost to shake one's confidence in analysis. But in recent years tests made by various laboratories, and especially when they are using the official methods of the association, exhibit an agreement which is all that could be desired, and must do much to give confidence both to the analyst himself and to the public.

The German association "Verband landwirthschaftlicher Versuchs-Stationen im Deutschen Reiche," has a somewhat narrower constitution, and proceeds by somewhat different methods. But it also tests all its methods of analysis, by somewhat similar means, very thoroughly, and adopts methods and makes changes only after lengthened deliberation and thorough independent examinations in the laboratories of its members. It also meets annually for discussion and report. Its proceedings are reported officially in "Die landwirthschaftlichen Versuchs Stationen."

In this country we have no official methods, and every one of us is a law unto himself. Where do we get our methods, and how do we test them? I suppose we get them from books, or we were taught them by our masters when we were students or assistants. Some of us devise methods of our own, and test them for ourselves to our own satisfaction. I expect the result would be interesting if a lot of us and our methods were submitted to such a test as the American association applies.

While there are, no doubt, most excellent methods of analysis in use by all reputable analysts, there are also, if one may judge by some of the methods which occur in popular text-books of agricultural analysis, methods of which one cannot speak so warmly. But, quite apart from absolute accuracy of method, it is a good thing to have uniformity of method. None of the methods for the estimation of, say, phosphoric acid in such impure materials as we have in fertilisers can lay claim to absolute accuracy. There are several very good methods and many very good modifications of each of these methods, though not one of them can be looked upon as perfect. Any one of these methods would be good and should give good and agreeable results within narrow limits. But if we all use different methods, even though all are good methods, we very greatly extend the chances of disagreement.

There is another class of methods where it is even more important to have uniformity. These are methods which do not determine a definable entity, such as phosphate or potash, but are merely comparative, and determine something variable in composition and various in nature. The results of such methods are comparable only when the work is carried out under absolutely uniform conditions. A good example of such a method is the determination of fibre in feeding stuffs. The fibre found is neither a physiological nor a chemical substance, but a group of substances characterised by a certain common insolubility. It cannot be defined except in terms of the method by which it is determined. It is not "indigestible" fibre, as it is so often inaccurately called in this country. Nor is it "woody" fibre, another most inaccurate and misleading term often applied to it. I cannot too strongly protest against the continued use of these misleading terms, even by chemists of the highest position and repute. The term "crude fibre" used by the Americans, corresponding to the "Roh Faser" of the Germans, is a much better term. Until we have so improved our methods of analysis as to be able to accurately determine either the chemical or physiological constituents of our feeding stuffs, any departure from uniformity of method means results which are not more accurate, but which are not comparable. Thus, to suggest, as some do, that it is more accurate to use hydrochloric acid than sulphuric acid in determining fibre, or that the solutions should be of $2\frac{1}{2}$ per cent. strength instead of the usual $1\frac{1}{4}$, or to insist on any other variation whatever from the usual method on grounds of greater accuracy, merely shows that the person making the suggestion quite fails to comprehend the nature of the method with which he is dealing. I have no doubt that there are methods of determining crude fibre more desirable than the one at present generally used, but unless we can insure the general adoption of one of them, so as to again secure uniformity of method, it is useless to suggest their use, and even if such a new method were generally adopted any advantage obtained would be purchased by rendering all the mass of already recorded results useless for purposes of comparison with results obtained by the new method.

It does not matter that the metre is not exactly $\frac{1}{10000000}$ of the distance from the equator to the pole so long as we all use the same metre. But if some people took to using altered and improved metres of their own while the rest of the world adhered to the old standard, we should have endless confusion. So it is, to a considerable extent, in chemistry. While many of our methods are fairly accurate instruments, many others only give comparative results, and for none of them can we claim absolute perfection, any more than for any other human instrument. While, then we ought to use as perfect an instrument as is possible in the circumstances, it is also most important that all our instruments should be standardised by the same standard. In all technical work it is most important that we should all work by the same standards, so that, as far as possible, our results should be strictly comparable. Even if our methods give only comparative, and not absolute, results, no harm is done so long as we all work by the same methods, for both buyers and sellers will be treated by the same unit of measurement and will adjust themselves to it.

One of the most crying needs of the agricultural chemistry of this country is an association, or court of reference, or some other body with sufficient powers and authority to command respect, to lay down standards and official methods of analysis. I do not think we shall ever have the agreement which is so desirable, or succeed in inspiring that public confidence in the trustworthiness of analysis which we would like, till we have some authoritative body to test methods and lay down official methods to be followed by all official chemists. As it is, we do not know, except, perhaps, in the cases of our personal friends or of the few chemists who have published their methods, by what methods our brother analysts, whose results may have to be compared with ours, work.

As our Government laboratory does not publish the methods which it uses in its position of reference laboratory, one naturally turns to Professor Thorpe's text book of quantitative analysis with interest, to see the methods given there. The only method given for determining the phosphates in manures is the volumetric uranium method. This is a method, so far as I know, not generally used in this country, or in any other country except France. As Professor Thorpe is the Principal of the Government laboratory, one would naturally be apt to draw the conclusion that this method is used by the Government chemists in their position as referees under the Fertilisers and Feeding Stuffs Act. I applied to the Government laboratory some time ago for information as to the methods which they use for fertilisers, and particularly for phosphates. I have to acknowledge the courtesy with which my queries were received and answered. I was at once informed that no official methods are recognised, but that in the case of phosphates the ammonium-molybdate method with reprecipitation with magnesia mixture, weighing as magnesium pyro-phosphate is usually followed. It is well to know, at least, that the Government laboratory follows a method generally recognised in this country. There are, however, a great many variations in details which can be introduced into the molybdate method. The work of foreign official associations, as well as of various individuals at home and abroad, has clearly shown that even small variations in details may cause distinct variations in results with the molybdate method. Hence, to get strictly comparable results, we need not only to follow the same general method, but to strictly observe uniformity in detail. In the case of the molybdate method, the method of making the solution, the temperature and conditions of precipitation, the method of washing, dissolving, and reprecipitating have all been shown to be of importance if strict uniformity of results is required. In laying down an official method it is not sufficient to agree upon a general method, all the details must also be carefully considered and accurately laid down for general adoption.

I do not know to what extent my brother analysts make use of foreign official methods, but I confess to a strong partiality for them. We can so easily find out how they have been arrived at in every detail, and how every detail of the method is justified. We can find just what difficulties have been encountered by a large body of independent analysts in working with them, and how those difficulties have been investigated and overcome. We can find within what limits they have proved accurate in well-defined cases, and in the hands of a considerable body of trustworthy analysts. I do not under-rate the work of those chemists at home who have given much time, care, and labour to working out and testing methods of analysis. I merely point out that they are at a great disadvantage as compared with the official system, which does more than the individual can possibly do. No matter how careful and accurate we may be, it is but natural that we should have a certain bias in favour of our own work. Not only can we not test our own work with absolute fairness, but we cannot foresee all the difficulties which will arise when it is placed in the hands of others. Which of us has not had the disappointment of trying some plausible method, enthusiastically spoken of by its author, and finding that in our hands it led to all sorts of difficulties. But when a method is tested by a large number of independent chemists of standing, all of whom are working under conditions which almost preclude personal bias, than we really get a method fully and fairly tested, and all its defects and difficulties laid open for improvement. Until then we have official methods of our own in this country those analysts are in my opinion wise who base their methods on the well-tested foreign official methods.

But, surely, it is not impossible for us to devise some machinery in this country by which official methods could be devised and tested so as to have the necessary authority and to command the necessary respect and confidence. Of course we must take into account the great difference of system in this country. We have no official experiment stations to act as a basis, and our recently created official agricultural chemists are not in a position at all comparable to that of their brethren abroad. A "Court of Reference" has long been wanted to make standards, &c., under the Food and Drugs Acts, and has received the approval of two special committees of the House of Commons, one of which has also approved of an excellent scheme for its constitution. Yet Parliament seems to be in no hurry to take the matter up. I do not think we can look for any help from the legislature for a long time to come in the formation of any official body to devise and test methods of agricultural control analysis. But a start might be made by an organisation of representative agricultural chemists.

This organisation might be included inside one of the already existing societies. If possible, it should secure the adherence and support of the Government chemists, though, judging from past experience, considerable difficulties might be experienced in obtaining this. But even without them, it would be useful if some working agreement could be come to among district agricultural chemists as to standards and methods of work.

## Discussion.

Dr. Wm. Frew said that the subject of control analysis was a very important one, and that steps should be taken by the Society of Chemical Industry to secure the recognition of official methods. The conditions of affairs in brewing analysis in this country was similar. The various brewing chemists used different methods, and, as a consequence, the results obtained by one chemist could not be compared with those obtained by other analysts, whereas on the Continent the matter of control analysis had been definitely settled by a convention held in Vienna some time ago, and the use of the standard methods established there rendered possible the comparison of the results of different chemists.

Mr. Macdonald suggested that the discussion of Mr. Hendrick's paper should be postponed, as several of the gentlemen directly interested had found it necessary to leave before having had the opportunity of expressing their views. He said that he could speak from the mercantile point of view, which was probably as important as that of the analytical chemist. It was an extraordinary state of affairs that a Government should make a law for the prosecution of people for selling stuffs and yet not make provision for obtaining a uniform and proper analysis. Anyone who had to deal in chemical manures felt this to be a grievance, and not only was there a loss of money involved, but there was a penal clause attached to the results which was positively shameful. It would not matter a single straw to a mercantile man whether the results were accurate or not, if he knew that he was buying a certain thing and could sell exactly on the same analysis. He knew what he was doing, but as the case stood now he bought, if he could, by a low chemist and he sold, if he could, by what was called a high chemist. It was a very nice game so long as it lasted, but, supposing he, by mistake or by misfortune, bought by a high chemist and sold by a low one, then there was sighing and sorrowing. The whole thing was ridiculous as it stood just now, and certainly some innovation needed to be started by the Government. He agreed with Dr. Frew in thinking that there was no society which was more capable of taking up this matter than their own Society, but he would suggest that it should not be confined to their Society. They required the association of other people. They sent to a foreign country, such as Germany or the West Indies, where there were professional chemists of reference. It was a serious matter indeed. They wanted a harmonious system, not only for this country, but for the world.

Mr. Biggart said that, while quite in sympathy with all the observations of Mr. Hendrick, there was one very important matter which he had overlooked, namely, the question of sampling. Even although they had a universal method to-morrow, if it was not preceded by sampling, it would all go for nothing. He had in his mind a case in point. He had a great deal to do with the question of sugar analysis; in fact, he might say that he had perhaps tested 40,000 or 50,000 tons of sugar in his day. Their method was to compare their tests against those of the foreigners, and there had been a great number of complaints against the methods in this country as being insufficient. A great deal had been done to throw light on the subject, and while they differed from them to a certain extent, if they worked on the same sample they got identical results, so that the difference which they complained of was not a difference in analysis but a difference in samples. Samples were taken on this side on landing, and the test was compared against the test on the other side as shipped, and so a difference came in, and they blamed them for those differences; and if they sent over their samples they could not make it any better, in fact they might be a little worse. While it was most desirable that the methods should be made common, still, at the same time, that must be preceded by a thorough and efficient method of sampling.

Mr. Carrick Anderson asked if the better way would not be to appoint a small committee from the section, with a view to their conferring with the other sections. He would suggest a committee of three.

Mr. Macdonald said he quite agreed with Mr. Biggart that the method of sampling was as necessary as a right method of analysis, but the Government failed to recognise that a sample which was drawn as carefully as possible was of no account. In selling manure a guaranteed analysis must be given for every bag. The thing was ridiculous. He had an experience which was one of a most painful kind. A cargo might come in, and it did not matter whether it was nitrate of soda or guano, but an official analysis was drawn, and yet, in spite of that, and that there was absolute proof of the stuff being delivered, they were bound to give an analysis for every 2-cwt. bag, and they were liable to be prosecuted penally for it, and run the risk of difference in the chemical result, showing that there was a necessity for dealing with a sample as much as dealing with an analysis. He would suggest that a committee should be formed.

Mr. T. Gray thought that Mr. Anderson's suggestion should be adopted, and proposed that a committee composed of Mr. Hendrick, Dr. Aitken, and Dr. Aikman should be appointed to act with the chairman and local secretary. He pointed out the necessity of securing the co-operation of the members of the other sections, and thought that they also should have an opportunity of contributing to the discussion. When they had read Mr. Hendrick's paper in the Journal the committee would be in a position to ascertain their views and to determine what further steps should be taken.

The nominations were seconded and unanimously adopted.

Mr. Henrick said he would be glad to act, and thought that the decision come to by the meeting was the best method of giving effect to the proposals made by him in his paper. The committee might put themselves into communication with the members of the other sections, and thus secure a general discussion of the subject. He was particularly anxious to hear the views of other analysts, and regretted that several of the members who were specially qualified to speak with authority on the subject had not found it possible to wait for the discussion.

Dr. A. P. Aitken wrote: Everyone who has had much experience with analysis of fertilisers and feeding stuffs must have felt the desirability of having some recognised authority to whom to appeal when the correctness of his analysis was challenged. It is one of the chief merits of the Fertilisers and Feeding Stuffs Act, 1893, that such an authority can be appealed to in the person of the chief analyst, and that at a small fixed fee. Unfortunately, however, his services are not available except in the restricted case of an analysis made by a *distant analyst*. As I read the Act, those who have not employed the distant analyst cannot secure the services of the chief analyst as a referee on any terms whatever. It is thus unfortunate that the one chemist in the country whose duty it is to be come master of the best methods of analysing fertilisers and feeding stuffs, and who is a highly-paid official, to whom time, trouble, and expense are matters of no importance, should be prevented by the conditions of his appointment from making his services available, except within somewhat narrow limits. Whether owing to that circumstance or (as we may fondly hope) to the rarity, now-a-days, of discrepancies in analytical reports his services are very rarely asked for. It would go far to meet one of the difficulties inherent in our system—or want of system—if the chief analyst were able to be appealed to by any analyst whose results were challenged. If, in addition to that, the chief analyst were to publish, let us say in the "Journal of the Board of Agriculture," a detailed description of the methods he employs, there would be little need for any futher departmental or professional organisation. Inaccuracies in analysis are due to many causes, and the analytical method is only one of these. The best methods will not secure accuracy unless they are carried out with the most conscientious care and with the exercise of more skill than is required for the mere strict adherence to a prescribed routine. I quite admit the desirability of having some organisation in this country for the purpose of discussing, devising, and testing methods of analysis of fertilisers, feeding stuffs, and agricultural products. We are all much indebted to the American and German organisations for the excellent work they have done and are doing in that direction. The fact that they are engaged constantly in that work doubtless makes it the less needful that we in this country should engage in it. The facilities they possess in their well-equipped agricultural colleges and equipment stations, supported out of national funds, enables them to devote their time to testing and devising methods of analysis without in any way encroaching upon their private time or their private purse. It is their natural duty and their state-paid work. Almost all the agricultural analyses of the country are done in these public institutions, and the fees charged are not intended to be remunerative. It is quite different in this country. There are no agricultural control stations here upheld by Government. Agricultural analyses are done chiefly by independent chemists and at as moderate a scale of fees as is charged by the state-aided control stations in Germany in many cases. It is a poor business and nothing but the most rigid economy can enable it to be carried on with a margin of profit. There is no doubt that that offers a strong inducement to adopt quick, cheap methods of analysis. The best methods are usually the most expensive, both as regards time and material. The employment of the best reagents and the maintaining of these in a state of purity requiring constant supervision and the added expense where the best material and skill are employed more than double the cost of analysis. When, in addition to that, the practice is adopted of making all analysis in duplicate, as is the practice in many laboratories it means the employment of a double staff of workers. The difficulty agricultural analysts have to face in this country is that of getting adequate remuneration for their work. It is hard to see how such analysts, unless they are in large practice, enabling them to carry on their work in batches and departments, or unless they are salaried officials or are otherwise provided for or assisted, can adopt the best methods of analysis. To ask an analyst who finds it difficult to "make both ends meet" to join an organisation in which he shall be required to spend time and money in testing methods of analysis and reporting thereon is not likely to meet with much success. Some time ago I undertook a series of analyses at the request of the American Agricultural Organisation. It occupied the whole time of one of my assistants for about two months and was the only work of the kind undertaken in this country. That is not to be wondered at. It is a very expensive thing, quite suitable for government laboratories, but beyond the means of a private analyst. By all means let us have an organisation to test and devise the best means of analysis of fertilisers and feeding stuffs, if we can get members to work in it, but we need not hope for the general adoption of their methods unless they can be shown to be cheaper and quicker than those in use, or unless the association can at the same time be effective in raising to at least double the usual amount the fees obtainable by agricultural analysts.

## THE "SPECKLING" OF MALTS.

BY WILLIAM FREW, PH.D. (MUNICH).

Some time ago I was consulted with regard to a certain malt which contained a large proportion of blackened corns; in the practical maltsters' parlance, it was "speckled." Those "speckled" corns presented the appearance of having been badly singed in certain places—so badly singed that the husk was quite black.

On closer examination, however, I found that this charring was altogether confined to the husk; the body (flour) of the blackened corns was no darker than that of the uninjured grains. On further inquiry I found that those black marks only appeared after the malt had been brought down to the lower floor of the kiln; that only those corns which lay on or near the (cast-iron) tiles showed these marks, and that "speckling" only became serious when the temperature rose above a certain degree. I was also told by a practical maltster who has worked both single and double floor kilns, that he had never seen "speckling" in malt made on the old-fashioned single-floor kilns, but that he had seen it often in malt dried on a double kiln having iron tiles on the lower floor. As far as I can discover, this speckling has, up to the present, been attributed to actual singing or scorching on the kiln. On examining the malt in question, however, I found that it was remarkably free from corns whose flour had been browned—the unmistakable evidence of overheating. I also found that it was impossible for actual scorching to have taken place as the temperature of the air below the bottom floor of the kiln had never risen above 185–190° F. (measured by a Sendtner's self-registering thermometer placed immediately beneath the lower floor).

At this stage it was suggested to me by Mr. Klingler, of Wellpark, that this charring was caused by the action of small quantities of free sulphuric acid present in the husk of certain corns, which by concentration and heat produced this blackening. His idea was that, owing to insufficient draught, the top layer of the green malt on the upper floor of the kiln remained moist for too long a period of time. During this time air containing a proportion of sulphurous acid (from the fuel) was passing through this wet grain, the husk of which, in its moist state, absorbed some of the sulphurous acid, which would be almost immediately oxidised to sulphuric acid. When those corns were brought down to the lower floor and exposed to a higher heat, concentration of the dilute sulphuric acid would take place, and at a certain heat, especially if the affected corns were in contact with the metal tiles, the husks would char.

It only remained now to obtain some evidence in support of this highly interesting theory. I found, on looking more closely at the black marks on the speckled malt, that the blackening resembled a stain more than the mark of burning. On applying the tip of the tongue to a blackened corn I was conscious of a sharp, acid taste, which was not evident in the case of an uninjured grain. Distilled water was dropped on to a piece of blue (non-bibulous) litmus paper; in certain of the drops blackened corns were placed and in others uninjured corns. The red stains in the case of the speckled corns were found to be very much more intense than those produced by the normal corns.

I then picked out 100 speckled corns and 100 uninjured ones from the same sample of malt and in each case boiled the unbroken corns with 50 c.c. of distilled water for the same length of time. On adding hydrochloric acid and barium chloride to the filtrates and boiling it was found that, whereas in the case of the normal corns practically no turbidity was produced, a distinct white precipitate formed in the filtrate from the blackened corns.*

The blackening then is due to the charring action of sulphuric acid on the cellulose of the husk, the sulphuric acid being produced by the oxidation of sulphurous acid which has been taken up by the moist husks from the furnace gases. The cause, of course, of this undue absorption is want of draught in the kilns. (This I know is the case with the kiln in question.)

I fear in many double-floor kilns in this country, sufficient attention has not been paid to the very important matter of draught. The designers too often forget that the air has to pass through two thicknesses of malt, and they, in consequence, arrange that the double-floor kiln shall pass only that amount of air which would suffice for a single-floor kiln. This explains why "speckling" seems to be so much more prevalent in malt made on double-floor kilns, whilst on single kilns it is hardly ever noticed, if at all.

A most significant fact in connection with "speckling" is that, as far as I know, it never occurs on a German air-kiln, where the products of combustion do not pass through the malt, whereas browning of the body of the corn can, and does, take place. I am also informed by the maltster before-mentioned that "speckling" is much more troublesome where coal is used instead of coke, *i.e.*, where the furnace gases contain more sulphurous acid.

The presence of this free sulphuric acid in a malt, even although it is only in small quantities, cannot but be injurious; and one longs for the time when the present antiquated and inefficient system of kiln drying malt will give place to one on more rational and scientific lines.

# Yorkshire Section.

*Meeting held on Thursday, March 24th, 1898.*

MR. THOS. FAIRLEY IN THE CHAIR.

## GLYCERIN ESTIMATION.

BY F. W. RICHARDSON, F.I.C., AND ADOLF JAFFE.

Nine years ago an admirable *résumé* of the chief methods in vogue for the estimation of glycerin was given by Otto Hehner (this Journal, 1889, 4—9), and to this summary, and to that also given in Benedikt and Lewkowitsch's valuable work upon "Oils, Fats, &c." (2nd Ed., 1898, 207, &c.), the inquirer may be referred.

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* Filter paper moistened with the filtrate from the blackened corns charred on being heated to a sufficient temperature; that moistened with the filtrate from the normal corns was hardly affected.

Our only apology for entering the arena of the glycerin controversy is our desire to outline what we believe to be brief and reliable methods for the analysis of crude glycerins and spent lyes.

We are strongly of opinion that for the analysis of crude glycerins, now so much in demand, there should be some official method. At the present time there would seem to be about as many processes as there are analysts, and we need not wonder that discordant results are frequently obtained, to the detriment of trade and the discredit of chemical science. Even with the same principles, different methods of working may give rise to serious discrepancies.

It has been our lot to analyse large numbers of spent lyes and crude glycerins, and we have been occasionally annoyed at the purchaser's rejection of a bulk of the last-named commodities because his analyst had found a fifth or a quarter of a per cent. less glycerin than ourselves!

The guarantee for crude glycerin, for the manufacture of dynamite, refers to an article containing not less than 80 per cent. of glycerin, and not more than 10½ per cent. of salts. In the light of such a guarantee, we have known a purchaser reject a delivery because his analyst had found 79·87 per cent. of glycerin! When we are made aware of the fact that no two of the best processes, in the ordinary way of working, give results more closely concordant than a quarter of a per cent., the necessity for some official method of working is sufficiently obvious.

As the rejection of a large bulk of crude glycerin by a purchaser is always a matter of expense and annoyance to the vendor, it is very necessary that absolute accuracy of the measuring apparatus and careful attention to a standard temperature should be observed, if the analysis is to be free from the suspicion of an error sufficient to cause the trouble referred to.

We have applied the different methods adopted for the estimation of glycerin, with special reference to crude glycerin and spent lyes, and have come to the conclusion that all others than the bichromate and acetin processes are utterly unreliable.

An analysis of a crude glycerin, an average sample of many bulks, made by ourselves may be of interest:—

| | | |
|---|---|---|
| Glycerin | 77·30 | |
| Fat acids (as soap) | 0·80 | |
| Soda ($Na_2O$) „ | 0·08 | 10·21 per cent. of "salts." |
| Sodium carbonate | 2·80 | |
| „ chloride | 6·56 | |
| „ sulphate | 0·34 | |
| Alumina and silica | 0·43 | |
| Water (with traces of organic colouring matters) | 11·69 | |
| | 100·00 | |

It is somewhat surprising that there should be any difficulty in the estimation of the salts present in a crude glycerin; yet, as Hehner has pointed out, different analysts do obtain very different results with the same sample. To estimate the salts, we evaporate the glycerin off from 2·5 grms. of the sample weighed in a platinum dish and placed over a low Argand flame; when the residue appears solid it is carbonised in the space of a few seconds at a higher temperature. The black ash is lixiviated, and the insoluble carbonaceous residue is collected upon a very small filter; after two or three washings, this is incinerated in the platinum vessel previously used, and when the operation is ended the clear colourless solution of the soluble salts is evaporated down upon the ash; after heating the residue over a low Argand flame for a few minutes, the salts are ready for weighing.

H. D. Richmond's method of sulphating the carbonised residue before incinerating, with a subsequent multiplication of the ash by 0·8 is, to say the very least, decidedly fortuitous, and although it may yield very good results in the majority of instances, we know of cases where its application would have led to serious error. The ordinary mode of procedure is to incinerate the glycerin residue over an Argand flame until the residue is white; this process is, in our experience, very slow, and if the operation is hastened in order that it may be expeditious, there is sure to be a very appreciable loss through the volatilisation of the salt.

The results obtainable by the simple method we described are always quite concordant, and we wish that some such process would become official.

The best and most expeditious of all methods for the estimation of glycerin is that depending upon complete oxidation by bichromate of potash in a solution containing a large proportion of sulphuric acid. Legler (Analyst, January 1887) and Hehner (Analyst, XII., 44, &c.) are the originators of this process. Legler based his calculations upon the loss of carbonic acid; Hehner determines the loss of bichromate by titration with iron—

$$3C_3H_8O_3 + 7K_2Cr_2O_7 + 28H_2SO_4 =$$
$$7K_2SO_4 + 7Cr_2(SO_4)_3 + 40OH_2 + 9CO_2$$

There can be no doubt that the latter method is at once the more accurate and expeditious, and also the one most applicable to dilute solutions of glycerin.

In our practice we have considerably modified the method originally adopted by Hehner.

For example, we find that it is quite unnecessary to heat for two hours with the bichromate; 20 minutes' time with our stronger solution is all that is necessary; again, we only need one bichromate solution.

### *Estimation of Glycerin in Crude Glycerin.*

25 grms. of the samples are made with ordinary water, up to 50 c.c. of solution, and of this 25 c.c. are taken, and precipitated with 7 c.c. of the official solution of basic acetate of lead (Liquor Plumbi Subacetatis B.P.). The mixture is filtered through a Swedish filter into a 250-c.c. flask. Repeated washings are made with about 150 c.c. of cold water. The excess of lead (which should be small) is precipitated by an excess of dilute sulphuric acid. After making to the mark and shaking, the liquid is poured on to a dry Swedish filter, 20 c.c. of the filtrate (representing 2 grms. of the original sample of crude glycerin) are pipetted into a beaker, the mouth of which is closed by a funnel with short stem; then 25 c.c. of Hehner's standard bichromate solution are added; finally, 25 c.c. of pure sulphuric acid are cautiously mixed with the other fluids. After 20 minutes' heating in a water-bath, the oxidation is complete. After cooling, the liquid is made to 250 c.c. with tap water, and this solution is then titrated upon 20 c.c. of a solution containing 2·982 per cent. of the double sulphate of iron and ammonia, using ferricyanide of potassium to determine the end reaction in the usual manner.

The portion of the iron solution taken represents 0·01 grm. of glycerin; therefore, if A is the number of c.c. of the bichromate mixture required, and $x$ the percentage of glycerin sought, we have the simple formula—

$$x = (0{\cdot}25 - \frac{250}{A} \times 0{\cdot}01) \times 500$$

(0·25 is the equivalent of glycerin represented by the 25 c.c. of bichromate used, containing 74·86 grms. per litre).

The appended table gives the percentages of pure glycerin corresponding to the measures of bichromate solution required.

In the case of a spent lye we take 2·5 grms. and dilute to 50 c.c.; the precipitation of chlorides and organic impurities is effected by the addition of a slight excess of the solution of basic lead acetate, and the operation proceeds as in the case of the crude glycerin, with the exception that the lead sulphate is filtered off and the filtrate is concentrated to about 25 c.c. before the addition of the bichromate and the sulphuric acid is made.

*Table for Percentages of Glycerin from Crude Glycerins.*

| Solution. | Glycerin. | Solution. | Glycerin. | Solution. | Glycerin. |
|---|---|---|---|---|---|
| C.c. | Per Cent. | C.c. | Per Cent. | C.c. | Per Cent. |
| 24·0 | 72·95 | 25·8 | 76·55 | 27·6 | 79·71 |
| 24·1 | 73·13 | 25·9 | 76·74 | 27·7 | 79·90 |
| 24·2 | 73·35 | 26·0 | 76·93 | 27·8 | 80·04 |
| 24·3 | 73·55 | 26·1 | 77·10 | 27·9 | 80·20 |
| 24·4 | 73·75 | 26·2 | 77·29 | 28·0 | 80·36 |
| 24·5 | 73·98 | 26·3 | 77·47 | 28·1 | 80·52 |
| 24·6 | 74·19 | 26·4 | 77·65 | 28·2 | 80·77 |
| 24·7 | 74·39 | 26·5 | 77·835 | 28·3 | 80·85 |
| 24·8 | 74·59 | 26·6 | 78·01 | 28·4 | 81·00 |
| 24·9 | 74·80 | 26·7 | 78·18 | 28·5 | 81·15 |
| 25·0 | 75·00 | 26·8 | 78·36 | 28·6 | 81·30 |
| 25·1 | 75·20 | 26·9 | 78·53 | 28·7 | 81·45 |
| 25·2 | 75·40 | 27·0 | 78·70 | 28·8 | 81·60 |
| 25·3 | 75·60 | 27·1 | 78·77 | 28·9 | 81·75 |
| 25·4 | 75·79 | 27·2 | 79·04 | 29·0 | 81·90 |
| 25·5 | 75·98 | 27·3 | 79·21 | 29·1 | 82·05 |
| 25·6 | 76·17 | 27·4 | 79·38 | | |
| 25·7 | 76·36 | 27·5 | 79·55 | | |

The table gives the percentages of glycerin directly from the measures of bichromate required to oxidise the iron :—

*Table for Percentages of Glycerin from Spent Lyes.*

| $K_2Cr_2O_7$ | Glycerin. | $K_2Cr_2O_7$ | Glycerin. | $K_2Cr_2O_7$ | Glycerin. |
|---|---|---|---|---|---|
| C.c. | Per Cent. | C.c. | Per Cent. | C.c. | Per Cent. |
| 12·6 | 2·064 | 19·4 | 4·90 | 26·6 | 6·24 |
| 12·8 | 2·187 | 19·8 | 4·95 | 26·8 | 6·27 |
| 13·0 | 2·308 | 20·0 | 5·00 | 27·0 | 6·30 |
| 13·2 | 2·428 | 20·2 | 5·05 | 27·2 | 6·32 |
| 13·4 | 2·540 | 20·4 | 5·10 | 27·4 | 6·35 |
| 13·6 | 2·648 | 20·6 | 5·14 | 27·6 | 6·37 |
| 13·8 | 2·756 | 20·8 | 5·17 | 27·8 | 6·40 |
| 14·0 | 2·860 | 21·0 | 5·21 | 28·0 | 6·43 |
| 14·2 | 2·96 | 21·2 | 5·28 | 28·2 | 6·45 |
| 14·4 | 3·05 | 21·4 | 5·32 | 28·4 | 6·48 |
| 14·6 | 3·15 | 21·6 | 5·37 | 28·6 | 6·50 |
| 14·8 | 3·24 | 21·8 | 5·41 | 28·8 | 6·54 |
| 15·0 | 3·33 | 22·0 | 5·45 | 29·0 | 6·55 |
| 15·2 | 3·42 | 22·2 | 5·49 | 29·2 | 6·57 |
| 15·4 | 3·508 | 22·4 | 5·53 | 29·4 | 6·60 |
| 15·6 | 3·59 | 22·6 | 5·57 | 29·6 | 6·62 |
| 15·8 | 3·67 | 22·8 | 5·61 | 29·8 | 6·64 |
| 16·0 | 3·75 | 23·0 | 5·65 | 30·0 | 6·66 |
| 16·2 | 3·82 | 23·2 | 5·69 | 30·2 | 6·69 |
| 16·4 | 3·90 | 23·4 | 5·72 | 30·4 | 6·70 |
| 16·6 | 3·97 | 23·6 | 5·76 | 30·6 | 6·74 |
| 16·8 | 4·04 | 23·8 | 5·80 | 30·8 | 6·75 |
| 17·0 | 4·12 | 24·0 | 5·83 | 31·0 | 6·76 |
| 17·2 | 4·18 | 24·2 | 5·86 | 31·2 | 6·79 |
| 17·4 | 4·25 | 24·4 | 5·90 | 31·4 | 6·81 |
| 17·6 | 4·32 | 24·6 | 5·93 | 31·6 | 6·83 |
| 17·8 | 4·38 | 24·8 | 5·96 | 31·8 | 6·85 |
| 18·0 | 4·44 | 25·0 | 6·00 | 32·0 | 6·87 |
| 18·2 | 4·50 | 25·2 | 6·03 | 32·2 | 6·89 |
| 18·4 | 4·56 | 25·4 | 6·06 | 32·4 | 6·91 |
| 18·6 | 4·64 | 25·6 | 6·09 | 32·6 | 6·93 |
| 18·8 | 4·68 | 25·8 | 6·12 | 32·8 | 6·95 |
| 19·0 | 4·74 | 26·0 | 6·15 | 33·0 | 6·97 |
| 19·2 | 4·79 | 26·2 | 6·18 | 33·2 | 6·98 |
| 19·4 | 4·84 | 26·4 | 6·21 | 33·4 | 7·00 |

We will give three typical results to show how quickly the oxidation is effected under the stated conditions:—

| — | At 210° F. | Per Cent. of Glycerin. |
|---|---|---|
| | Minutes. | |
| 1 | 5 | 77·15 |
| 2 | 15 | 77·30 |
| 3 | 25 | 77·30 |

We have not found it necessary to treat the solution of the crude glycerin with silver oxide, as recommended by Hehner; indeed, we have been somewhat surprised to find that, on the whole, the amounts of glycerin found have been rather larger with silver oxide and lead subacetate than with the latter substance alone. The lead removes every trace of colouring matter, and in our mode of procedure the chlorides never interfere.

Artificial crude glycerins were made by dissolving quantities of salt and soap varying from 2½ and ½ to 7½ and 1 per cent. respectively with 0·097 grm. of glycerin; the quantities of glycerin found were 0·095, 0·095, and 0·099.

Pure glycerin-free soap solutions were made with the fat acids of cocoanut oil, cotton oil, and sheep fat; after decomposing the soaps with sulphuric acid and filtering, the filtrates reduced no bichromate. No doubt some of the soluble fat acids (butyric, &c.) from the cocoanut oil were present in the filtrate; but, as Johnstone (Chem. News, LXIII., p. 111) and others have shown, boiling acid bichromate solutions are not changed by butyric and the lower fat acids.

It may be asked why we do not prefer the acetin to the bichromate method, as the former is certainly more exclusive in its principle, the triacetyl derivative of glycerin being a definite compound not obtainable from other non-glyceride organic matters. We esteem the acetin process highly, but consider that for general laboratory purposes it requires too much attention. If exact conditions are not observed, conflicting results may be obtained. A commercially pure glycerin of 1·2601 sp. gr. at 60° F. gave, with the acetin method, when most carefully conducted, 97·16 per cent. of glycerin by the acetin process, but when strict attention to a number of apparently minor details was not observed, only 96·0 per cent. of glycerin was obtained. Moreover, the bichromate method, as we apply it, gives much less trouble and requires no special attention to minor details, as the bichromate solution is very stable, and, after oxidation, any length of time may lapse before titration is effected.

It is stated that even in this day there are analysts who use the lead oxide process for glycerin estimation; yet it is very obvious that such a method can only be used with pure glycerin solutions, if any approach to accuracy is desired.

A commercially pure glycerin, dried upon 20 times its weight of lead oxide at 260° F., gave us 97·95 per cent. of glycerin—a result sufficiently near the truth (97·27 per cent. by Lenz's table at 60° F.), but a crude which yielded 77·3 per cent. to bichromate (as confirmed by the acetin method) gave us 81·47 per cent. by lead oxide, after allowing, of course, for the salts present. There was 0·88 per cent. of pure dry soap present in this glycerin, but even this would not reduce the error below an excess of 2½ per cent. of glycerin.

Glycerin, heated with lead oxide to about 130° C., loses a molecule of water, $C_3H_8O_3 - OH_2 = C_3H_6O_2$; therefore the glyceryl residue must be multiplied by $\frac{92}{74}$ or 1·243, to give the equivalent of glyceryl hydrate, *i.e.*, glycerol.

To use this process with crude glycerins and spent lyes, it would be necessary to concentrate and extract with absolute alcohol, the alcoholic solution of glycerin being finally evaporated down upon the lead oxide. In our experience such a procedure is not only tedious, but too often liable to give inaccurate results. Sodium chloride and organic matters, and of course soapy and fatty substances, are all more or less soluble in alcohol, and contribute their quota towards rendering the alcoholic extraction *plus* lead oxide evaporation very unreliable. We have experimented with different modifications of the permanganate method for the estimation of glycerin, and we unhesitatingly discard them all in favour of the processes described. There are so many objections to the Fox and Wanklyn process, even as worked out by Benedikt and Zsigmondy, that we forbear from wearying you by an enumeration of them. For general purposes the alkaline permanganate has the great objection that, unlike the acid bichromate, it converts the lower fat acids (butyric, &c.) into oxalic acid, and this result, in the case of crude glycerins or spent lyes from curd soaps, or indeed any soaps made partly from cocoanut oil, will lead to serious inaccuracies.

Cross, Bevan, and Gantter measure the carbonic acid evolved, when the glycerin is oxidised, by acid bichromate. Now, while it is certain that excellent results may be obtained in this way, we consider such a process not nearly so well adapted for the exigencies of general laboratory practice as the method we have described.

Legler's procedure seems very simple, as he uses a Mills's carbonic acid apparatus, and estimates the carbonic acid by loss. As we are firmly of opinion that the ideal carbonic apparatus—one which will really allow *only* of the loss of the whole of the $CO_2$—has not yet been invented, we are very dubious as to the accuracy of any results so obtained.

For rough purposes, where a mere general discrimination is required, or even where some check upon analytical data obtained in the ordinary way is needed, we have worked out a formula for calculating the percentage of glycerin from the specific gravity of, and the percentage of salts (obtained as we have described) in, the crude article. The organic impurities, such as colouring and saponaceous substances, require that a special number or constant for deduction from the specific gravity of the crude glycerin should be assumed; this number we find to be greater in proportion as the glycerin present is smaller.

A. With crude glycerins with from 78 to 83 per cent. of glycerin we deduct ........................ 0·012
B. With crude glycerins with from 77 to 65 per cent. of glycerin we deduct ........................ 0·015
C. With crude glycerins with from 60 to 50 per cent. of glycerin we deduct........................ 0·023

We multiply the percentage of salts present by 0·0073, and to this add A, B, or C, and then deduct the sum from the specific gravity of the crude glycerin taken at 15·5° C. For example:—

A crude glycerin yielded 10·96 per cent. of salts and a specific gravity of 1·3048 at 60° F.; here we know from tabulated results that the percentage of pure glycerin is but little less than 80 per cent.; therefore we use the number A:—

$$A + (0{\cdot}0073 \times 10{\cdot}96) = 0{\cdot}0920$$

$$1{\cdot}3048 - 0{\cdot}0920 = 1{\cdot}2128$$

By Lenz's table, calculated to 15°·5 C. by Richmond, this gravity represents 79·39 per cent. of glycerin; the actual estimation gave 79·25 per cent.

A few comparisons taken from a series of crude glycerin analyses, without making any selection, give the following interesting and, on the whole, satisfactory data:—

| Specific Gravity of Crude Glycerin at 15°·5 C. | Found. | | Calculated. | | |
|---|---|---|---|---|---|
| | Per Cent. Salts. | Per Cent. Glycerin. | Specific Gravity of Glycerin Solution. | Per Cent. of Glycerin. | Error. |
| 1·3050 | 10·96 | 79·50 | 1·2130 | 79·46 | −0·04 |
| 1·3009 | 10·40 | 79·40 | 1·2120 | 79·11 | −0·29 |
| 1·3053 | 10·90 | 81·60 | 1·2133 | 79·68 | −1·92 |
| 1·3075 | 10·20 | 83·60 | 1·2200 | 82·14 | −1·46 |
| 1·2988 | 10·08 | 79·05 | 1·2132 | 79·54 | +0·49 |
| 1·2575 | 14·00 | 50·50 | 1·1323 | 51·20 | +0·70 |
| 1·2950 | 11·48 | 73·67 | 1·1960 | 73·25 | −0·42 |
| 1·2916 | 11·60 | 70·00 | 1·1919 | 71·70 | +1·70 |
| 1·2950 | 11·69 | 73·72 | 1·1953 | 72·93 | −0·79 |
| 1·2990 | 11·04 | 75·00 | 1·2034 | 75·89 | +0·89 |
| 1·3018 | 10·90 | 76·55 | 1·2072 | 77·28 | +0·63 |
| Average result | .. | 74·79 | .. | 74·73 | −0·06 |

These calculations cover crude glycerins containing from 50 to 83·6 per cent. of glycerin and 10 to 14 per cent. of salts, and the greatest error is only 2 per cent.

With a mixture of a large number of crude glycerins, the percentage found was 77·30; the glycerin by calculation amounting to 78·3, or an error of *plus* 1 per cent.

### Discussion.

Mr. G. Ward thought it most desirable that a generally accepted method, which might be called official, should be adopted in the estimation of glycerin. Differences crop up in the analyses, due, not to want of skill or care, but owing to the process employed. He approved of the bichromate method, because the reagent was a stable one, and it was generally available.

Mr. H. Broadbent expressed his thanks to the authors of the paper. In testing, his experience had been chiefly with pure glycerins, and whilst paying a tribute to the quality of pure glycerins now made by the best manufacturers, he had observed that glycerin kept fo some time occasionally acquired a dark or blue-black colour, and should like to know the probable cause of this coloration.

Mr. Ward had also observed this colour, but found that it only occurred in bottles full of glycerin closed with corks. He thought that the colour was caused by tannin from the cork acting on minute traces of iron in the glycerin.

Mr. B. Watmough agreed with the authors of the paper respecting the acetin method, and the precautions necessary to obtain reliable results. He thought the rough approximation by calculation from the specific gravity, allowing for the salts present, might also be very convenient as a check.

Mr. Fairley stated that, in his experience, the bichromate and the acetin methods were found to be the most accurate and reliable. He thought the modifications in detail, made by the authors in the working of the bichromate method, might prove most convenient and useful. He suggested the use of boiled or oxygen-free water in place of tap-water.

Mr. Richardson, in reply, stated that he had not had much experience with pure glycerins. He thought that the explanation given by Mr. Ward respecting the probable cause of colour in keeping certain glycerins was a very likely one. From the very small amount of free oxygen introduced by adding tap-water, he thought that the difference due to the use of tap-water would be so minute as to be of no practical importance.

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## I.—PLANT, APPARATUS, AND MACHINERY.

*The Filling of Cylinders with Compressed Carbonic Acid Gas.* Imp. Inst. J., April 1898, 4, [40], 106—107.

Some official correspondence with the Government of India having taken place as to the conditions under which cylinders of compressed carbonic acid gas would be safely carried by railway in India, Sir Frederick Abel, in a letter to the Under Secretary of State for India, suggested the carrying out of experiments by Prof. W. C. Unwin, to determine the pressures in cylinders of compressed gas at various temperatures. Sanction was given for these experiments, at Sir Frederick Abel's request. The following is a short account of the experiments and the results of the investigations, which were carried out at the Central College of the City and Guilds of London Institute for Technical Education:—

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* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Physical Properties of Carbonic Acid Gas.*

At atmospheric pressure and 32° F., the density of carbonic acid is 1·529 and its co-efficient of dilatation is 0·002067. Hence, 1 cb. ft. weighs 0·1234 lb., and 1 lb. occupies 8·102 cb. ft. The value of P.V. under these conditions is 17,145 foot-pounds.

As to the condition of carbonic acid at other temperatures, elaborate investigations have been made by Amagat, which are contained in a number of papers in the *Annales de Chimie et de Physique*, and the *Comptes Rendus* of the Academy of Sciences. The most important of these for the purpose of this investigation is the paper in the *Annales de Chimie et de Physique*, 3 serie, tome 22, 1881. In this there is a table giving values of P.V. for carbonic acid at pressures from 30 m to 320 m. of mercury and at temperatures from 18° to 100° C. From this table it is possible to find the volume of carbonic acid at any pressure and temperature within a wide range.

*The Experimental Investigation.*

For the conduct of these experiments, a pit was sunk in which a tank of water could be placed. A steam-pipe was led to the tank, so that the water in the tank could be heated to any required temperature. The cylinder of carbonic acid was placed in the tank, and gradually heated by admission of steam at intervals. From time to time the temperature of the water in the tank, and the corresponding pressure in the cylinder, was noted. The cylinder used was a steel oxygen-cylinder of rather extra strength, with an opening at each end to facilitate filling with water for calibration. From three weighings it was found that the cylinder contained 21·947 lb. of water, and that its volume was 0·3518 cb. ft. A powerful stirring apparatus, consisting of a vertical shaft with three sets of paddles, was worked almost continuously in the tank to ensure, as far as possible, uniformity of temperature.

The following values of the pressures for various proportions of filling of the cylinder were obtained:—

*Pressure in Cylinders at Different Temperatures and Proportions of Filling.*

| Temperature, 0° F. | Proportion of Filling in Pounds of Carbonic Acid per Pound of Water Capacity. | | | | |
|---|---|---|---|---|---|
| | ⅞ | ¾ | ⅔ | ⅝ | ½ |
| | Pressures in Atmospheres. | | | | |
| 90 | 200 | 104 | 83 | 80 | 77 |
| 100 | .. | 127 | 101 | 94 | 85 |
| 110 | .. | 153 | 118 | 109 | 96 |
| 120 | .. | 180 | 135 | 124 | 107 |
| 130 | .. | 211 | 153 | 140 | 117 |
| 140 | .. | .. | 170 | 156 | 128 |
| 150 | .. | .. | 190 | 172 | 139 |

It may be taken as certain that the cylinders used for compressed carbonic acid in India are obtained on a specification never more stringent, and sometimes less stringent, that the cylinders used for compressed oxygen in this country. Further, that they are never stronger or thicker than oxygen cylinders of the same size. Now, oxygen cylinders are regularly filled, and are certified as so filled, at a pressure of 120 atmospheres; and, under rather exceptional conditions of temperature, they may be exposed to a pressure of, at most, 140 atmospheres. This pressure may therefore be accepted as the limit of safety for such cylinders, according to the practice of this country.

If this limit of 140 atmospheres be assumed, then it appears that carbonic acid cylinders filled with ⅞ lb. per lb. of water capacity will be unsafe at quite ordinary temperatures. When filled with ¾ lb. per lb., they become unsafe at a temperature between 100° and 110° F. When filled with ⅔ lb. per lb., they become unsafe at a temperature of 121° F. When filled with ⅝ lb. per lb., they become unsafe at 130° F. Lastly, when filled with ½ lb. per lb., they become unsafe at 150° F. Within the limits of temperature above stated, the carbonic acid cylinders would be as safe as, but not safer than, the oxygen cylinders largely used in this country. It is stated in the correspondence that in India, for short periods in certain localities, the temperature may reach 180° F. If that be so, a cylinder filled with only ½ lb. per lb. of water capacity would be strained up to its test pressure. Perhaps it is more reasonable to take 155° as a temperature to which cylinders may certainly at times be exposed in India. Then the pressure would not only exceed the limit of working pressure hitherto laid down as the safe limit, but would very nearly reach the test limit, when the cylinder was filled with ⅔ lb. per lb. of water capacity. It is certain, therefore, that at any rate ⅔ lb. per lb. of water capacity is quite the maximum which should in any case be allowed, and that, even so, pressures may occur beyond those hitherto admitted to be allowable or safe.

If it is assumed that, under the highest temperature to which the cylinders are liable to be exposed in India, the pressure in the cylinders should not exceed 120 atmospheres, or, say, 1,800 lb. per square inch, the following are the minimum thicknesses of cylinders which should be used:—

*Thickness of Cylinders in Inches.*

| External Diameter in Inches. | Lap-welded Wrought Iron. | Lap-welded Steel. | Seamless Steel. |
|---|---|---|---|
| 4 | 0·22 | 0·19 | 0·18 |
| 5½ | 0·30 | 0·27 | 0·25 |
| 6 | 0·33 | 0·29 | 0·27 |
| 6·8 | 0·375 | 0·33 | 0·31 |
| 7 | 0·39 | 0·34 | 0·32 |
| 8 | 0·44 | 0·39 | 0·37 |

With such cylinders no danger need be apprehended if the pressure occasionally reached 140 atmospheres. They need not even be expected to burst at 200 atmospheres, although then the margin of safety would practically have vanished.

Judging by the results of these experiments, it may certainly be inferred that the limit of filling assigned by the Home Office Committee of ⅔ lb. of gas per lb. of water capacity for cylinders in the tropics is the greatest amount of filling consistent with safety. Further, if the temperature should actually rise to 180°, as has been stated, even the filling to that amount would be dangerous. It appears, therefore, that the reduction of the limit of filling from ¾ lb. per lb. to ⅔ lb. per lb., which appears to have been adopted in India, is reasonable.

It is doubtful whether the stated amount of filling of these cylinders is actually determined by the carbonic acid gas manufacturers quite as accurately as they suppose. Instances have occurred in which the actual filling of cylinders differed from the reputed filling by quite ¼ lb. The filling has to be determined by the difference of weight of the cylinder empty and full, and, since the gas weighs much less than the cylinder, that difference is necessarily liable to much greater proportional error than the weighings of the cylinder.—A. S.

*Drying-Installations, Modern; Physical Bases for.* Pfeifer. Thonind. Zeit. 22, [10], 97—99.

From the progressively decreasing power possessed by air of absorbing moisture as the temperature falls, it follows that low initial temperatures are uneconomical for drying, owing to the relatively large volume of air required. The maximum effect is obtained by employing hot air at a high initial temperature, and allowing it to remain in contact with the mass to be dried until saturated with moisture. From experimental results, plotted as curves, it is concluded that when the heated air will come in contact with any ironwork in the drying chamber, the maximum initial temperature permissible, is 200°—300° C., the effluent air under favourable conditions escaping, saturated with moisture, at a temperature of 50° C. A higher final temperature is uneconomical, since the full evaporative power is not utilised. The results alluded to show that 1 kilo. of coal, with a calorific power of 7,200 heat units is theoretically capable of evaporating 7·5 to 11 kilos. of water, according to the temperature employed; the higher this temperature, the greater the evaporative power, and the lower the mechanical power required to remove the air from the apparatus. Low initial temperatures are economical only when the substance treated cannot withstand heat, or where cheap or waste sources of heat are available. In cases in which furnace gases are used in place of air, the physical conditions are practically the same, the water vapour formed during combustion being taken into account.—C. S.

## PATENTS.

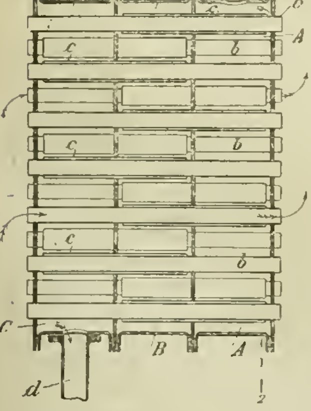
*Superheating Steam; Impts. in Apparatus for.* S. Chatwood, Newgate Street, and S. R. Chatwood, Bolton. Eng. Pat. 1494, Jan. 19, 1897.

The superheater consists of three superposed horizontal fire-brick chambers, through which the products of combustion from a fire-box below pass in succession backwards and forwards. In the two upper chambers are laid horizontal superheating pipes through which the steam is passed, entering the tubes in the top chamber at the coolest part, and passing to the hottest part of the middle chamber. In the top chamber the pipes are directly exposed to the flame, but in the lower they are mainly heated by radiation from the walls and roof. By another contrivance, the chambers and superheating pipes are vertically arranged. The pipes have a partition or partitions so placed as to admit of two or more superheating operations being carried on at one time.—R. S.

*Superheating Steam; Impts. in Apparatus for.* S. Chatwood, Newgate Street, and S. R. Chatwood, Bolton. Eng. Pat. 2650, Feb. 1, 1897.

The pipes, through which steam is passed to be superheated, and which are heated on the outside, are provided internally with spirally-twisted bars, obstructions, inclined vanes, or central bars, whereby the steam receives a rotary motion, is mixed or agitated, or is forced against the sides of the pipes.—R. S.

*Superheating Steam, A New or Improved Means for.* J. Grubinski, Warsaw. Eng. Pat. 25,237, Oct. 30, 1897.



The apparatus comprises a casing divided by partitions into three compartments, A, B, C, and provided with a steam inlet *a* and outlet *d*, flues *b* passing through the casing and partitions, and open-ended tubes *c* wider than the flues and surrounding the latter, these tubes serving to connect adjacent compartments of the apparatus. The steam entering at *a*, and passing downwards to the outlet *d*, is thus caused to pass through the annular spaces between the tubes *c* and flues *b*.—R. A.

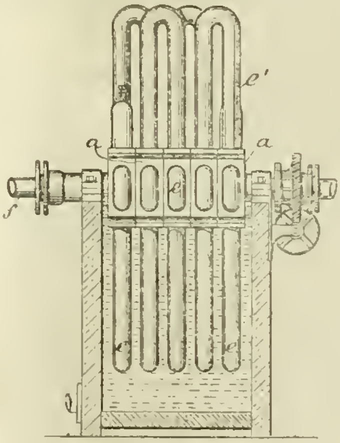
*Steam and Vapours, Impts. in and connected with Apparatus for the Condensation of.* J. Klein, South Parade, Manchester. Eng. Pat. 23,846, Oct. 16, 1897.

The apparatus consists of a condenser, a cooling arrangement for the condensing water placed above or alongside the condenser, a tank for receiving the condensing water, and a single pump for circulating the water from the tank through the cooler, &c. When the cooler is placed above the condenser, the water is delivered by the pump into the top of the cooler, from which it passes, over the condenser, into the tank. When the cooler and condenser are side by side, the delivery stream of condensing water is divided, so as to flow partly over each. The condenser may also be enclosed in a vertical shaft or chimney provided with an air-inlet at the base.—R. A.

*Corrosive Liquid [Sulphuric Acid, &c.], An Improved Tank for Conveying in Bulk.* J. V. Paterson, Coin Street, London. From J. Paterson, and A. J. Oke, both of Cape Town, South Africa. Eng. Pat. 3491, Feb. 10, 1897.

The tank is constructed with an outer shell of steel or iron, and with an inner lining, spaced apart from the shell, of a non-corrodible alloy of lead, with from about $\frac{1}{2}$ per cent. to 5 per cent. of antimony or copper. The joints of the lining are chemically burnt, and the space between it and the shell is packed with any suitable material which is insoluble in the liquid. The cover of the tank is similarly constructed. The bottom of the tank is formed with a well, from which the liquid may be conveniently pumped. For sea-carriage, the tank is made cellular.—R. A.

*Heating, Cooling, and Mixing Liquids or Chemicals, Drying or Superheating and Condensing Steam; Impts. in Apparatus for.* J. Whiteley, Clowes Street, Salford. Eng. Pat. 3627, Feb. 11, 1897.



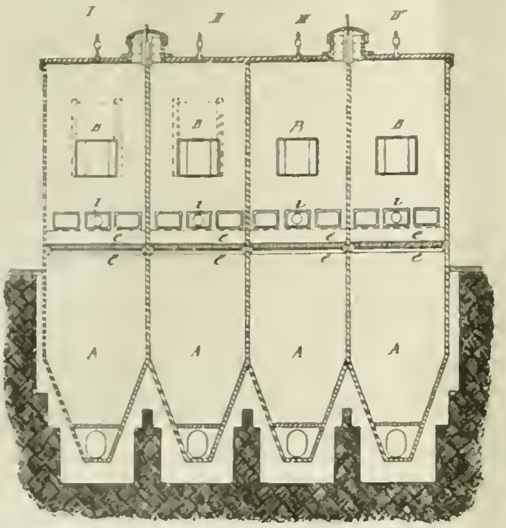
This invention relates to improvements in the rotary type of heaters, for feed water and other liquids, described in Eng. Pat. 19,383 of 1894, the axis of the apparatus being built up of a number of sections *a*, suitably secured together, and each formed with ports adapted to communicate with the circulating pipes *e*, $e^1$.—R. A.

*Separating Heavy from Light Particles by Centrifugal Action, An Improved Process for.* L. Maiche, Paris. Eng. Pat. 4087, Feb. 15, 1897.

In centrifugal separators for pulverised mineral, &c., the material is mixed with viscous liquid, such as starch-paste, syrupy or mucilaginous extracts from seaweeds, &c., instead of with water.—R. A.

*Clearing or Clarifying and Filtering Apparatus [Water, Sewage, &c.], Impts. in.* A. W. T. Hülssner and F. Rohrig, both of Leipzig, Germany. Eng. Pat. 4221, Feb. 16, 1897.

This invention relates to filtering apparatus, which is self-cleaning, either by the use of benzene, petroleum, or other oil, or by the filtered liquid. The filtering apparatus comprises four filtering chambers, I., II., III., IV., arranged in series, as shown, the liquid being admitted under pressure into the lower chambers A, and filtered upwards through the filters *e* into the upper chambers B. The filtered liquid passes outward through the chambers *i*, and pipes into a common delivery channel. After the filtered liquid has been discharged from a chamber B, the oil or other cleansing liquid is passed downward through the filter *e*, and subsequently upwards through the same, a fresh supply of unfiltered liquid being then admitted to the chamber A. The same cycle of operations takes place in all the four sets of chambers, but the stages in the cycle are always different. By suitably manipulating the supply, delivery, and connecting cocks, so as to connect the chambers in different orders, at intervals, a continuous discharge of filtered liquid is obtained. The solid or deposited matter is collected in suitable antechambers, and discharged at intervals.—R. A.



*Chemical Fire Engines, Impts. in.* J. E. Bott, Colwyn Bay, North Wales, and G. H. Morrison, Liverpool. Eng. Pat. 5730, March 4, 1897.

The fire engine has two horizontal cylinders, ranged side by side, charged with sufficient of an alkaline salt (such as sodium bicarbonate) to give a strong solution on filling the cylinders several times over with water. The impulsive force is derived from a charge of liquefied carbonic acid, or a compressed gas or air, contained in a smaller cylinder, set between the lower parts of the main cylinders, and so connected that the gas may be turned into the two alkali-charged cylinders alternately. The main cylinders are charged with water from a hydrant, and are put into connection with the delivery hose alternately, so that a continuous stream of the alkaline solution may be directed on the fire to be extinguished. The various connections and accessories are described and shown in detail.—E. S.

*Drying Wheat, Brewers' Grains, Fish Guano, and other Substances; Impts. in Apparatus for.* J. C. W. Stanley, West Kensington, and A. W. Maxwell, Lee, Kent. Eng. Pat. 8321, March 31, 1897.

The drier consists of a long narrow vessel having a rounded bottom, heated by direct fire or by steam. It is provided with a longitudinal rotating shaft, dipping into the materials to be dried, and provided with a worm divided up into separate vanes. Jets are provided for delivering compressed air into the mass under treatment, while the vapour produced is conveniently withdrawn from the top by means of steam ejectors in the exit pipes. A double drier is also shown, in which the materials circulate backwards and forwards through the double vessel, during treatment, instead of travelling only one length of the vessel, as in the single form.—R. S.

*Cooling and Washing Gases; Apparates for.* F. J. Collin, Dortmund, Germany. Eng. Pat. 10,988, May 3, 1897.

A vertical column is provided at the top with a series of distributing or spraying jets for the washing liquid, and with a side opening for the entrance of the gas to be cooled and washed. The main space of the column has vertical tubes leading from a top plate just below the gas inlet to a bottom plate. Through these tubes passes downwards the mixture of gas and liquid, while around them circulates the cooling liquid, entering below and leaving at the top. The gas collects in a lower space, but before reaching the outlet, has to pass down pipes fitted into a top plate, and dipping into liquid in a chamber beneath. The liquid escapes through and its level is regulated by a siphon. The mode of working the apparatus may be modified.

—R. S.

*Artificial Ice, Impts. in the Manufacture of.* Marquis d'Osmond, Rue Louis le Grand, Paris. Eng. Pat. 30,022, Dec. 18, 1897.

The process of manufacture consists in freezing successive layers of water in a tank, by the passage of currents of cold air through tubes or conduits traversing the mass of water and communicating at the bottom with a cold-air main, the conduits being raised after the congelation of the first layer of water, for effecting the congelation, in turn, of a super-adjacent layer, and so on. The conduits are placed closer together near the walls of the structure, to effect a more rapid congelation thereat, and strengthen the walls by the ice formed. After the formation of each layer of ice, and before moving the conduits to form the next layer, a current of hot air is passed through them, to detach them from the adjacent ice. By this method the ice is first formed in a series of hollow chambers filled with water, this contained water being totally congealed in the lower layers of chambers, whilst the upper layers of chambers are in the earlier stages of formation. At the surface of each layer a number of perforated plugs are employed, to permit the escape of the water expelled from the ice chambers during the congelation of the water remaining therein. In another arrangement, a tank or building is employed, which is traversed by a series of removable air conduits, for the introduction of cold air to freeze the surrounding water, and of warm air to detach them from the block of ice so formed.—R. A.

## II.—FUEL, GAS, AND LIGHT.

*Electric Glow-Lamps; Temperature of the Filament.* P. Janet. Comptes Rend. **126**, [10], 734—736.

By measuring the change of resistance of the filament with varying difference of potential between its terminals, and then, by special arrangements, measuring the resistance at closely-succeeding instants during the cooling of the filament after switching off the current, the authors arrive at the amount of heat lost during cooling. From this, the mass of the filament, and the specific heat of carbon, the temperature can be calculated. Four 65-volt 10-candle lamps gave respectively 1,720°, 1,610°, 1,630°, and 1,620° C. These figures approach that (1,800° C.) of Le Chatelier, whose lamps were probably pushed farther than those of the authors, as indicated by the lower ratio of resistance hot to resistance cold, which he obtained.—J. P. D.

*Acetylene, Inconveniences of.* Josef Vértess. Chem. Zeit. 21, 174.

The author has been able to study the use of acetylene on a large scale at the central station of the town of Veszprim (Hungary). The carbide generally contains uncombined carbon, which is carried in the form of fine dust into the pipes, and with the water of condensation forms obstructions in them. In theory 1,000 grms. of carbide should yield 360 litres of acetylene; the continental works guarantee only 300, and practice shows that 280—290 is the general yield. Besides free carbon, the carbide contains metallic compounds of sulphur, phosphorus, and nitrogen, and consequently the acetylene evolved contains $H_2S$, phosphuretted hydrogen and ammonia, the impurities reaching 3—4 per cent. Acetylene ought to be purified in the same way as coal-gas, and until this is realised it cannot make much progress as an illuminating agent. The burning of unpurified acetylene in closed rooms is dangerous to health, the most dangerous impurity being phosphoretted hydrogen. There is no doubt that some of the so-called spontaneous explosions have been caused by the ignition of this gas at the temperature existing in the generator. The worst inconvenience of acetylene is the "smoking of the flame." The best burners are those with 10—12 small flames, as each flame has a separate atmosphere; even with these, smoking sets in after 200—300 hours burning. The cause is the decomposition of the acetylene by the heat of the burner; the carbon deposits in the narrow opening of the burner and the flame begins to smoke. Sometimes an oily liquid is formed in the burner, probably a hydrocarbon formed by polymerisation of the acetylene.

Much work is required before all the defects specified are overcome; the main conclusion, however, is that it is absurd to attempt to produce acetylene without proper purifying plant.—J. T. W.

*Natural Gas and Oil Gas in the United States.* Engineer, 1898, **85**, 308.

In the United States both natural gas and oil gas are used as substitutes for producer gas in steel-melting furnaces, heating furnaces, and boilers. In the Pittsburgh district natural gas is piped from a distance of 100 miles. It costs 5*d.* per 1,000 cb. ft., and requires no re-generating. It contains 92·6 per cent. by volume of methane, and possesses about seven and a half times the calorific value of producer gas. At present prices there is no advantage in using one over the other, but natural gas tends to become dearer. Where crude oil is used instead of producer gas, it is blown in, and at the same time vaporised by a steam jet through the gas port. The air only is re-generated. About 400 lb. of oil are used per ton of ingots, as compared with about 980 lb. of coal in the producers. The oil has about 25 per cent. more calorific value than Welsh coal. Whether or not it pays to burn it, says Mr. A. P. Head, depends on the local conditions and the price of the oil, which latter, like innumerable other articles of common use, is not regulated in America entirely by the balance of supply and demand, but rather on the measure of success which attends the efforts of the combination which is so fortunate as to control it.

*Chimney Smoke, Colorimetric, Determination of Density of.* P. Fritzsche. Zeits. Anal. Chem. **37**, [2], 92.

*See under* XXIII., *page* 377.

*Prussian Blue in Spent Gas Purifying Material* [*Oxide*], *Determination of.* Lubberger. J. für Gasbeleuchtung, 1898, **41**, [8], 124.

*See under* XXIII., *page* 382.

*Incandescence Mantles of Commerce, Analysis of.* E. Hintz. Zeits. Anal. Chem. **37**, [2], 94.

*See under* XXIII., *page* 378.

*Calcium Carbide, Analysis of.* H. Bamberger. Zeits. f. angew. Chem. 1898, [9], 196.

*See under* XXIII., *page* 382.

## PATENTS.

*Cooling and Washing Gases, Apparatus for.* F. J. Collin, Dortmund, Germany. Eng. Pat. 10,988, May 3, 1897.

*See under* I., *page* 336.

[*Incandescence*], *Lighting and Heating by Means of Gas, Mineral Oil, and the like; Impts. in and relating to.* P. G. de Schodt, Brussels. Eng. Pat. 1252, Jan. 16, 1897.

This system includes (1) the gas-compressing apparatus, (2) the suction and mixing device, (3) the burner, and can be used either with coal-gas or "a draught of air saturated in the cold state with mineral oil." The gas compressor comprises double-acting bellows, which receive their alternate upward and downward strokes from a piston, upon the opposite sides of which, through a four-way cock, the operating fluid (water, liquefied $CO_2$, steam, &c.) successively acts. The cock is reversed at the end of each stroke by the fall of an oscillating weighted arm, connected by a rack and pinion with the piston rod. By this means a pressure of 100 to 150 mm. of water is obtained. The air suction and mixing device on the Bunsen principle is situated at the base of the burner. It consists of the usual conical injector nozzle, which projects into a small chamber which is perforated round the nozzle with air inlets. The upper part of the chamber, which leads to the burner, is constructed with a "parabolic surface" (roughly resembling the interior surface of the "shoulder" of an ordinary bottle), and part of the upward current of gas and air, on encountering the parabolic surface, "is thrown back to the focus of the parabola, the said focus being centrally situated within the gaseous column issuing from the injector." This backward eddy promotes the mixture of the gas and air. Concentrically with the injector and chamber rises the burner tube, which consists of a gently converging truncated cone connected by a non-conducting ring with an inverted truncated cone, which diverges gradually to the burner mouth, the latter being surrounded by an asbestos washer and capped with wire gauze. For street lighting, with gas at its normal pressure, the air supply to the mixing device is converted into a strong upward current by "a tubular regenerator heated by the products of combustion of an independent flame," this arrangement being situated in the base of the lamp post.—H. B.

*Incandescence Bodies for Illuminating Purposes, Impts. in the Manufacture of.* Gustave Leon Albert Count de Hamel de Manin, South Kensington. Eng. Pat. 5817, March 4, 1897.

The fabric is to be impregnated "with an aqueous solution or thin paste of a mixture of chloride of lime or salt of calcium with caustic soda or other alkali," then dried and burnt off as usual. Sixteen prescriptions are given and claimed, such as "hydrated or crude powdered lime, 1 part; precipitated chalk, 1 part; solution of chloride of lime, 4 parts; oxide of magnesia, 1 part; caustic potash to saturate; hydrochloric acid to suit consistency." "The addition of methylated spirit to all the above solutions appears to be beneficial."—H. B.

*Liquid Hydrocarbons, Impts. in or relating to the Burning of.* H. C. Capel, Middlesex, and T. Clarkson, Surrey. Eng. Pat. 5978, March 6, 1897.

The arrangement is of the type comprising an oil reservoir, vaporiser, and air-mixing chamber. The reservoir is closed, is fitted with a pressure-gauge and safety-valve, and is only partly filled with oil, the enclosed air being kept under pressure. To facilitate the preliminary heating of the vaporiser and mixing chamber, there is placed beside each, an oblong metal box packed with sheets of asbestos millboard or equivalent material, the edges of which, projecting slightly above the box, form a wick-burner. At one side of the box is a nozzle so arranged that air may be blown through it (from the compressed air in the reservoir) across the wick, so that when some oil is poured upon the wick, the oil lighted, and the blast turned on, the blow-pipe flame produced, quickly heats the vaporiser or mixing chamber respectively. Underneath the jet from which the vaporised oil is injected into the air-mixing chamber, is arranged a drip receptacle leading to a pump, and there is claimed, "in an oil or similar burner, returning the condensed vapour to the reservoir by the same pump which maintains the air-pressure therein." There is also claimed an openwork grid, consisting of a series of straight and corrugated metal strips arranged side by side, the grid being fixed on the top of the mixing chamber, to prevent the flame from striking back into the chamber. The complete apparatus, and the various combinations of parts, are claimed.—H. B.

*Coke, Impts. in the Manufacture of, and in Apparatus and Appliances connected therewith.* P. Muschamp, Barnsley, and S. Burridge, Sheffield. Eng. Pat. 6404, March 11, 1897.

The coke is discharged from the retorts into a (preferably movable) cooling chamber surrounded by a water-jacket, and the radiant heat converts part of the water into steam, which is then admitted into the interior of the chamber and completes the cooling of the coke to a suitable temperature for discharging. The evolved steam may also be utilised for propelling the chamber, or for working the discharging appliances.—C. S.

*Incandescence Gas Lighting, Impts. in Bunsen Burners specially adapted for.* J. Moeller, Westminster. Eng. Pat. 6775, March 15, 1897.

In the improved burner claimed there are three concentric tubes, the innermost tube, resembling in form the usual laboratory Bunsen burner, having air-inlets at its base; a short tube closed at its top, inverted over and surrounding the first, so that the mixture of gas and air, on ascending the first or innermost tube, is deflected at its top, and caused to descend the annular space. The current then passing outwards through slits in the lower part of the short tube, ascends an annular space within the outermost tube to the opening of this tube at the top, which is furnished with wire gauze, mantle support, &c.—H. B.

*Combustible Substance adapted for use with Fuel, Impts. in Production of.* Count A. D. De Micheroux, Marseilles. Eng. Pat. 7172, March 19, 1897.

To reduce the quantity of sulphur and smoke, whilst increasing the calorific power developed during the combustion, the inventor prepares a mixture of liquid or solid fatty acids (oleic, stearic, cerotic, margaric, &c.), 2—18 per cent.; and hydrocarbons, 92—82 per cent.; heated together and incorporated with finely powdered and dried protoxide of iron, 2—8 per cent.; and dolomite, 2—6 per cent.; as well as 6—12 per cent. of lime or other alkaline substance, the whole being stirred until cool. The product is mixed with the fuel to be improved, which has previously been dried and crushed, the proportion of the first-named taken, ranging from 1 to 20 per cent. according to the requirements revealed by analysis.

The function of the iron oxide is to absorb sulphur, whilst the dolomite and alkali act as a deodorant and preventor of smoke. The resulting fuel may be compressed into briquettes, along with 5—10 per cent. of coal tar, petroleum, or shale, and protected by a coating of sodium or other silicate.—C. S.

*Gas Retorts, Incandescent Furnace for Heating.* A. Forrett, Blackwell, Derbyshire. Eng. Pat. 9315, April 13, 1897.

By extending the front of the furnace 2 ft. beyond the front of the retorts, the furnace can be fed direct with hot cokes, drawn from the latter, and delivered through an aperture in the cover plate, thus saving a large amount of heat otherwise lost. The air supply is taken up the front, and enters the furnace near the top.—C. S.

*Atmospheric Burners for Combustible [Hydrocarbon] Vapours, An Impt. in.* J. S. Bickford, Camborne. Eng. Pat. 9912, April 20, 1897.

From a space in the upper part of a block, which is supplied with oil from any suitable reservoir, there projects a pipe, having a slight slope upwards from the horizontal; it acts as a vaporising chamber, and from its end descends a pipe to lead off the vapours. The latter pipe is bent back at right angles and forms a jet, which is directed into the middle one of three nearly horizontal parallel pipes, each of which at its farther end communicates with a chamber within the lower part of the block. The two side pipes are closed at their free ends, but are perforated along their upper parts. Above these three pipes is supported a curved sheet of wire network. The jet of vapours entering the middle pipes, draws in air; the combustible mixture passes into the block chamber and back along the side tubes, issuing through the perforations, mixing with more air, and finally burns above the network. The burner is claimed. —H. B.

*Waste Gases arising from Retort Settings and other Furnaces, New Method of Utilising.* R. G. Shadbolt, Grantham, and J. W. Broadhead, Elland. Eng. Pat. 10,838, May 1, 1897.

The inventors propose to utilise the waste heat of furnace gases by mixing these with air and passing them through a second producer, whereby the carbon dioxide is reduced to monoxide, which in turn is oxidised by the secondary supply of air. The admission of the waste gas and air supplies may be adjusted according to the requirements of each particular case, and it is claimed that the recuperation may be continued through a series of producers until the gases become inert through the accumulation of nitrogen, sulphur oxides, &c.—C. S.

*Oil-Burners for the Production of Non-Luminous Flames, especially for Heating Incandescent Bodies; Impts. in.* M. Graetz, Berlin. Eng. Pat. 15,317, June 26, 1897.

Improvements in lamps of the type described in patentee's Eng. Pat. 2295 of 1897 (this Journal, 1898, 141). The first claim is, "In an oil-burner for the production of a non-luminous flame, the combination of the two concentric wick-tubes, the inner of which is contracted at or near its upper end, of a spreading disc having a downwardly bent rim and apertures in said rim, a cap surrounding the outer wick-tube, and terminating at about the height of the lower edge of said spreading disc; and another cap surrounding said first cap, and terminating at about the height of the lateral apertures of the said spreading disc." Openings are provided at the bases of the two caps to admit air to the annular channel between the two caps, and to the space between the mantle and the glass chimney.—H. B.

*Fuel, such as Coal, Impts. in or connected with the Combustion of, and Apparatus therefor.* E. Pollacsek, Budapest, Hungary. Eng. Pat. 16,722, July 14, 1897.

To nullify the injurious action of the sulphur compounds produced in the combustion of fuel containing that body, the inventor proposes to introduce dissolved or suspended caustic lime, or other base, in the form of spray, into both the oxidising and reducing parts of the flame, and therefore before the heated gases of the furnace reach the object to be heated. By this means it is claimed that the sulphur is combined and rendered innocuous; that any excess of water in the spray is converted into water-gas, and that the presence of moisture in the upper part of the furnace aids the combustion of the smaller particles of coal in the fuel. The lime liquor is delivered from a reservoir through a pipe and is sprayed by a steam jet, so as to have a temperature of from 70° C. to boiling-point as it issues from the nozzle.—C. S.

*Gas Producers, Impts. in.* B. Talbot, Pencoyd, Pa., and W. B. Hughes, Wissahickon, Pa., U.S.A. Eng. Pat. 27,379, Nov. 22, 1897.

This invention relates to gas producers generating carbon monoxide from coal or other fuel, in a furnace having a determinate air supply.

One of the improvements relates to the simplification of the water-seal of the ash hopper, and another to the disintegration of clinkers by appliances for poking.

The lower portion of the producer is preferably made larger in diameter than the upper part, so that pokers can be introduced through the flange thus formed between the two halves of the apparatus, such pokers being movable in all directions, and worked either by hand or by mechanical appliances. Or, again, the lower portion of the furnace may be made to revolve whilst the upper portion is stationary; and in this case the pokers merely swing to or from the centre, the breaking of the clinker being caused by its impact against the pokers as the furnace revolves.

The ash hopper dips into a water-seal, through which projects a vertical pipe for the supply of air to the fuel; but the upper part of the hopper is exposed to the air in order to cool down the ashes before they reach the water, and in this manner to prevent the formation of an excess of steam, which would lower the efficiency of the furnace. Where the furnace revolves, wing scrapers on the ash box loosen the ashes in the seal pit and facilitate their removal by a conveyor provided for that purpose.

Other features of the invention are: the provision of pipes for the delivery of steam or water in order to soften the clinker and ash on the hearth-plate before removal; and the extension downwards of the magazine of fuel in the upper part of the generator to below the junction of the fixed and movable parts, the result being the storage of a large quantity of unignited fuel above the hot zone, which fuel acts as a soot catcher and preventor of waste.—C. S.

*Gas, Inflammable, and Composition to be used therein; Process and Apparatus for the Manufacture of.* G. W. Lee, F. Elliott, and G. Tatham, Sydney, N.S.W. Eng. Pat. 30,689, Dec. 28, 1897.

In this process air is saturated with moisture and then passed through a chamber containing porous material, such as wood shavings impregnated with equal parts of ether and citronella oil and four parts of water, the resulting gas being mixed by deflecting its course with a number of baffles, and discharged into a gasometer or main. The saturator consists of a horizontal cylinder, rather more than half full of water, in which is rotated a second cylinder, divided by a number of longitudinal vanes, and perforated at the ends with apertures through which air is alternately drawn in and expelled during the rotation, by which means saturation with water vapour is produced.

The gas is highly illuminating, and may also be employed for heating purposes if mixed with air.—C. S.

*Combustible Liquids intended for Lighting or Heating Purposes, Process of Producing Pressure in Vessels or Receptacles containing.* H. H. Lake, London. From La Cie. Continentale d'Incandescence et du Chauffage, Brussels. Eng. Pat. 1517, Jan. 19, 1898.

The air contained in a closed reservoir is subjected to the pressure of a column of liquid (*e.g.*, water) contained in a vertical tube forming the outlet of a wide shallow vessel and reaching nearly to the bottom of the air chamber. This pressure is then transmitted through pipes to the lamps, &c. containing the combustible liquid, and is there applied to the surface of the latter, which is consequently forced up into the burners through suitable conduits.—C. S.

*Electric Furnaces [for Carbides, &c.], Impts. in.* C. S. Bradley, Avon, State of New York, U.S.A. Eng. Pat. 27,888, Nov. 26, 1897.

"For the reduction or formation of refractory materials. The object of the invention is to permit a continuous and uninterrupted operation of the furnace and withdrawal of the product, and to protect said product from the action of the air when at a high temperature. The furnace is especially designed for employment in the manufacture of metallic carbides. It comprises a receptacle for the charge to be operated upon, in which is inserted an electrode, means being provided for continuously moving the receptacle with relation to the electrode so as to bring fresh portions of material under the action of the electric current." "Solid carbide of calcium is a conductor of electricity, and the copper plugs make a good contact with the same." Other details are supplied. Claim 7 is: "An electric furnace comprising a rotary wheel turning on a horizontal axis, removable rim-sections forming a receptacle for a charge over an arc of the wheel, a current-leading electrode connecting with one pole of an electric generator, conducting plugs in the wall of the wheel-rim projecting into the receptacle space, and a commutator for cutting the plugs successively into circuit."—J. C. R.

*Calcium Carbide, Impts. in Furnaces for the Manufacture of, or the like.* T. Keene, Birkenhead. Eng. Pat. 6648, March 13, 1897.

The furnace is provided with an outer casing, the inner chamber consisting of a series of truncated cones placed with their narrow ends downwards, and united by perforated walls, so that the hot gases in rising, heat the descending charge. The cones are placed vertically, or nearly so, over the electrodes off which their sloping sides take part of the weight of the charge, the descent of which is regulated by a central, hollow, perforated shaft, adapted to be rotated and moved vertically.—G. H. R.

*Calcium Carbide, Impts. in the Art of Producing.* J. Leede, Minneapolis, U.S.A. Eng. Pat. 32, Jan. 1, 1898.

Quicklime is stirred into about an equal weight of melted coal-tar pitch, and the resulting mass is heated in an ordinary retort furnace. The hard, coke-like product thus obtained is ground, "and then intensely heated by electricity or otherwise," to obtain crystallised calcium carbide.—E. S.

*Acetylene Gas Lamp.* F. H. Smith, Dunblane, N.B. Eng. Pat. 4790, Feb. 23, 1897.

The generator is below the lamp, and constructed as part of it. It consists of two separable chambers, the upper of which contains water, which drips from a valve, which closes when the pressure of gas exceeds that of the water, on to calcium carbide in the lower chamber.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Manufacture of.* G. Baldwin, West Dulwich, Surrey, formerly of Holloway, London, and C. Crastin, Holloway, London. Eng. Pat. 5445, March 1, 1897.

A closed water tank with gasholder communicates with an overhead cistern, and supplies water to a generator containing carbide in baskets. The gas generated, passes into the upper part of the combined water tank and gasholder, and thence to the burners, but if the production exceed the consumption, it depresses the level of the water in the tank below the feed-pipe to the generator, and thus cuts off the supply of water to the carbide. A float on the water in the tank falls with the level of the water, and closes valves on the water and gas pipes shortly after the supply of water to the generator is cut off, and any after-production of gas must thereafter pass out at the bottom of the generator, and carry with it any water remaining in the latter. Generators in duplicate, waste and relief cocks, an electric signal, and numerous accessories and parts are described in detail.—J. A. B.

*Acetylene, Impts. in or relating to the Manufacture of.* W. W. Strode and G. H. White, both of Osnaburgh Street, Middlesex. Eng. Pat. 6658, March 13, 1897.

The displacement of the bell of a gasholder increases as the bell sinks, owing to increasing thickness of the walls of the bell. The displacement regulates the supply of water to carbide arranged on trays in generating vessels, so that the supply of water increases with the consumption of gas and consequent depression of the bell, and thereupon the production of gas is increased. The gas is purified by passage through water, sawdust, oxide of iron, and powdered charcoal. A regulating tank and feed tank are attached to the tank of the gasholder.—J. A. B.

*Acetylene Gas Generators, Impts. in.* T. and J. Lyons and J. Pearson, all of Blackburn. Eng. Pat. 7043, March 18, 1897.

Two bell gasholders are used in turn. In the crown of each is a hole, through which passes a cylinder containing a perforated carbide container. The cylinder is supported from the flanged cover of the hole in the crown of the bell by means of a spindle, bosses on which screw, with right-handed thread, into one end of the cylinder, and with a left-handed thread into the other. The container is charged with carbide, the cover of the cylinder screwed down, and then, by turning the spindle, the cylinder is liberated and drops on to a stop, whereupon water gains access to the carbide through the hole in the lower end of the cylinder. The device is intended to facilitate opening and re-charging the containers when *in situ*.—J. A. B.

*Acetylene Generators, Impts. in or relating to.* J. G. A. Kitchen, Manchester. Eng. Pat. 7918, March 27, 1897.

Water is poured in up to a certain level in a vessel, which has a flexible base, and a perforated carbide container is placed therein, so that it dips to a small extent into the water. A burner is attached to the top of the vessel. If the consumption of acetylene be less than the production, the pressure of the gas within the vessel distends its flexible base, and the level of the water is lowered below the bottom of the carbide container, generation of gas ceasing until the pressure is reduced. In the event of the apparatus being upset, a shield prevents the water flowing on to the carbide.—J. A. B.

*Acetylene Gas Lamp or Generator.* F. H. Smith, Dunblane, N.B. Eng. Pat. 7929, March 27, 1897.

Water escapes from the upper to the lower of two chambers through a pipe which reaches nearly to the bottom of the lower chamber. On reaching a certain level in the lower chamber, the water flows through a slot in an upright pipe into a chamber containing carbide. The gas generated, passes into the top of the lower water chamber, and, unless removed therefrom through an exit pipe, forces water back into the upper chamber, and interrupts its flow to the carbide chamber.—J. A. B.

*Acetylene Generators, Impts. in and relating to.* J. G. A. Kitchen, Manchester. Eng. Pat. 8270, March 31, 1897.

Carbide is pressed by a spring or weight on to a grating, against the under side of which lies the free end of a wick, which passes through a tube into water in a lower chamber, and conveys water to the carbide. The gas generated, passes through the carbide to an exit at the top of the apparatus. The wick is raised and lowered by a toothed wheel and a spindle extending outside the chamber.

—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Manufacturing.* J. H. Exley, Huddersfield. Eng. Pat. 8551, April 3, 1897.

In order to bring a second generator into operation when one is exhausted, the upper end of the first generator is connected with the lower end of the second, so that water flows from the first into the second, and the generation of gas is uninterrupted.—J. A. B.

*Acetylene Gas Generators, Impts. in.* F. H. Smith, Dunblane, N.B. Eng. Pat. 15,754, July 2, 1897.

Two carbide containers are in the lower of two chambers, the upper of which supplies water to the lower through a pipe reaching nearly to the bottom of the latter. In the lower chamber are two upright pipes having slots at the same level, and a third having a slot at a higher level. These pipes are attached to three ports of a five-way cock, the other two ports leading to the carbide containers. The stem of the cock is bored, so that when one carbide container is in communication with one of the pipes having a slot at the lower level, and is supplied with water thereby, the other container is in communication with the pipe having a slot at a higher level, so that, when the first container is exhausted, the water rises to the upper slot, and passes forthwith to the second container. The gas generated passes to the upper part of the lower chamber, and controls the level of the water therein.—J. A. B.

[*Acetylene*] *Gas Generating Lamps, Impts. in.* F. Rhind, Bridgeport, Connecticut, U.S.A. Eng. Pat. 20,051, Aug. 31, 1897. (Under Internat. Convention.)

Water is supplied to a mass of calcium carbide from an adjacent reservoir through a suitable wick. Various means of adjusting or throttling the wick in order to control the supply of water are described.—J. A. B.

*Acetylene Gas Generators, Impts. in.* J. and J. Mücke, both of Berlin. Eng. Pat. 30,637, Dec. 28, 1897.

Bags or drums of finely woven fabric are partially filled with calcium carbide and placed in the generator. The residue from the carbide is thus easily removed, and cannot by contact with the metal parts of the apparatus injure them. The water used remains clear. It is stated that the gas so produced "will give a light which is considerably more intense and white than hitherto."—J. A. B.

*Acetylene Burners, Improved Device in connection with, for Preventing the Formation of Soot.* E. H. J. Schülke, Berlin. Eng. Pat. 926, Jan. 12, 1898.

A disc or plate provided with an opening or openings corresponding to the outlet or outlets of the burner, is attached to the latter, so that it is a short distance above it. The gas burns at the opening in the disc, and therefore soot does not form on the burner.—J. A. B.

*Acetylene Gas Generators, Impts. in.* E. Bournonville, Jersey City, U.S.A. Eng. Pat. 1013, Jan. 13, 1898.

A closed tank with conical base terminating in a valved waste pipe, has a shoot, by which carbide is fed into it, and generates gas, which escapes through a purifying box in its top to a bell gasholder, which floats in an annular seal surrounding the tank. On a rod attached to the bell is a pawl, which, when the bell descends nearly to its lowest position, engages a ratchet wheel, which, by suitable gearing, revolves through one segment of a segmental wheel, each pocket of which contains carbide. The carbide is thus discharged in turn from each pocket of the wheel into the shoot leading to the generating tank. Carbide coated with oil is preferred; on its decomposition, the oil forms a protecting layer on the surface of the water in the generating tank.—J. A. B.

*Flash Powder for the Taking of Photographs by Artificial Light.* A. Weiss, Strassburg. Eng. Pat. 1126, Jan. 14, 1898.

*See under* XXI., *page* 374.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Petroleum, Export of, from the Caucasus in* 1896. C. H. Export, **19**, [50], 653.

According to figures supplied by the Association of Naphtha Producers at Baku, the total exports of petroleum (crude, refined, and residuum) from Baku and Novorossisk in 1896 amounted to 45,177,452 poods (9,211,665 poods being from the latter centre). The European trade took 14,404,531 poods in tanks and 7,119,568 poods in drums and barrels, whilst the total despatched to the Far East in tanks and cases amounted to 23,323,353 poods. The chief European buyers were: England, 3,590,148 poods; Bulgaria, 643,909 poods; Belgium, 869,432 poods; Germany, 747,282 poods; Italy, 1,199,327 poods; Servia, 190,619 poods; Roumania, 93,713 poods; Turkey, 6,636,172 poods; and in the extreme Orient: Nearer India, 11,133,110 poods; further India, 1,449,864 poods; Sunda Islands, 1,524,293 poods; Philippine Islands, 274,518 poods; China, 6,156,328 poods; Japan, 2,332,552 poods; Siberia, *viâ* Vladivostok and Nikolajewsk, 266,624 poods.

In the year 1897, complaints of lack of orders have been rife, and prices have consequently fallen and freights risen to such an extent as to involve producers and exporters in considerable loss.—C. S.

*Petroleum Waste-Products, Utilisation of.* R. Zaloziecki. Chem. Rev. Fett- u. Harz-Ind. **5**, [2], 27—29.

Two factories in Russia occupy themselves with the recovery of the sludge acid from the refining process, by concentrating the acid—freed from tarry products by previous dilution—to 60° B. in cast-iron pans, and then by distillation to 66° B., the product—which contains 2 per cent. of salts from the Caspian Sea water used in dilution, and 1 per cent. of organic matter—finding employment for various purposes. The author attempted to accelerate the process and obviate the loss of acid, caused by the reducing action of the organic matter, by adding carriers of oxygen; the beneficial results, however, were limited to the waste acid from ozokerit- and paraffin-refining, no improvement being effected in the case of the acid from petroleum refineries.

In the decomposition of the sludge acid, dilution, air, and light are the three chief factors, the first-named causing precipitation, whilst the second and (probably) third lead to sulphonation and oxidation, the precipitate from fresh sludge acid mainly consisting of resinous polymerisation products, whilst that from older acid, or such as has been warmed to 60°—100°, chiefly contains sulphonic acids. In purifying the separated acid, an addition of ½ to 1 per cent. of bleaching powder or potassium chlorate is highly efficacious. By allowing the acid to react at high temperatures on clay, the author obtains light-coloured solutions free from organic matter and utilisable for making aluminium sulphate or alum; and almost the whole of the acid (90 per cent.) may be thus recovered by observing the following precautions:—The use of pure air-dry, pulverised fireclay or other non-marly clays free from iron; diluting the acid with 50 per cent. (vol.) of water; mixing the clay and acid in equal proportions; heating the mixture for six hours at 180°—210° C. Impure lyes can be worked up for alum, and pure alumina can be obtained by decomposing the above solution with clarified waste lye from petroleum works, and treating the product (sodium aluminate) with acids.

To prepare lacquer varnishes and paints from the separated tarry matters, the author boils them with an equal volume of water and neutralises with basic oxides, carbonates, or organic bases, the resulting red-brown mass being then either freed from water and converted into lacquer by dissolving in a cold mixture of very strong alcohol and benzene, oil of turpentine, or tar oil; or it is dissolved direct in an equal volume of 50 per cent. alcohol and benzene, &c. These products are classified with the "mixed resin metallic soap lacquers," and are quick-drying owing to the siccative properties of the petroleum- and sulphonic acid salts. They are unacted on by air and light, though affected by water, and are suitable for use on wood, metal, and leather. An addition of 5—15 per cent. of linseed-oil varnish renders the mixed and resin lacquers more elastic. Fresh sludge acid is most suitable for making these products, since it contains more of the lacquer-forming ingredients than older acid; and the petroleum acids are more efficient as a source of siccative power than the sulphonic acids. The greatest yield (66—75 per cent. of the total acid tar) is obtained from the waste acid from lubricating-oil refineries.—C. S.

*Petroleum Residuum [Masut], Character of.* N. Kromer. Zeits. Anal. Chem. **37**, [3], 176—181.

One hundred samples of masut taken from various depôts and vessels over a large area in Russia were examined, the distillation being performed in Engler flasks, and in accordance with the precautions laid down by Beilstein and Engler, 310° C. (corr.) being the highest temperature employed.

The specific gravity of the distillates (at 15° C.) ranged from 0·8069 to 0·8707, the commencement of distillation from 110° to 250° C., and the burning point (Pensky-Martens) between 47° and 165° C. In quantitative composition, the distillates up to 310° formed, in 11 samples, 12·9—16·8 per cent.; in 26 samples, 17·0—20·9 per cent.; in 34 samples, 21·2—26·7 per cent.; in 17, 27·0—30·2 per cent.; in 7, 32·9—37 per cent.; and in five single instances, 6 per cent, 41·9 per cent., 44·4 per cent., 72·95 per cent., and 82·75 per cent. of the total substance, and consisted principally of hydrocarbons.

As regards burning point, 30 per cent. of the samples fell below the limit of 70° C. prescribed by the (Russian) Internal Communication Department in May 1897 for residuum and crude oil for transport on the Volga.—C. S.

*Paraffin, Commercial; Testing the Setting Points of.* R. Kissling. Chem Zeit. **22**, [22], 209.

*See under* XXIII., *page* 380.

## IV.—COLOURING MATTERS AND DYES.

*Indigo, Some Points of Difference between Natural and Artificial.* J. Rhodes. J. Soc. Dyers and Colourists, 1898, **14**, 59—61.

Comparative trials have been made with "Indigo Pure," the synthetic product of the Badische Anilin und Soda Fabrik, and natural indigo.

Artificial indigo paste, 20 per cent., when allowed to stand for some time, becomes partially solid. Printing trials with "Indigo Pure" and a pure sample of indigotin, showed that, although the artificial product goes on to the cloth much quicker than the natural one, the former loses, in subsequent soaping, 10 per cent. more of its shade than the latter. A colour made up of equal quantities of indigotin from natural and artificial indigo, compared with natural indigotin, showed the same loss in shade on soaping, which proves that artificial is not so fast as natural indigo.

In a third trial, using a colour containing three parts of natural to one part of artificial indigo and comparing it with the natural product, the dyeing is a little more rapid, and there is practically no difference in the loss of shade on soaping.

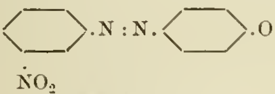
In a final trial made with indigotin, freed from indigo gluten and indirubin, and artificial indigo, the dyeing of the latter is again much more rapid, but the loss in shade on soaping is very much greater.—C. M.

*Azo Dyestuffs obtained by Combining Diazo Compounds with Nitrophenols and Nitronaphthols.* J. Altschul. Chem. Zeit. 1898, **22**, 115—116.

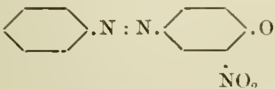
The introduction of a nitro group into the molecule of a diazo compound causes, according to its position, a difference in shade, which, as the following table shows, is only marked in the case of the combination of the *p*-compound with β-naphthol:—

| Diazo-Compound from | Combined with | |
|---|---|---|
| | α-Naphthol. | β-Naphthol. |
| Aniline ............... | Catechu brown | Orange-yellow |
| *o*-Nitraniline ........ | Brown | Yellowish-orange |
| *m*-Nitraniline........ | Reddish-brown | Orange-yellow |
| *p*-Nitraniline ........ | Brown | Turkey-red |

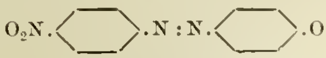
The author has investigated the dyestuffs obtained by combining diazobenzene chloride with o-, *m*-, and *p*-nitrophenol. The first-named compound combines tolerably readily in concentrated solution, whilst *p*-nitrophenol hardly combines at all, and the *m*-compound behaves in an intermediate manner. The dyestuff from diazobenzene and o-nitrophenol crystallises from alcohol in yellow prisms, melting at 127° C., and dyes wool a faint straw colour from an acid bath. Comparing the dyestuffs from *m*- and *p*-nitraniline and phenol with that from *o*-nitrophenol, the former is very similar, whilst that from *p*-nitraniline differs greatly from the other two, and is a strong orange. As their constitutional formulæ show, the dyestuffs of weak tinctorial power have a somewhat analogous constitution in possessing a nitro group meta to the azo group, whilst the strong dyestuff has it in the para position.



*m*-Nitrobenzene-azo-phenol.



Benzene-azo-o-nitrophenol.



*p*-Nitrobenzene-azo-phenol.

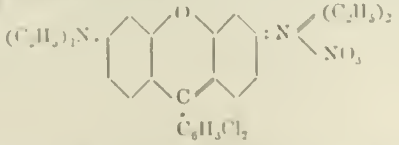
In the case of the nitronaphthols investigated, it was found that combination took place more readily than with the nitrophenols, the following table giving the shades obtained with the various amines.

| Diazo Solution from | Combined with | | | | |
|---|---|---|---|---|---|
| | α-Naphthol. | β-Naphthol. | 1.3′-Nitronaphthol. | 1.2′-Nitronaphthol. | 1.4-Nitronaphthol. |
| Aniline | Catechu-brown | Orange-yellow | Orange | Orange-yellow | Brick-red |
| *p*-Toluidine | Brown | Orange | Orange | Orange | Terra-cotta |
| *m*-Nitraniline | Brown | Orange yellow | Orange-yellow R | Orange-yellow Y | Brownish-red |
| *p*-Nitraniline | Brown | Turkey-red | Orange | Yellow-orange | Reddish-brown |
| β-Naphthylamine | Blackish-brown | Bluish-red | Brownish-red | Scarlet | Brownish-red |
| Benzidine | Puce | Brownish-violet | .. .. | .. .. | Brownish-violet |
| Primuline | .. .. | Turkey red | Brownish-red | Yellowish-red | .. .. |

The dyestuff from sulphanilic acid and 1.2′-nitronaphthol gives an orange very similar both in strength and shade to that from sulphanilic acid and β-naphthol, the only difference being that the former is slightly less soluble. It is noteworthy that, just as in the case of *p*-nitraniline, the introduction of a nitro group para to the hydroxyl should with diazotised aniline cause an alteration of shade to brick-red compared to the brown with α- or the yellow obtained with β-naphthol.—T. A. L.

*2.5-Dichlorobenzaldehyde.* R. Gnehm and R. Schüle.
Annalen, **299**, 347—367.

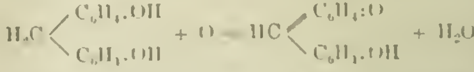
In continuation of a previous paper (Annalen, **296**, 62), the authors describe certain derivatives obtained by the action of 2.5-dichlorobenzaldehyde on ammonia, α-naphthylamine, and β-naphthol with the formation of benzylidene derivatives. Carbon bisulphide at the ordinary temperature yields a polymeric 2.5 chlorothiobenzaldehyde. By reacting with potassium cyanide on the bisulphite compound of the aldehyde, it is converted into the nitrile of 2.5-dichloromandelic acid. This on saponification yields the acid itself. When the aldehyde is condensed with aniline in presence of hydrochloric acid on the water-bath, it forms di-*p*-diamido-2.5 dichlorotriphenylmethane, but it was not found possible to convert either this substance or its acetyl derivative into a 2.5-dichloro-magenta. By condensing the aldehyde with benzene and toluene, 2.5-dichlorotriphenylmethane and 2.5-dichlorophenylditolylmethane were obtained respectively. Phenol and *o*-cresol, when condensed with the aldehyde in acetic acid solution with concentrated sulphuric acid, gave condensation products, the alkaline solutions of which oxidise in the air to dirty red dyestuffs. Resorcinol also combines with dichlorobenzaldehyde, and the product dissolved in caustic soda-lye turns bluish-red in the air with a distinct yellow fluorescence. When diethyl *m*-amidophenol is condensed with the aldehyde in acetic acid solution by means of zinc chloride, a product is obtained which, on further heating for half an hour with sulphuric acid, again loses water, forming the leuco compound of a dyestuff, the nitrate of which has the following constitution:—



and is known as Dichloro-rosamine. The nitrate dissolves sparingly in water with a bluish-red colour and yellow fluorescence. It is easily soluble in concentrated sulphuric acid and in hydrochloric acid, with a light orange-red colour, whilst alkalis precipitate the reddish-violet carbinol from solutions of the rosamine. The dyestuffs New Fast Green 2 B and 3 B and Glacier Blue obtained from 2.5-dichlorobenzaldehyde are sparingly soluble in water, and attempts were made to obtain more soluble sulphonic acid derivatives, but no satisfactory results were obtained in the formation of a chlorobenzaldehyde sulphonic acid either by sulphonating chlorobenzaldehyde or by chlorinating benzaldehyde sulphonic acid. On the other hand, it was found possible to sulphonate with sulphuric monohydrate at 100° C. the leuco compound of tetramethyldiamido-2.5-dichlorotriphenylmethane. The sulphonic acid, on oxidation with lead peroxide, gives a greenish-blue dyestuff sparingly soluble in organic solvents, easily soluble in water with a blue colour very similar to that of New Fast Green 3 B. Dilute caustic soda is at first without action, but decolorises the solution after some time. At the conclusion of the paper, the authors describe the preparation of benzaldehyde o-sulphonic acid and dyestuffs therefrom, the substance of which has already been abstracted (Eng. Pat. 5068 of 1896; this Journal, 1897, 137). The dyestuff from ethylbenzylaniline sulphonic acid is known commercially as "Erioglaucine" (Geigy).—T. A. L.

*Diphenylmethane Dyestuffs Derived from the Condensation Products of Aldehydes with Phenols and Phenol-Carboxylic Acid.* L. Kahl. Ber. **31**, 143—151.

According to Möhlau and Koch (Ber. **27**, 2887; this Journal, 1895, 475), oxyfluorones are formed from those condensation products of aldehydes with phenols that contain at least four hydroxyl groups in the molecule, two being in the ortho and two in the para position to the aldehyde radicle. If the two ortho hydroxyls be absent, the oxidation of the condensation product yields a kind of aurin. Thus the simplest aurin — formaurin — would be obtained by oxidising *p*-dioxydiphenylmethane—



$$H_2C\begin{cases}C_6H_4.OH\\C_6H_4.OH\end{cases} + O = HC\begin{cases}C_6H_4:O\\C_6H_4.OH\end{cases} + H_2O,$$

although this reaction has not actually been effected. In carrying out this research, it was found that formaldehyde always combines with phenol in the proportion of one molecule of the former to two of the latter. The same holds good with regard to acetaldehyde and benzaldehyde so far as monovalent phenols are concerned, but with polyvalent phenols, 2 mols. of the aldehyde condense with two of the phenol. Attempts to convert methylene dipyrogallol into an oxyfluorone were unsuccessful, from which it follows that the compound has the following formula:—

$$[5.4.3.1](OH)_3C_6H_2.CH_2.C_6H_2(OH)_3[1.3.4.5].$$

By dissolving this substance in 10 times its weight of cold concentrated sulphuric acid, allowing it to stand 24 hours in a closed vessel, and then to absorb moisture in an open dish, the solution deposits opaque black crystalline granules of formopyrogallaurin. This compound dissolves tolerably easily in hot water, and dyes chromed wool violet. The action of acetaldehyde on pyrogallol yields, according to the conditions, two condensation products, but no dyestuff could be obtained from either. Formaldehyde condenses with 1.3-dihydroxynaphthalene (Ber. **29**, 1619; this Journal, 1896, 534) to form methylenedinaphthoresorcinol, which crystallises in plates and melts at 164° C. When dissolved in the cold in concentrated sulphuric acid, and the solution afterwards allowed to absorb moisture, crystals of formaldehydroxynaphthofluorone are deposited, which are sparingly soluble in water and alcohol, but dissolve in alkaline carbonates and caustic alkalis with a yellowish-red colour and a strong yellowish-green fluorescence. Although it was not found possible to obtain formaurin by oxidising *p*-dihydroxydiphenylmethane, the dicarboxylic acid of this substance is obtained as follows:—Methylene disalicylic acid is prepared by condensing formaldehyde with 2 mols. of salicylic acid; this is then dissolved in sulphuric acid in the cold and treated with the amount of nitrosyl sulphuric acid corresponding to 1 mol. of $N_2O_3$. After standing some time, the melt is poured into ice-water and the dyestuff is purified by conversion into the sulphite compound, from which it is liberated by warming with dilute acids. It forms a yellowish-red powder, easily soluble in alcohol, ether, acetone, and glacial acetic acid, and also in alkaline carbonates with a yellowish-red, and in caustic alkalis with a dark red colour. In a similar manner, *o*-cresotic acid condenses to form methylene dicresotic acid, which, on oxidation in sulphuric acid solution with $N_2O_3$, also yields a dyestuff. Acetaldehyde condenses with resorcylic acid to form a compound, which, however, could not be obtained crystalline, and decomposed on heating without melting. The reaction between acetaldehyde and gallic acid takes place much less easily than with formaldehyde. The product obtained forms colourless prismatic crystals which decompose on heating, without melting. It dissolves in alkalis and alkaline carbonates, and these solutions turn yellowish-brown in the air. Sulphuric acid dissolves it with a yellow colour, which is turned violet on adding nitrosylsulphuric acid. Benzaldehyde condenses with gallic acid to a somewhat similar compound. The solution in concentrated sulphuric acid is brown, and is turned dirty reddish-violet by nitrosylsulphuric acid. The substance dissolves sparingly in alkaline carbonates, more easily in caustic alkalis, and in ammonia with a fugitive reddish-brown colour.—T. A. L.

### *m-Phenylenediamine, Disazo Compounds from.* K. Eiermann. Ber. **31**, 188—191.

As already shown (Ber. **30**, 2899; this Journal, 1898, 40), the principal constituent of Bismarck Brown is *m*-phenylene-disazo-*m*-phenylene diamine, but since the base might have had one of the two following constitutions—

$$H_2N.C_6H_4.N_2.C_6H_3(NH_2)C_6H_3(NH_2)_2$$

or

$$H_2N.C_6H_4.N_2.C_6H_2(NH_2)_2.N_2.C_6H_4.NH_2$$

the author has prepared the first by combining diazotised *m*-amido-acetanilide with *m*-amido-acetanilide, re-diazotising, combining with *m*-phenylene diamine, and finally hydrolysing the product; whilst the second is obtained by combining two molecular proportions of diazotised *m*-amido-acetanilide with one of *m*-phenylene diamine and hydrolysing as before. The former compound, after dissolving in benzene, separates in shining red crystals containing benzene, and melting at about 134° C.; whilst the latter also forms reddish-yellow crystals containing benzene, and melting at 118° C. Neither substance, when boiled with benzene and phenol, forms a compound with the latter, thus differing from the disazo compound obtained by Täuber and Walder, and affording an indirect proof of the constitution of this latter.—T. A. L.

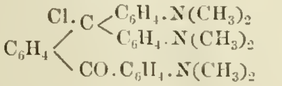
### *Phthalic Green, Preparation and Constitution of.* A. Haller and A. Guyot. Rev. Gén. 1898, **2**, 1—4.

The name Phthalic Green has been given by O. Fischer (Ann. **206**, 112) to a dyestuff obtained, it is stated, from phthalyl chloride and dimethylaniline.

The authors, after having vainly tried to prepare the compound in question, using pure phthalyl chloride, found that it was readily produced by the action of tetrachlorophthalic anhydride (which is present in commercial phthalyl chloride) on dimethylaniline in carbon bisulphide solution in the presence of aluminium chloride. The reaction, which is very lively at first, is complete after a few minutes, the product then separating into two layers, the lower of which is a viscid, tarry mass. On boiling the latter with water, filtering, and cooling, the dyestuff is obtained in small golden needles or plates, of the composition (dried at 100° C.) $C_{32}H_{34}N_3OCl + H_2O$, the yield amounting to 60—95 per cent. It forms a platinichloride—

$$(C_{32}H_{34}N_3OCl)_2 3PtCl_6H_2$$

crystallising in bright red plates. Rosenstiehl has investigated the dyeing properties of the compound and finds that it bears a close resemblance to Malachite Green, from which it principally differs in giving a bluer shade. This is attributable to the fact that it is, like Patent Blue, an ortho-substituted derivative of Malachite Green. Rosenstiehl has proposed for it the formula—

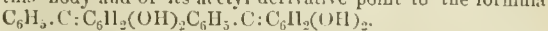


$$C_6H_4 \left\langle \begin{matrix} Cl.C \left\langle \begin{matrix} C_6H_4.N(CH_3)_2 \\ C_6H_4.N(CH_3)_2 \end{matrix} \right. \\ CO.C_6H_4.N(CH_3)_2 \end{matrix} \right.$$

—E. B.

### *Fluorescence.* B. Pawlewski. Ber. **31**, [3], 310. (See also this Journal, 1896, 193.)

According to R. Meyer, the property of fluorescence is conditioned by the presence of—(1) A pyrone ring situated between more complex nuclei. (2) The phthalic acid residue,—anthracene derivatives and bodies containing nitrogen or sulphur being excepted. A number of fluorescing substances are, however, known, of which the constitutions do not harmonise with this theory; to this number must now be added a Resorcinolbenzeïn—obtained by the the action of benzylic chloride on resorcinol. Analyses of this body and of its acetyl derivative point to the formula

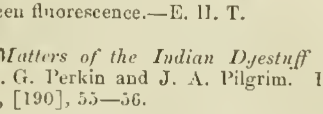


$$C_6H_5.C:C_6H_2(OH)_2C_6H_5.C:C_6H_2(OH)_2.$$

Its solutions in various solvents show a very strong, beautiful green fluorescence.—E. H. T.

### *Colouring Matters of the Indian Dyestuff Delphinium Zalil.* A. G. Perkin and J. A. Pilgrim. Proc. Chem. Soc. 1898, [190], 55—56.

"Asbarg" consists of the dried flowers and flowering stems of the *Delphinium zalil*, found in great quantity in Afghanistan, and which is much used in India for the production of a yellow colour on alum-mordanted fabrics. The flowering stems are nearly devoid of dyeing property. For the supply of material we are indebted to the authorities of the Imperial Institute. Three colouring matters exist in this plant in the form of glucosides. The sparingly soluble colouring matter $C_{16}H_{12}O_7$ forms glistening yellow needles, soluble in alkalis with a yellow colour. Fused with alkali it yielded phloroglucinol and protocatechuic acid, and by means of hydriodic acid it yielded quercetin with the evolution of 1 mol. of methylic iodide. By methylation it was converted into quercetintetramethyl ether, and by acetylation into a tetracetyl derivative of the formula $C_{16}H_8O_7(C_2H_3O)_4$, colourless needles, m.p. 195°—196°. It was evidently *iso-rhamnetin*, a quercetinmonomethyl ether, recently isolated from the yellow wallflower, *Cheiranthus Cheiri* (Trans. Chem. Soc. 1896, **69**, 1650). As by oxidation in alkaline solution this yields vanillic acid, it has most probably the constitution represented by this formula :—



With alumina as mordant it dyes a purer yellow than quercetin. The chief constituent of the more soluble portion was recognised as identical with *quercetin*, the colouring matter of quercitron bark. The residual colouring matter, present only in small quantity, was not obtained in a pure condition. It resembled quercetin in percentage composition and in its decomposition products, but differed from it in not reacting with alcoholic potassium acetate, and in the melting point of its acetyl derivative. "Asbarg" resembles quercitron bark in dyeing property, but has only 35 per cent. of the tinctorial power of this dyestuff. It contains, when freed from the flowering stalks, 3·47 per cent. of colouring matter (not as glucoside).

## PATENTS.

### *Colouring Matters, Impts. in the Manufacture of.* H. R. Vidal, Paris, France. Eng. Pat. 5689, March 3, 1897.

The dyestuffs are obtained by heating phenylhydrazine or its homologues, or naphthylhydrazine, with a mono-, di-, or tricarboxytriphenylhydroxycarbinol, triphenylamido hydroxycarbinol, or triphenylamido carbinol, the carbox groups being ortho or meta relative to the amido or hydroxyl groups. For example, 5 kilos. of phenylhydrazine are heated to 100 C. for 12 hours with 5·5 kilos. of *o*- or *m*-dicarboxytrihydroxyphenylcarbinol in an enamelled vessel provided with an agitator. When the mass has become quiescent and of a red-blue shade, the operation is finished, and the melt is extracted with dilute hydrochloric acid, precipitated by an alkali, filter-pressed, and dried. The resulting products possess "great vividness" and dye mordanted cotton and animal fibres directly.—T. A. L.

*Colouring Matters* [*Black, Blue*], *Impts. in the Manufacture of.* H. R. Vidal, Paris, France. Eng. Pat. 5690, March 3, 1897.

By heating together *p*-phenylene diamine and hydroquinone in presence of sulphur, different products result from those obtained by the action of sulphur on *p*-phenylene diamine or of sulphur and ammonia on hydroquinone. One of the dyes produced, is soluble in acids without reduction, and gives black shades on animal fibres whilst the other dissolves in alkalis and dyes vegetable fibres, a dull blue. A mixture of 5·5 kilos. of *p*-phenylene diamine, 5·5 kilos. of hydroquinone, and 3·2 kilos. of sulphur is heated in a cast-iron vessel provided with an agitator to 170 C. The melt is then treated with a solution of sodium carbonate or with an acid, the filtered solution being precipitated with an acid or an alkali. The black dyestuff so obtained dyes animal fibres from an acid bath. The insoluble product gives dull blue shades on vegetable fibres when dyed in presence of caustic soda or sodium sulphide.—T. A. L.

*p-Amidophenol and the like, Impts. in the Preparation of.* H. R. Vidal, Paris, France. Eng. Pat. 5697, March 3, 1897.

The process claimed consists in heating hydroxyazobenzene or paranitrophenol with sodium sulphide in the presence of caustic soda at 180° C., water being added from time to time until the yellow colour disappears, when the mass is dried, dissolved in water, and the *p*-amidophenol precipitated by acid. By the same method, *o*-nitrophenol and 1.2.4- and 1.2.6-dinitrophenols may also be reduced.—A. C. W.

*Colouring Matters* [*Black*], *An Improved Manufacture of.* H. R. Vidal, Paris, France. Eng. Pat. 6913, March 16, 1897. (Date under Internat. Convention, March 11, 1897.)

The dyestuffs described in Eng. Pat. 6694 of 1897 (this Journal, 1898, 239), when heated with sulphur and caustic soda, are converted into products which dye unmordanted cotton deep shades of black. The following example is a typical one. A mixture of 28 kilos. of the dyestuff from *p*-sulphanilic acid and *p*-amidophenol, together with 25 kilos. of a 33 per cent. caustic soda solution, is heated in an iron vessel provided with an agitator until dissolved, when 6 kilos. of sulphur are added, and the temperature is maintained at 170 C. until the mass becomes too thick to stir. The resulting product dissolves to a blue-black in alkali, and gives deep black shades on unmordanted cotton. In those cases in which a dyestuff derived from a diamine is employed, caustic soda must not be used, since the dyestuff is insoluble therein.—T. A. L.

*Azo Dyestuffs or Colouring Matters* [*Blue, Green, Brown, Black*] *suitable for Dyeing Cotton; Impts. in the Manufacture and Production of.* G. W. Johnson, London. From Kalle and Co., Biebrich-on-Rhine, Germany. Eng. Pat. 8235, March 30, 1897.

The dyestuffs obtained according to this invention are derived from at least one molecular proportion of a naphthalene derivative (amidonaphthol sulphonic acid, dihydroxynaphthalene sulphonic acid, or *m*-naphthylene diamine sulphonic acid) combined with two molecular proportions of a diazo compound. An example illustrating one of the methods employed is as follows:—The diazo solution from 9·3 kilos. of aniline, 38 kilos. of strong hydrochloric acid, 500 litres of water, 500 kilos. of ice, and 6·9 kilos. of sodium nitrite, is added to 1,000 litres of water containing, in the cold, 23·8 kilos. of 1.3.3′-naphthylene diamine sulphonic acid and 25 kilos. of soda ash. When the formation of the monazo colour is complete, there is added to the liquid the intermediate compound from 18·4 kilos. of benzidine and 13·8 kilos. of salicylic acid. After stirring for 10 hours, the whole is heated up, and the dyestuff is salted out, filter-pressed, and dried. Other dyestuffs are obtained in a similar manner by using the compound from tetrazodiphenyl or its homologues, and equimolecular proportions of 1.4-naphthol sulphonic acid, 1.4-naphthylamine sulphonic acid, or 2·1′·3′-amido naphthol sulphonic acid G. The shades from this particular series vary from bluish-red to bluish-violet.—T. A. L.

*Alizarine Lumps, Impts. in the Manufacture of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 21,398, Sept. 17, 1897. (2nd Edition.)

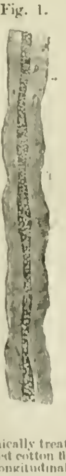
The patentees propose to mix alizarin paste with silica in the form of tripoli or kieselguhr, fuller's earth, pipeclay, or the like, in order to obtain alizarin lumps of any desired percentage. This substance has the advantage, amongst others, over those hitherto proposed, that it neither dissolves nor swells up in water. It is, moreover, cheaper than starch (Eng. Pat. 5444 of 1894; this Journal, 1895, 265), and the percentage of alizarin can be reduced as low as 10 per cent. About 1,000 kilos. of 20 per cent. alizarin paste are stirred up with 4,000 to 6,000 litres of water and mixed with about 200 kilos. of one of the above-mentioned insoluble mineral substances. After filter-pressing, the press-cakes are dried at about 55 C.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Mercerised Cotton, Structure and Properties of Mechanically Treated.* E. Hanausek and R. Zaloziecki. Dingler's Polyt. J. 1898, **307**, 180—181.

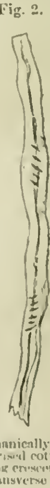
In continuation of previous work on this subject (this Journal, 1898, 42), the authors have investigated the structure of cotton fibres which, after mercerising, have been subjected to the mechanical treatment devised and carried out by F. Mommer and Co., of Barmen, consisting of pressure with plates or rollers engraved with parallel lines. The object of this treatment is to increase the silky lustre of the fabric. After pressure, the surface of the fabric is seen to be marked with alternate ridges and furrows. The furrows, *i.e.*, the pressed portions, were marked with a

Fig. 1.



Mechanically treated mercerised cotton fibre, showing longitudinal and transverse markings.

Fig. 2.

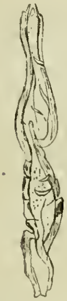


Mechanically treated mercerised cotton fibre, showing crescent-shaped transverse slits.

coloured pencil in order that individual fibres taken from these portions might be recognised under the microscope.

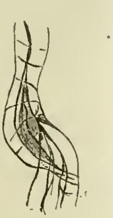
Fibres which have been mercerised, but not mechanically pressed, when treated with ammoniacal copper oxide, exhibit longitudinal striations in the cell wall and a granular inner space (see Figs. 4 and 5, this Journal, 1898, 43). Fibres subjected to the after-treatment described are marked longitudinally and also transversely at intervals (Fig. 1). On careful examination of the coloured pressed fibres, other and more characteristic markings were observed, which differ considerably from the markings due to the spinning process. In the latter case the defects in the fibre are either longitudinal slits or faults extending across the whole breadth of the fibre, but in the pressed mercerised fibre, a number of small crescent-shaped slits or holes are seen, which do not extend right across the fibre (Fig. 2). If the treatment of the fibre has been too severe, its edge may also appear torn, and the structure is practically destroyed (Fig. 3).

Fig. 3.



Mechanically treated mercerised cotton fibre, showing crescent-shaped slits and torn edges. Fibre destroyed.

Fig. 4.



Non-mercerised cotton fibre from outer part of yarn during the spinning process.

If non-mercerised fibres be subjected to the same mechanical treatment, they become altered in much the same manner as by certain operations of the spinning process, the fibres being broadened, broken, and covered with twisted markings (Fig. 4). Hence the effect of the mercerising process is evidently to increase the resistance of the fibre to mechanical strains.—R. B. B.

## PATENTS.

*Carbonising of Rags, New or Improved Apparatus for the.* J. G. Lorrain, London. From B. H. Scheibler, Monjai, near Aachen. Eng. Pat. 25,703, Nov. 14, 1896.

In the manufacture of artificial wool (shoddy), it is only the wool contained in the rags that is of any use. The cotton is usually destroyed by hydrochloric acid vapour. This, if allowed to act on the rags when they are perfectly dry, does not alter their colour, and makes them much more valuable for their required purpose.

In this invention, the rags are placed in a perforated polygonal drum, fitted radially with arms, which do not reach quite to the centre. A hollow shaft, which, in an adjacent chamber, is heated by means of furnace gases, enters at one end. Arrangements are made by which compressed hot air is allowed to enter the chamber containing the drum with the rags, either by way of the hollow shaft and through the rags, or from the outside into the space surrounding the drum.

In this manner the rags are perfectly dried. After this drying, hydrochloric acid is allowed to drop slowly into a funnel, from which it passes through the heated part of the hollow shaft into the drum and carbonises the cotton in the rags.—C. M.

*Waterproofing Threads, Impts. in and Apparatus for.* F. W. Ohner, London. Eng. Pat. 633, Jan. 9, 1897.

In this invention, threads of cotton, hemp, wool, silk, or other fibre are passed, preferably over rollers, through a solution of celluloid, either once or many times.

After leaving the rollers, the thread passes between scrapers or brushes, preferably of felt.

The solution of celluloid is surrounded by a hot-water jacket, and, if desired, the celluloid solution may be coloured with suitable colouring matters.—C. M.

*Vegetable Fibres, Fabrics, and the like; Impts. in the Method of and Means for Mercerising.* E. K. Dutton and Co., Manchester. From E. Crépy, Lille, France. Eng. Pat. 9056, April 9, 1897.

Two rollers, placed horizontally, and with their axes parallel, are fixed in a tank. One is driven by suitable machinery, and the other is fitted with a screw arrangement, whereby the distance between the rollers can be adjusted.

After a preliminary boiling, hanks of cotton-yarn are stretched over the rollers and subjected to a high tension. Whilst rotating slowly over the rollers, the mercerising fluid is allowed to drop slowly on to the yarn from a small reservoir above the tank. When the latter has been subjected for a sufficient length of time to the action of the mercerising agent, it becomes "dead"; that is, it will contract no more. It is then either removed from the rollers and washed, or, if a second reservoir, containing water, be fixed above the tank, it can be washed whilst still on the rollers.—C. M.

*Vegetable Tissues, Impts. in Mercerising.* O. Imray, London. From Meister, Lucius, and Brüning, Hoechst, Germany. Eng. Pat. 11,313, May 6, 1897.

Cotton fibre, on treatment with alkaline lyes, dilates, becomes plastic like clay, and, if left, finally shrinks. This causes a decrease in width of about 30 per cent., and in length of about 16 to 18 per cent.

If the dilated fibre be passed through cylinders, under high pressure, without any loss of time, there is only a shrinkage of about 1 to 2 per cent. in area. This slight contraction can be avoided by diluting the mercerising agent with colloidal substances such as British gum, soluble water-glass, or sodium aluminate. If water-glass be used, it is mixed in the proportion of about 10 to 20 per cent. of the weight of the lye. The fibre is quickly passed through this mixture, then through cylinders covered with cloth under high pressure, and finally rolled up and washed.

It is necessary to roll up the fibre to exclude air, which otherwise, in the presence of alkali, changes the fibre to an oxycellulose.—C. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Suède or Glove Leather, The Dyeing of.* Der Gerber, **24**, [562], [563], [564], and [565], 30, 43, 55, 67.

The difficulties met with in the dyeing to shade of suède leather are perhaps greater than in any other class of leather goods, because (1) the number of shades are enormous; (2) the fashion is ever constantly changing; (3) reliable recipes are impossible; (4) to produce the required shades, only the three primary colours, red, yellow, and blue, can be used; (5) the material (suède leather) varies in itself to a very great extent.

No reliable recipes can be given, because the minutest detail in manipulation completely alters the shade. No two batches of skins will give identical results, as difference in breed, difference in texture or age of the skin, whether they consist of lamb or kid skins, whether they are dried or salted, also the method of extraction of the dye wood, all have an appreciable effect upon the results obtained. The chief colours which are produced on suède leather are as follows:—

*Straw Yellow.*—Produced with weld liquor or fustic. When brighter colours are desired, small quantities of picric acid may be added to the bath

*Flesh Colour, or Chamois.*—Weld liquor, with small additions of Brazil wood, a small trace of logwood to prevent the production of too brown a shade.

*Lilac or Violet.*—A mixture of two-thirds logwood and one-third Brazil wood.

*Rose Colour and Pink.*—Brazil wood, with small additions of fuchsin. Cochineal would be most suitable for these shades, but it has been found impracticable for use on alum leathers.

*Olive.*—Two-thirds fustic, one-third logwood, to be varied at will.

*Sea- or Water-Green.*—Fustic and logwood.—J. G. P.

### *Colouring Matters. Note on the Influence of Dissociation on the Dyeing Power of.* E. Knecht. J. Soc. Dyers and Colourists, 1898, **14**, 59—61.

In a communication to the Journal of the Society of Dyers and Colourists, 1893, 158, it was pointed out that when a drop of magenta solution is allowed to be absorbed by filter paper, the colourless halo which forms round the central coloured disc, contains about half the hydrochloric acid in the magenta. These experiments have been continued with other colouring matters, and it is found that the amount of dissociation—*i.e.*, the size of the colourless halo—increases, up to a certain point, with increasing dilution, while the size of the disc decreases.

The phenomenon is most apparent with basic colours, whilst less so with acid, and the direct cotton colours.

Coloured substances, such as caramel, and also alcoholic solutions of basic dyes, show no halo, and have no dyeing properties; generally speaking, those dyestuffs which show on filter paper the greatest dissociation, are most readily absorbed by the fibre.

It is known that excess of mineral acid in solutions of basic dyes weakens their dyeing properties, and it is shown that the halo—*i.e.*, the dissociation—also gradually decreases with increasing excess of acid.

Experiments have been made showing the relation between strength of dye solution and capillarity; this relation agrees fairly well with that between the size of the halo and the disc.—C. M.

### *Alkaline Mordanting Compounds.* C. Gros-Renaud. Rev. Gén. 1898, **2**, [13], 12—21.

*Sodium Stannate.*—Hausmann, of Logelbach, was the first to prepare an alkaline compound of tin for mordanting purposes; this was the potassium salt. Later, F. Steiner, of Accrington, replaced the potash by soda.

A method of obtaining this salt, which is little known, is due to Ed. Haeffely, of Mulhouse:—40 kilos. of litharge are digested in a solution of 22·5 kilos. of caustic soda containing 22 per cent. of dry caustic soda. When the whole is dissolved, 8 kilos. of granulated tin are added. The lead separates in a spongy condition, and is oxidised for use again.

A product of constant composition may be prepared, without any special apparatus, as follows.—9 kilos. of stannic chloride are dissolved in a stoneware vessel in 25 litres of hot water, and are treated with 15 litres of a solution of caustic soda at 36° B. A clear solution, having a density of 28° B., is obtained.

For use in preparing tissues for printing, the solution at 28° B. is diluted to 6°—18° B. The tissues, impregnated with this, are allowed to lie for a few minutes, and are then passed through a bath of sulphuric acid at 1°—6° B.

*Ammonium Stannate.*—To prepare this, 1 vol. of stannic chloride solution at 55° B., 1 vol. of water, 2 vols. of glycerin at 28° B., and 3 vols. of ammonia at 21° B., are mixed together. Stannic hydrate at first separates, but redissolves in the course of 10—15 minutes. Cotton is mordanted by simply impregnating it with the solution and exposing it to the air, or drying.

*Sodium Aluminate.*—This also was introduced into dyeing by Hausmann, in 1792. To prepare it, alumina hydrate (25 kilos. containing 25 per cent. of alumina) is dissolved in caustic soda (8 litres at 36° B.), the mixture being heated at 30°—40° to effect solution. If deemed desirable, hydrochloric acid may be added to the solution until precipitation commences. For use, it is diluted to 6°—8° B., the precise degree depending mainly upon the pressure applied to the rollers through which the impregnated tissue passes in the padding machine. The drying is accomplished by means of hot air, or in any other convenient manner, but care must be taken that the temperature to which the tissue is exposed is not excessive, as otherwise it is liable to become yellow and "tender" when over-dried. For printing purposes, the sodium aluminate prepared from precipitated alumina is to be preferred. The fixing of the mordant is effected in a bath of 1,000—1,200 litres of water in which 80 kilos. of ammonium chloride are dissolved. To maintain the bath, 25 grms. of this salt are added for each piece of cloth which passes through, and after the passage of 10—12 pieces, 5—6 litres of hydrochloric acid at 21° B. are added, the bath being emptied after 150 pieces have been treated.

*Sodium Chromite.*—Less difficulty is experienced in mordanting cotton with alkaline compounds of chromic oxide than with the ordinary chromic salts.

Gonfreville, in his work "De la Teinture des Laines," refers to the application of alkaline solutions of chrome in mordanting, but it was left to H. Koechlin (this Journal, 1885, 115) to demonstrate the value of such preparations in cotton dyeing. Koechlin recommends the following quantities as most suitable for the preparation of the bath:—1 part of chromic acetate at 16° B., 3 parts of water, 2 parts of caustic soda at 36° B., $\frac{1}{18}$ part of glycerin at 28° B. Schmid prefers to dissolve precipitated chromic hydrate (17·3 kilos.) in caustic soda (40 litres at 30° B.), and to dilute with water (15—20) litres) and glycerin (1 litre at 28° B.). To preserve this solution, caustic soda may be added to it, the excess being saturated, before use, with the requisite quantity of chromic hydrate. The solution in any case, however, cannot be long preserved.

The following dyestuffs dye without assistance the mordant deposited from alkaline solution:—Magenta, Poirrier's Violet 140, Malachite Green, Phosphine, Methylene Blue, Alizarin, Alizarin Orange, Alizarin Blue, Anthracene Blue, Gallocyanin, Cœrulein, Logwood, Persian berries, Quercitron bark, Orchil. Bismarck Brown, Victoria Blue, the Eosines and Alkali Blues, dye with the addition to the bath of chrome alum.

*Calcium Plumbite.*—This compound has been extensively employed in the production on cotton of lead chromate yellows and oranges.

Good results are obtained by a modification of the usual method of mordanting, which consists in first impregnating the cotton with a solution of basic lead acetate and then passing it into a solution of calcium plumbite. Ammonium plumbite, for mordanting purposes, comes in value next to calcium plumbite, the plumbites of sodium and potassium not being sufficiently soluble to be of use.

*Sodium Zincate.*—This was first employed by Köchler and Weber. It is of importance in consequence of its use in connection with the dyeing on cotton of the Xylidine Ponceaux and certain Oranges. The cotton for this purpose is impregnated with a mixed solution of a chromic salt and magnesium acetate, dried, and passed into a solution prepared by dissolving 60 kilos. of zinc sulphate in 120 litres of caustic soda at 38° B. and 1,200 litres of water. It is left for some time, then it is rinsed and dyed. The mordant thus obtained, dyes well with Gallocyanin, Alizarin, Alizarin Orange, and Alizarin Blue. (See also this Journal, 1890, 386.)

"*Alkaline Antimony Mordant.*"—The solution proposed by Kohler has the advantage of giving to the fibre mordanted with tannic acid a larger quantity of antimony oxide than is obtained from tartar emetic; thus, whereas the latter gives to the fibre about 46·5 per cent. of the antimony trioxide it contains, the alkaline solution yields 61·2 per cent. It is necessary that the antimony oxide employed for the preparation of the alkaline solution should be free from antimony sulphide. By the aid of 100 grms. of glycerin, 10 grms. of caustic soda may be made to dissolve 20 grms., and 120 grms. of caustic soda, 120 grms. of antimony trioxide.

*Alkaline Iron Solutions* have been prepared by Balanche and others.—E. B.

*Turkey Red [Calico Printing], Alkaline Discharge of.* W. Trjapkine. Rev. Gén. 1898, **2**, 4—12.

The author discusses the influence of certain components of Turkey-red colour lakes on their capacity for being discharged.

Regarding the dyestuffs, less resistance to the alkaline discharge mixtures is offered by Anthrapurpurin, Flavopurpurin and Purpurin, than by Alizarin.

The proportion of fatty matters contained in the lakes is of less moment in the alkaline discharge style than in the older process of discharging. There is, however, a certain limit which must not be exceeded: thus, a solution containing per litre 300 grms. of Turkey-red oil of 50 per cent. strength gives colour-lakes which are discharged with difficulty in fine patterns.

On account of the sparing solubility of calcium alizarate, lime has great influence on the fastness to alkali of the Alizarin lakes, the difficulty of discharging increasing with the proportion of calcium present in them. Lime has less influence on the fastness of the trihydroxyanthraquinone colour-lakes.

The Turkey reds for the alkaline discharge process are dyed by ordinary methods, the only modification of importance being the employment of more concentrated Turkey-red oil solutions than were formerly used. The trihydroxyanthraquinone dyestuffs are preferably employed in dyeing, but mixtures of Alizarin and Anthrapurpurin are also used to produce reds of shades more closely resembling those dyed for the older discharge style.

The essential agents of the alkaline discharge are caustic soda and sodium silicate, which are thickened for printing with calcined starch, dextrin, gum Senegal, wheaten and maize starch. The last two give very thick colours, and are therefore only used for thickening the blue dyestuffs, which are not required to penetrate the tissue to the same extent as the other discharge colours. Thinner colours are used for the Alizarin lakes than for the lakes of other dyestuffs in order that the action may be more complete.

*White Discharge.*—The customary mixture of caustic soda, sodium silicate, glycerin, and turpentine, gives a satisfactory white on tissues dyed with Anthrapurpurin or mixtures of Anthrapurpurin and Flavopurpurin. In the case of reds dyed with Alizarin, the results are less pleasing, and in order to clear the whites, it is necessary to submit the tissues to a vigorous steam-chemicking.

Zinc dust has been tried as an addition to the white discharge colour, with the object of converting the Alizarin into desoxyalizarin, which has very little colouring power, but the improvement obtained is not sufficient.

It has been observed that certain metallic oxides, particularly zinc and stannous oxides, when admixed with the discharge colours, facilitate the production of a discharge. Such compounds are, consequently, employed in the discharge of the faster lakes; for instance, those dyed with Alizarin. The two metallic oxides do not act quite similarly. It is found that when the clearing bath used, has a density of 1°—1·25° B., pure whites are obtained with both; but that when the clearing bath is at 0·25° B., the sodium stannite mixture alone yields a white, the mixture containing sodium zincate giving impressions which are more or less coloured, not merely with the unremoved products of decomposition of the colour lake, but with the zinc alizarate which has been formed. A discharge mixture containing sodium zincate is especially serviceable for use in conjunction with the yellow discharge colour, since a concentrated clearing bath is required for use in connection with this. In other cases the sodium stannite mixtures are to be preferred. A special advantage attending the use of the latter, with reds dyed with Anthrapurpurin and Flavopurpurin, is that they enable the final clearing processes, namely, soaping and chemicking, to be dispensed with.

The sodium stannite mixture is prepared by adding stannous hydrate, or more commonly stannous chloride, to a thickened solution of caustic soda, to which a little glycerin has been added, at a temperature not exceeding 35°—40° C. Above this temperature, decomposition of the sodium stannite into sodium stannate and metallic tin occurs, and the results obtained in printing are less satisfactory. After the stannous salt has been added, the mixture is stirred occasionally during 1—2 hours before the sodium silicate is added. Finally turpentine is added. Not more than 2—3 days' supply of colour should be made at a time, as the colour does not keep well.

The sodium zincate mixture is obtained by adding zinc sulphate to the thickened caustic soda-glycerin mixture heated at 50°—60° C. Zinc oxide may be used in place of the sulphate.

The colours are composed as in the following examples:—*Alkaline thickening:* 3·75 kilos. of gum (? Senegal) solution (10:10), 3 kilos. of dextrin solution (10:10), 1·25 litres of water, 25 grms. (? kilos.) of caustic soda solution at 50° B., 1·75 kilos. of dry caustic soda. *Sodium stannite standard mixture:* 30 litres of alkaline thickening, 1·25 litres of glycerin, 6·5 kilos. of stannous chloride. *Alkaline white discharge with sodium stannite:* 10 litres of sodium stannite standard mixture, 3 litres of sodium silicate at 38° B., 50—75 grms. of Indigo (30 per cent. paste), 0·2 litre of turpentine. *Sodium zincate standard mixture:* 2·5 kilos. of gum Senegal solution (10:10), 2 kilos. of dextrin solution (10:10), 2 litres of water, 1 litres of glycerin at 28° B., 2 kilos. of zinc oxide, and 22 litres of caustic soda at 50° B. *Alkaline white discharge with sodium zincate:* 10 litres of sodium zincate standard mixture, 3 litres of sodium silicate at 38° B., 50—75 grms. of Indigo (30 per cent. paste), 0·5 litre of glucose at 35° B., and 0·2 litre of turpentine.

*Pink Discharge.*—Excellent results are obtained with half-discharge colours containing sodium stannite. The precise composition of the printing mixture is determined by the shade of pink to be produced, the nature and intensity of the red to be discharged, and the depth of engraving of the printing roller: thus, for an Anthrapurpurin Red, a printing colour containing per litre about 35 c.c. of caustic soda at 50° B. answers, while for a red dyed with equal quantities of Alizarin and Anthrapurpurin, a colour containing per litre about 120 c.c. of such a solution, is required. Gum Senegal is very suitable as a thickening agent for the pink discharge colours. The consistency of the colour should be rather thin, and the mixture should be made only as required. The tissues, after printing, must be well and very regularly dried. The following is a mixture suitable for discharging a mixed Anthrapurpurin-Alizarin Red:—3 litres of stannous hydrate in paste (corresponding with 500 grms. of stannous chloride per litre), 3 litres of water, 1·5 litres of glycerin at 28° B., 1·75 litres of caustic soda at 50° B. (to be added gradually, while keeping cold), 7·75 litres of gum solution (10:10), 3 litres of sodium silicate at 38° B., and 0·25 litre of turpentine.

*Yellow Discharge.*—This consists essentially of lead hydrate dissolved in caustic soda, and sodium silicate. The proportion of lead oxide is about 225—250 grms. per litre. This is more than can be obtained in solution at the ordinary temperature. The printing mixture, indeed, contains a certain quantity of lead hydrate in a state of suspension, which has separated out as the colour has cooled. It may also contain, as Ditte has shown, lead oxide, formed by the dehydrating action of the concentrated alkali solution.

By the addition of glycerin or glucose to the mixtures, a complete solution of the lead hydrate may be obtained. When, however, such compounds are employed, the fixation of the lead on the fibre is hindered: glucose is for this reason less to be recommended than glycerin.

The following mixtures yield yellow discharge effects, after steaming, clearing, and chroming:—*Yellow discharge with lead hydrate:* 2·75 litres of dextrin solution (10:10), 1·75 litres of water, 13·5 litres of caustic soda at 50° B., 11 kilos. of lead hydrate, 2·25 litres of glycerin at 28° B., 5 litres of sodium silicate at 38° B., 0·25 litre of turpentine. The lead hydrate for the above is prepared by precipitating 10 kilos. of lead nitrate, dissolved in 15 litres of water with 4·5 kilos. of caustic soda at 36° B., the precipitate being washed, drained, and pressed to a weight of 11·5 kilos. *Yellow discharge with lead nitrate:*—2·25 litres of dextrin solution (10:10), 1·75 litres of water, 15 litres of caustic soda at 50° B., 2 litres of glycerin, 9·25 kilos. of lead nitrate, 5 litres of sodium silicate at 38° B., 0·1 litre of turpentine. *Yellow discharge with lead chloride:* 4 litres of dextrin solution (10:10), 2 litres of water, 15 litres of caustic soda at 50° B., 8·5 kilos. of lead chloride, 2 litres of glycerin, 5 litres of sodium silicate at 38° B., 0·25 litre of turpentine. The lead chloride is prepared by dissolving 10 kilos. of lead nitrate in 15 litres of water, and precipitating with 37·5 kilos. of common salt solution at 10° B. The precipitate is washed, and pressed to 9 kilos. weight. *Pale yellow discharge:* 2·75 kilos. of lead nitrate, 1·5 litres of water, 1·5 litres of dextrin solution (10:10), 8 litres of caustic soda at 50° B., 3 litres of sodium silicate at 38° B., 0·2 litre of turpentine. (See also this Journal, 1884, 55, and 1896, 355.)—E. B.

### PATENTS.

*Coloured, Marbled, Variegated, or like Papers; A New or Improved Process for.* O. H. Stone, Birmingham. Eng. Pat. 8114, March 30, 1897.

Colouring matters in the solid state or in solution are applied to the paper pulp on the machine, preferably at the "wet" end of the machine. The colour may be either irregularly sprinkled on the pulp or applied in lines or other patterns by means of brushes or other mechanical device.—R. B. B.

*Hank-dyeing Machines, An Impt. in.* H. Bentley and J. W. Burgoyne, Bradford. Eng. Pat. 7809, March 26, 1897.

The improvement consists in the employment of a carrier mounted upon the sticks or rollers inside the hanks and adapted to move round with the hanks in order to prevent them from "lapping," *i.e.*, winding directly on to the sticks or rollers. The carrier may be made either of metal or of openwork cloth or netting.—R. B. B.

*Coloured or Decorative Effects on Paper or other Surfaces, A Process or Means for Producing.* J. Imray, London. From The Société Anonyme A. Roudillon et Cie., Paris, France. Eng. Pat. 10,901, May 1, 1897.

The surface of the paper, &c. on which the effects are to be produced is immersed in water. A solution of Judea bitumen in "crystallisable benzene," containing also colophony and kauri gum, is dropped on the surface of the water and spreads out, forming a pellicle which exhibits beautiful interference colours. These colours may be transferred to the surface of the paper, either by running off the water in the vessel, or by slowly unwinding the paper from a roller and drawing it through the pellicle. In either case the paper is dried by passing through heated chambers. The pattern of the colours may be varied by varying the form and positions of the drops which form the pellicle, by agitating the pellicle during formation, or by adding drops of substances which become insoluble on exposure to light. Designs may be formed on the coloured surface by means of stencils laid upon it so as to prevent the deposit of pellicle in certain portions, or by printing on the coloured surface.—R. B. B.

*Bleaching Cotton, Linen, and other Vegetable Textile Materials; Impts. in.* C. Delescluse, Marommes, France. Eng. Pat. 27,270, Nov. 20, 1897.

This patent describes a method of bleaching vegetable fibres, in which the preliminary treatment with alkali is entirely dispensed with, being replaced by special chemical treatment and subsequent stretching and drying operations. The chemical treatment consists in adding to the ordinary chlorine bath, a solution composed of about 2·5 kilos. of molasses, 2·5 kilos. of starch, and 10 kilos. of nitric acid at 36° B. These substances are mixed cold, the mixture heated and diluted with water to 1° B. This forms the bath in which are immersed bobbins holding skeins of the textile material, through which the liquid is drawn or forced. The molasses and starch of the mixture serve as a vehicle for the bleaching agents, reducing their corrosive action. The skeins of bleached material are taken from the round boxes in which they have been bleached, and are gradually stretched on reels, and in the stretched condition are exposed to steam and then dried by strong air currents. The cotton may, if necessary, be blued and dyed in the vat employed for the bleaching treatment.

The advantages claimed are that the fibre is not tendered, no yellowish tint is imparted to it, and the chlorine is entirely removed from the fibre.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Perchromic Acid, Alkali Salts of.* O. F. Wiede. Ber. 31, [5], 516—521.

According to a former communication, the author had succeeded in preparing only aniline and pyridine salts of the general formula $CrO_4.OM'$, where M′ is a monad radicle, the ammonia compound which he then prepared having the formula $CrO_4.3NH_3$. By using an ethereal solution of perchromic acid prepared as formerly described, cooling this to −5° or −10° C., and adding cooled ethereal solution of ammonia cautiously, avoiding excess, he has now obtained a precipitate which, after being freed from the ethereal liquid, drained, well washed with cold absolute alcohol and ether, and dried on a tile, forms a violet-black crystalline powder, the analysis of which corresponds with the formula $CrO_4.ONH_4 + H_2O_2$. The dry substance is stable for some days in the air, but moisture soon determines its decomposition, ammonium bichromate being left. On sudden heating, it hisses, and is converted into chromium trioxide with evolution of nitrogen oxides. It dissolves in ice-cold water to a neutral, blue-violet solution, rapidly decomposing in strong solution or when heated. Fixed alkalis act like water, while ammonia in the cold produces the compound previously described. Lead, barium, and silver salts give precipitates which rapidly decompose, whilst ferrous salts are at once peroxidised by it.

By adding to a cooled solution of perchromic acid prepared as above (but carefully separated from any crusts of ice deposited during cooling) a solution of potassium hydroxide in anhydrous ether, a similar compound of the formula $CrO_4.OK + H_2O_2$ was obtained. This resembles in its general behaviour the ammonium compound, but is less stable, the dry salt, at temperatures above 0° C., being comparable in explosiveness with iodide of nitrogen.

The author concludes that the original blue ethereal solution contains perchromic anhydride, $Cr_2O_9$; that water in presence of hydroxyl forms with this perchromic acid, $CrO_4.OH$, which can only exist in presence of (possibly as a compound with) hydroxyl; and that the hydroxyl shown in the formulæ of the salts is there as "hydroxyl of crystallisation."—J. T. D.

*Chromic Salts, Action of Alkaline Sulphites on.* A. Recoura. Bull. Soc. Chim. 1898, 19, 168—171.

When a large excess of sodium sulphite is added to a solution of a chromic salt, a green solution is obtained, in which the presence of chromium is not revealed by the ordinary reagents. When 3 mols. of sodium sulphite are added to the violet solution of 1 mol. of chromic sulphate, sulphur dioxide is evolved, and the colour changes to green; on boiling, the whole of the chromium separates out as a flocculent precipitate of the composition $2Cr_2O_3SO_2$. By evaporating the solution on the water-bath, a residue of the composition $Cr_2O_3SO_2$ is obtained, which gives a colloidal solution with water. These are not true basic salts, but contain the chromium in the form of a complex radicle, which is somewhat slowly decomposed, with precipitation of chromic hydroxide, by caustic soda or ammonia. The time required for the formation of a precipitate increases with the excess of sodium sulphite present, but is diminished by raising the temperature or using larger quantities of the reagent.—T. E.

*Sodium Carbonate, Native.* W. P. Blake. Eng. and Mining J. 1898, 65, [7], 188.

There has been recently examined, at the Arizona School of Mines, a sample of crude carbonate of soda from a deposit situated in the extreme north of the State of Sonora, Mexico, in the mining district of Altar, about

2½ miles from Adair Bay and directly upon the shore of the Gulf of California. The carbonate is found in the central portion of a basin-shaped depression, about 1 mile in length and breadth, and is spread out in a layer over an area of about 60 acres. The deposit varies in thickness from 1 to 3 feet, and is partly overlaid by a stratum of sandy earth about 3 in. thick. The lowest portions of the deposit are saturated with a solution of the carbonate in water; this solution underlies the bed for a depth of 2 ft., and is so concentrated that, when exposed to the air and sun, sodium carbonate rapidly crystallises out and forms in layers to a depth of nearly 3 ins., in less than 30 days. In the centre of the deposit is a spring or water hole, where potable water may be had in quantity. The sodium carbonate, when obtained on the large scale by digging, is mixed with insoluble clay and very fine sand, an earthy impurity which probably has been blown in from the surrounding hills. The following is an analysis of an average sample from the deposit:—Sand, silt, &c., insoluble in water, 13 per cent.; iron oxide and alumina, 2·8 per cent.; lime, 1·14 per cent.; salt, 4·7 per cent.; $Na_2SO_4$, 4·7 per cent.; $Na_2CO_3$, 75·86 per cent. The sample, leached with cold water, gave 75 per cent. of clean white carbonate of soda, including the small quantity of other soluble salts. Hot water gives a larger yield; but, on ignition, the product obtained by lixiviation becomes much reduced in weight, a considerable amount of water being driven off. A sample of selected crystals, when examined by R. H. Forbes and M. H. Walker, showed a specific gravity of 2·33, and contained some bicarbonate of soda, a trace of chlorine and sulphates, but no sulphides, lime, barium, silica, potassium, or ammonia. The equivalent of 71·96 per cent. of sodium carbonate was indicated on titration with standard acid.

It is estimated that the deposit will yield 1,000 lb. of the carbonate per square yard of area, and that the total cost of digging and placing on ship-board will be about 2 dols. per ton.—A. S.

### *Caustic Soda.* K. W. Jurisch. Zeits. angew. Chem. 1898, 174—176.

Owing to the greater purity of the sodium carbonate obtained by the ammonia-soda process compared with that made by the Leblanc process, when ordinary caustic soda is manufactured from the former, in order to reduce the test of the product, the carbonate is incompletely causticised, the liquor is not allowed to cool, and hence the so called "caustic salts" are not removed, and sodium chloride is added. The green colour due to the presence of manganate derived from the manganese of the lime is removed by adding sulphur. These differences in the processes render it easy to distinguish the products, and the analyses quoted below show that in each case the material was obtained from an ammonia-soda product.

1 is a sample of the outer white portion of a drum of caustic soda manufactured in a works in Bohemia, 1*a* the flesh-coloured to grey parts of the central core, which also contained yellowish and bluish-green portions; 2 is a sample of the outer light flesh-coloured portions of a drum of caustic soda, made in an Austrian ammonia-soda works; 2*a* the dark grey portion of the central core, which also exhibited coloured portions similar to 1*a*, only darker.

| — | 1. | 1*a*. | 2. | 2*a*. |
|---|---|---|---|---|
| NaHO | 86·740 | 86·240 | 84·220 | 84·174 |
| $Na_2CO_3$ | 6·157 | 5·874 | 8·353 | 6·807 |
| $Na_4SiO_4$ | 0·818 | 1·229 | 0·831 | 1·669 |
| NaCl | 3·979 | 3·804 | 4·692 | 4·456 |
| $Na_2SO_4$ | 2·747 | 2·522 | 3·381 | 2·900 |
| $Na_2SO_3$ | 0·034 | 0·019 | 0·003 | 0·001 |
| $Na_2S_2O_3$ | 0 | Trace | 0·002 | 0·001 |
| $Na_2S$ | 0·001 | 0·001 | 0 | 0 |
| $Fe_2O_3$ | 0·002 | 0·023 | 0·002 | 0·016 |
| $Al_2O_3$ | 0·001 | 0·016 | 0·008 | 0·018 |
| $Mn_2O_3$ | 0·004 | 0·006 | 0·007 | 0·017 |
| CaO | 0·005 | 0·093 | 0·013 | 0·174 |
| MgO | 0·002 | 0·011 | 0·006 | 0·006 |
| CuO | 0 | 0 | .. | 0·007 |
| KCl | Not detd. | Not detd. | Not detd. | Not detd. |
|  | 100·490 | 99·838 | 101·518 | 100·246 |

The cores in each case contain about 10 times the quantity of earthy and metallic impurities present in the outer portions. The copper oxide in 2*a* is derived from Robert's apparatus. In order to get rid of the core, or at any rate to diminish its size, the molten caustic soda must be allowed to settle, and care must be taken not to stir up the sediment in transferring to the drums.—E. W. W.

### *Ammonia-Soda Process, Waste Liquors of the.* K. W. Jurisch. Zeits. f. angew. Chem. 1898, [6], 130—135.

Schreib and Lunge have criticised figures formerly given by the author, disregarding the fact that those figures were given as the extreme limits between which the composition of ammonia-soda waste liquors may vary. The author now presents a number of figures representing the various conditions obtaining in various methods of working.

*a. Most Concentrated Liquors.*—Mallet-Boulouvard's system, distillation conducted by a steam-coil, so that the heating steam does not dilute the liquors. For every 1,000 kilos. of soda there are produced on an average 7,367 litres of waste liquor of 18° B., containing, per 100 c.c., 7·0 grms. of NaCl, 16·0 grms. of $CaCl_2$, 2·7 grms. of CaO as hydroxide and carbonate, and 0·005 grm. of $NH_3$. The monthly averages for several months lay between 6·30 and 7·10 NaCl, and between 0·0049 and 0·0070 $NH_3$.

*b. Most Dilute Liquors.*—In a small work, on Wegelin and Hübner-Pollaczek's system, furnished later on with two Savalle's columns, the distillation was carried on slightly above atmospheric pressure. Average per 1,000 kilos. of soda, 13,000 litres of waste liquor of 11°·5 B. The following table shows for a year (1882-83) the lowest, highest, and average figures for each constituent in grms. per litre:—

| — | Minimum. | Maximum. | Average. |
|---|---|---|---|
| Degrees Baumé | 9 | 13 | 11·5 |
| NaCl | 55 | 135 | 80 |
| $CaCl_2$ | 15 | 50 | 25 |
| $Ca(OH)_2$ | 17 | 32 | 25 |
| $CaCO_3$ | 2 | 40 | 5 |
| $NH_3$ | 0 | 0 | 0 |

Schreib and Lunge both commented on these numbers; it must be admitted that they would only occur in a small and badly-conducted work.

*c. Mean Liquors.*—Some figures from the practice in Solvay works. For daily tests, three determinations were made; the alkalinity ($Ca(OH)_2$, $CaCO_3$, $NH_3$) in 10 c.c. was titrated with normal acid, the free ammonia in 100 c.c. was determined by distillation into normal acid and back-titration, and the total chlorine in 1 c.c. titrated by silver nitrate (1 c.c. = 0·00355 grm. Cl). The figures which follow are examples:—

| — | English Work, July 1880. | Austrian Work. |  |  |  |
|---|---|---|---|---|---|
|  |  | February 1885. | May 1885. | December 1885. | May 1887. |
| Alkalinity | 14·5 | 12·8 | 10—18 | 4·5 | 5—12 |
| Ammonia | 0·6 | 0·8 | 0·2—2·5 | 0·75 | 0·2—0·4 |
| Chlorine | 27·2 | 21·6 | 12—25 | 18·0 | 35—40 |

In 1887 the process was worked with addition of salt; in 1885 without.

The author gives tables showing the hourly or half-hourly tests on March 23rd, 1885, of a Mond distillation tower after seven weeks' continuous use, the day before cleaning; working with addition of salt. In the third table two sets of figures are given, one in which the sample of liquor was delivered from the tower into an open vessel, the other in which it was delivered under water; the difference shows the escape of ammonia from the liquor when the pressure is suddenly reduced from that in the tower to that of the atmosphere. On the next page are given the maximum, minimum, and average figures from these tables.

### I.—Waste Liquors.

| — | Minimum. | Maximum. | Average. |
|---|---|---|---|
| Density | 1·037 | 1·125 | 1·0895 |
| Grms. per litre:— | | | |
| $CaH_2O_2$ | 0·370 | 60·754 | 21·900 |
| $CaCO_3$ | 5·400 | 25·150 | 13·467 |
| Free $NH_3$ | 0·038 | 1·972 | 0·365 |
| $NH_4Cl$ | .. | 7·169 | 0·597 |
| $CaCl_2$ | 15·984 | 69·042 | 49·048 |
| NaCl | 20·768 | 104·950 | 59·818 |
| Water, sand, and clay | 923·966 | 950·496 | 941·131 |
| Equivalents of lime | 0·88 | 6·70 | 2·02 |
| C.c. of standard solutions:— | | | |
| Alkalinity (10 c.c.) | 2·24 | 19·56 | 8·851 |
| Ammonia (100 c.c.) | 0·23 | 25·00 | 3·2675 |
| Chlorine (1 c.c.) | 6·40 | 26·00 | 19·7375 |

### II.—Milk of Lime.

| | | | |
|---|---|---|---|
| Density | 1·019 | 1·172 | 1·100 |
| Grms. per litre:— | | | |
| $Ca(OH)_2$ | 30·525 | 269·545 | 159·57 |
| $CaCO_3$ | 1·00 | 13·75 | 7·33 |
| Water, sand, and clay | 884·02 | 987·225 | 933·10 |
| Total CaO | 23·80 | 207·48 | 125·70 |
| Per cent. as $Ca(OH)_2$ | 93·40 | 98·31 | 95·89 |

### III.—Loss of Ammonia.

#### A.—*Sample taken in Open Vessel.*

| | | | |
|---|---|---|---|
| Density | 1·112* | 1·113* | 1·099 |
| C.c. of standard solutions:— | | | |
| Alkalinity | 8·87 | 1·91 | 11·967 |
| Ammonia | 0·10 | 11·58 | 1·237 |
| Chlorine | 21·35 | 26·23 | 16·982 |
| $NH_3$, grms. per litre | 0·017 | 1·969 | 0·210 |

#### B.—*Same Sample under Water.*

| | | | |
|---|---|---|---|
| $NH_3$, grms. per litre | 0·051 | 2·165 | 0·316 |

* The figures in each of these columns refer to one sample only, containing the maximum and minimum respectively of ammonia.

These observations were made in a work possessing two distillation towers, one of Mond's pattern, the other of more recent construction, with nine divisions in the lower part. For a daily output of 20,000 kilos. of soda, each tower yielded about 220 cb. m. of waste liquor. This would sometimes rise to 300 cb. m., but with such large bulks it would be preferable to increase all the dimensions of the tower, and to have 11 or 12 divisions in the lower part.
—J. T. D.

*Calcium Sulphate, Action of, on Alkali Haloid Salts.* A. Ditte. Comptes Rend. **126**, [10], 694—700.

When, in presence of excess of potassium chloride, solutions of potassium sulphate and calcium chloride are mixed, nothing is observed so long as the proportion of the latter salt is below a certain limit, but when this limit is passed, there is formed the double salt $K_2SO_4.CaSO_4.4H_2O$, which deposits in fine needles. For every mixture of calcium chloride and potassium sulphate, a state of equilibrium is reached when a certain proportion of this double salt has been formed and deposited, and by greatly increasing the amount of calcium chloride, it is possible to remove the sulphuric acid completely.

When gypsum in excess is allowed to react on solutions of potassium chloride of varying strengths, it appears to dissolve simply in the weaker solutions; but with stronger solutions the amount of CaO contained in the liquid is more than would correspond with the $SO_4$ found at the same time, pointing to a decomposition of the alkali chloride by the calcium sulphate. From these strong solutions the double salt mentioned above always separates. The same thing occurs with the bromide and iodide of potassium, and with the chlorides of sodium and ammonium, though in the case of the two last there is no separation of double salt.

It is to be noted that the reactions between calcium sulphate and the alkali haloids are all accompanied by very small heat changes, and that the sign of the heat-change in a given reaction may be reversed by greater or less dilution, so that probably, in the case of the potassium salts, the heat of formation of the double salt is an important factor in determining exchange.—J. T. D.

*Bauxite, the Commercial Analysis of.* W. B. Phillips and D. Hancock. J. Amer. Chem. Soc. 1898, **20**, [3], 209.

*See under* XXIII., *page* 378.

## PATENTS.

*Ammonia, Nitrate of, Process for the Separation and Purification of.* R. N. Lennox, London. Eng. Pat. 5483, March 2, 1897.

Ammonium sulphate and a nitrate, such as sodium nitrate, are mixed, nearly in molecular proportions, though preferably with a slight excess of one of the salts, and the mixture is heated in an iron or other suitable retort to a temperature not exceeding 230° C., while a vacuum or a pressure not exceeding, at most, 6 ins. of mercury, is maintained in the apparatus. Ammonium nitrate distils over into the earthenware receiver, and may be removed by aid of a steam jet, and re-distilled *in vacuô*, to obtain it in a pure state. "The scrubber is charged with a strong solution of nitrate of ammonia, or other material capable of absorbing any nitric acid and ammonia which may result from dissociation of nitrate of ammonia, so that vapours of neither nitric acid or ammonia reach the pump or exhauster."—E. S.

*Sodium Chloride or other Salts, Impts. in Apparatus for Electrolysing.* F. Taylor, A. Cooke, and H. W. D. Montgomery, Belfast. Eng. Pat. 6929, March 17, 1897.

*See under* XI. A., *page* 358.

*Ferric Sulphate, Impts. in the Means or Method of Manufacturing.* W. Wyld and J. H. Cox, Bradford. Eng. Pat. 8335, April 1, 1897.

Sulphuric acid is used in the heated state as it leaves the Glover tower, or other apparatus in which it is manufactured, to act upon powdered burnt iron pyrites, to form ferric sulphate. The sulphurous fumes and dust are collected for recovery of the sulphurous acid. If, instead of burnt pyrites, the iron oxide be employed, which is obtained as a residue when "spent oxide" from gas purifiers is burnt in sulphuric acid making, it is taken while hot for treatment, either with cold or hot sulphuric acid. The mixture of the iron oxide and acid is effected in an iron pan, with stirring.—E. S.

*Nickel Sulphate, Impts. in the Production of.* T. Savage, Birmingham. Eng. Pat. 9099, April 9, 1897.

Nickel or nickel ore is digested in sulphuric acid with addition of sodium nitrate. The proportions claimed are, 4 parts of sulphuric acid, 2 parts of nickel, and 1 part of sodium nitrate, the acid being diluted to a density of 1·2. The mixture is heated by steam pipes, and the nickel sulphate is crystallised out from the saturated solution.—E. S.

*Alkaline Cyanides and other Cyanated Products, An Improved Process for Manufacturing.* H. R. Vidal, Paris. Eng. Pat. 9215, April 10, 1897.

"Phospham" ($PN_2H$), mixed with sodium or potassium carbonate and strongly heated, yields a cyanate and a phosphate, according to the equation—

$$PN_2H + 2K_2CO_3 = K_2HPO_4 + 2KCNO.$$

The cyanate is dissolved out by water or alcohol. If carbon be added to the mixture, a cyanide is obtained on heating; or if iron be substituted for carbon, a ferrocyanide is produced. Sulphur, added to the original mixture with carbon, gives a sulphocyanide. To obtain cyanogen, a mixture of phospham and of dried potassium oxalate is heated, the reaction being represented by the equation—

$$PN_2H + K_2C_2O_4 = K_2HPO_4 + C_2N_2.$$

On replacing the normal by acid oxalate, with addition of carbon, hydrocyanic acid is obtained.—E. S.

*Nitric Acid, Process and Apparatus for Obtaining from Low-Graded Nitro-Oxygen Compounds.* P. Jensen, London. From L. Rohrmann and H. H. Niedenführ, Krauschwitz, Germany. Eng. Pat. 29,746, Dec. 15, 1897.

The regenerating nitro-oxygen compounds pass through a series of reaction columns, the last of which is elevated above the others, and is fed with water, which may be heated. The outflow from this column is led to sprinkle into the lower columns, which are ranged on a level, and are packed successively towards the upper one, with an increasing number of filling bodies (by preference the Lunge-Rohrmann plates,) " in order to meet artificially the weakened reaction consequent on the decreasing concentration of the gases under treatment." The liquid product from the lower columns runs into a receptacle, whence it is drawn off as pure acid.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Glassware for Chemical Purposes, Judging.* F. Förster. Zeits. für Instr.-kunde, **13**, 457; Dingler's Polyt. J. **307**, [7], 165—166.

The author points out that the action of water on glass is attributable to the liberated alkalis, and he regards the condition of the dissolved glass as one of distension rather than true solution.

At the ordinary temperature only a minute amount of silica is dissolved from the glass, but at 100° C. the quantities of alkali and silica dissolved are about equal, and though the latter is increased at higher temperatures, the ratio never corresponds to that existing in the glass.

In water-gauge glasses the proportion of lime is an important factor, window-glass, rich in lime, being superior to hard combustion tubing, richer in silica, whilst the best glass for the purpose is the Jena thermometer glass 59''', used for lining compound gauge glasses.

The solvent action of $\frac{1}{1000}$N alkali is little greater than that of water. 2N-alkali dissolves lime-alkali glass as such. Increasing the strength of the alkali has little effect, and concentrated alkali has less solvent power than dilute. Caustic soda is the strongest, and potash, ammonia, and baryta follow in the order named.

The presence of alkali carbonates increases the solvent action of water, but differently according to the kind of glass, the chief point in the reaction being the formation of calcium carbonate from the silicate.

The action of acids was determined by the loss in weight sustained by glass flasks after 6 hours' exposure at 100° C., the loss being found constant for each kind of glass, irrespective of the acid used—sulphuric, hydrochloric, nitric, or acetic; the strength thereof—$\frac{1}{1000}$N to 10N; or the temperature, 100° to 270° C. Very highly concentrated acids have a less action, and in no case is the glass attacked to such an extent as by pure water. This behaviour of acids is apparently due to their neutralisation of the alkali dissolved from the glass.

In comparing the lime-soda glasses with the other lime-alkali glasses containing less silica, the latter suffer more under the action of acids than of water, and more from concentrated acid than dilute. The same applies to the fused meta-silicate, $Na_2SiO_3$, whilst with water glass, ($Na_2O$, 3 $SiO_2$) the converse is the case as regards the concentration of the acid.

Lead glasses containing sufficient silica, are less susceptible to acid than to water, but when the lead content increases (*e.g.*, flint glass), a reversal occurs.

On good glass, concentrated sulphuric acid acts only when vaporised; gaseous carbon dioxide only attacks glass the surface of which is already decomposed through the influence of moisture.

The subjoined tables give the chemical composition and susceptibility of the best glasses for chemists' use: the sample No. 1, which approximates to Stas's glass, being one of the most resistant glasses now employed.

I.

| No. of Glass. | $K_2O$. | $Na_2O$. | CaO. | MnO. | $Al_2O_3$ (+ $Fe_2O_3$). | $SiO_2$. | $R_2'O : CaO : SiO_2$. | No. of Alkali Molecules per 100 mols. |
|---|---|---|---|---|---|---|---|---|
| 1 | 6·2 | 6·4 | 10·0 | 0·2 | 0·4 | 76·8 | 0·95 : 1 : 7·16 | 10·4 |
| 2 | 7·0 | 8·3 | 8·1 | .. | 0·3 | 76·3 | 1·44 : 1 : 8·80 | 12·7 |
| 3 | 11·8 | 4·9 | 7·6 | 0·1 | 0·5 | 75·1 | 1·50 : 1 : 9·24 | 12·8 |
| 4 | 4·3 | 10·0 | 7·8 | .. | 0·3 | 77·6 | 1·38 : 1 : 9·28 | 12·6 |
| 5 | 4·6 | 10·1 | 7·7 | .. | 0·4 | 77·2 | 1·54 : 1 : 9·36 | 13·0 |
| 6 | 0·6 | 14·3 | 11·2 | 0·4 | 2·9 | 70·6 | 1·18 : 1 : 5·88 | 14·6 |
| 7 | 9·7 | 9·0 | 6·8 | .. | 0·4 | 74·1 | 2·04 : 1 : 10·17 | 15·4 |
| 8 | 6·7 | 13·7 | 7·2 | 0·3 | 3·2 | 68·9 | 2·27 : 1 : 8·91 | 18·6 |

II.

| No. of Glass. | Alkali extracted by Water. (Expressed in thousandths of a mgrm. of $Na_2O$ per 100 sq. cm. of Surface.) | | Loss in Weight (mgrms. per 100 sq. cm. of Surface) after Three Days' Exposure at 100° C. to | |
|---|---|---|---|---|
| | After Eight Days' Exposure to Water at 20° C., following Three Days' Treatment with Water at the same Temperature. | After Three Hours' subsequent Exposure to Water at 80° C. | 2N Caustic Soda. | 2N Sodium Carbonate. |
| 1 | 13 | 27 | 37 | 59 |
| 2 | 14 | 56 | 40 | 77 |
| 3 | 14·5 | 45 | 38 | 79 |
| 4 | 15 | 50 | 38 | 73 |
| 5 | 18 | 66 | 42 | 79 |
| 6 | 27 | 98 | 31 | 41 |
| 7 | 32 | 217 | .. | .. |
| 8 | 77 | 654 | 46 | 45 |

—C. S.

[*Glass*], *Dry Etching.* Sprechsaal, **31**, [4], 88—89; [5], 121.

In this process ammonium fluoride is employed, its hygroscopic properties being utilised as follows:—The design (which must be of comparatively small dimensions) is applied to the glass by printing thereon with " strong " printer's varnish from an india-rubber plate, or in any suitable way. Whilst the impression is still moist, some powdered ammonium fluoride (kept at a temperature of about 105° F.) is applied to the surface with a soft brush, and caused, by gentle pressure, to adhere to the varnish, all the superfluous salt being carefully removed. In about 15—60 minutes, according to the degree of atmospheric moisture prevailing, the fluoride will have become liquid, and have penetrated to and etched the glass underneath. Better results can be obtained by printing with a medium soluble in water, *e.g.*, thick syrup, and by incorporating the fluoride therewith; or the design may be printed with the etching material direct, a mixture of 60 parts of fuming hydrofluoric acid, with 100 parts of dry ammonium fluoride, and 20 parts of sodium fluoride being used. In this latter method the rubber printing-plate must be held in moderately firm contact with the glass for $\frac{1}{4}$ to $\frac{1}{2}$ a minute. For touching up the print, or for etching with the pen, a fluid, freshly prepared from a little finely ground ammonium fluoride and sufficient sulphuric acid to produce an oily liquid, is used, the ordinary etching fluids sold for this purpose being less suitable, owing to their content of alkali silicate, which has a tendency to run off the pen more quickly than the other constituents of the liquid, and so cause blots and irregular etching.—C. S.

*Photo-Ceramics in Three Colours.* P. C. Duchochois. Anthony's Bull., through Scient. Amer. Suppl. 1898, **45**, 18,439.

*See under* XXI., *page* 373.

## PATENTS.

*Annealing Glass, Impts. in and relating to Leers or Kilns for.* A. D. Brogan and A. M. Malloch, both of Glasgow, and J. Craig, Hamilton, Lanark. Eng. Pat. 6840, Mar. 16, 1897.

The leer consists of a long brickwork chamber provided with rails or other supports adapted for carrying a number of light metal lattice-work frames for holding the glass plates. The frames are arranged parallel to each other in the leer, and are either vertical or inclined, and rest upon small rollers. A plate of glass, as it leaves the casting table, is pushed horizontally through an opening at the hot end of the leer, where it is met by a frame, which has been run on a bogie into a position to receive it. The bogie being in the line of the leer, the frame is raised by lifting gear into a vertical or inclined position, and is so carried along the length of the chamber by means of travelling endless chains. It is said that in this way a larger quantity of glass can be annealed in a given time, and with less risk of breakage, than is possible with the usual form of leer, in which the glass plates are fed forward horizontally.

—H. H. B. S.

*Pottery Kilns or Ovens, Impts. in.* R. F. Ayre and Craven Dunnill and Co., Ltd., Jackfield, Salop. Eng. Pat. 11,343, May 6, 1897.

Provision is made for the more perfect combustion of the fuel in that class of kiln in which the heated gases, instead of coming into contact with the goods, pass through flues surrounding the chamber in which they are packed. The principle consists in introducing air, preferably in a heated condition, into the flues surrounding the oven through which the gases of combustion pass on their way to the chimney.—H. H. B. S.

# IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Mortars, Hydraulic, Study of the Ultimate Constitution of.* R. Feret. Bull. de la Soc. d'Encouragement, 1897, [12], 1591—1625.

A series of tests was made with mortars containing different varieties of sand mixed with varying quantities of water, concerning which full details are given. After hardening for 48 hours in damp air, the test blocks were immersed in soft water for five months, and were then tested to destruction under different stresses. The transverse tests of square prisms, 16 × 4 × 4 cm., were practically the same as those of 13 × 2 × 2 cm. (10 cm. between the supports), when both sets of results were compared on a given unit basis. Any great variation in size, or want of homogeneity (as through superficial carbonating), would lead to non-proportionality. In tension, the smaller pieces were found to be relatively stronger than the larger blocks, the strength of blocks with a section of 16 sq. cm. being to that of similar blocks of 5 sq. cm. in the proportion of 0·65 : 1. After long term immersion (*e.g.*, six years) in sea-water, the tensile strength of mortars tends to become a constant quantity, independent of the binding medium and of the proportions of the mortar. The ratio between the tensile strength per sq. cm. and the transverse strength, multiplied by $\frac{3}{2} \times \frac{l}{b\,d_2}$ is, in round numbers 1 : 2, where $l$ = length, $b$ = breadth, and $d$ = depth of the transverse bars. Under compression the area of the test block exerts but little influence, provided that the height of each of the test pieces be nearly the same as its diameter. With compression pieces that have been exposed for six years to the action of sea-water, the strength of the material was practically proportional to the energy of the cement used; in this respect there is a marked difference between tensile and compressive stresses. After very many trials it is believed that compression tests are far more reliable than those by tension, and that the results are more uniform.

*Volumetric Elementary Composition of Mortars.*—The hardness, resisting power, porosity, and other qualities of hydraulic mortars depend not only upon the nature of the cement, but upon the sizes and relative positions of the constituents of the mortars. The absolute volumes of the cement ($c$), sand ($s$), water ($w$), and interstitial spaces ($i$), forming a litre of the fresh mortar, may with advantage be studied in determining the best composition to employ.

*Relation between Resistance to Compression and Volumes of Constituent.*—The following law has been arrived at after many experiments:—For all plastic mortars made with the same cement and with inert sand, the resistance to compression, after the same period and under identical conditions, is solely a function of the ratio $\frac{c}{w + i}$ or $\frac{c}{1 - (c + s)}$, whatever the nature and size of the sand-grains, and the proportion of the constituents, cement, sand, and water, of which each is composed. This ratio is not quite accurate, as the compressive strength (C) augments somewhat more rapidly than the ratio, and the formula finally adopted is $C = K\left(\frac{c}{1 - s}\right)^2$, where K is a constant characterising the energy of the cement used. In certain tests made K = 1965.

*Normal Tests of Mortars.*—Unless all the conditions of material, mixing, keeping, and testing be identical, the ordinary results of tensile testing are, for comparison, useless. But there is great difficulty in specifying these conditions exactly. All these difficulties (which are here dealt with at length) vanish, however, if compression tests be effected with blocks prepared from plastic mortars (made with inert sand), the ultimate volumetric composition of which is determined. The tests are made after the normal lapse of time, and thus the value of K is found, as shown above, for all mortars made with the same cement. This value may be termed the co-efficient of resistance. In specifications of tests, it would suffice to stipulate that the mortar should be made of, say, 1 part of cement with 3 parts of silicious sand, of such size that it passes a sieve with 2-mm. holes, but is retained on one with 0·5-mm. holes, and it should be mixed with sufficient potable water to give it a good plastic consistency. Cubes of 5 cm. in width may be used, and a 20-ton testing machine would then be sufficiently large for the work.

*Proportioning of Mortars.*—It is in general chiefly necessary to compare the net price and the essential properties of the mortars. Series of tests should be made of mortars composed of the particular cement selected, and different proportions of the several sands available, taken one by one. Curves are then constructed in which the abscissæ represent the net prices and the ordinates the compactness ($c + s$), and the resistance to compressive stress of the resulting mortars. The various curves are then compared, and the best sand for any given industrial purpose may be readily found.

*Granulometric Composition.*—The size of grain is all-important. The author has tested mortars made with sands just retained by sieves with 5, 2, and 0·5-mm. holes respectively, and has plotted the results in triangular form [by a method similar to that suggested by Stokes for ternary alloys], and has found that the indication of the proportion of cement in a mortar gives no clue to the properties of the mortar unless the character of the sand be also defined. A mortar with only 300 kilos. of cement per cb. m. of a sand, may give a better test than another with 600 kilos. of the same cement per cb. m. of another sand.

*Mortars with Three Solid Constituents.*—Where a fine material is used, in character resembling sand, the equation given above would be $C = K\left(\frac{c}{1-(m+s)}\right)^2$, where $m$ represents the volume of the added matter. Where it takes the place of cement (*e.g.*, in slaked lime), the equation would be $C = \frac{(K_1c + K_0m)\,(c+m)}{(1-s)^2}$, where $m$ is the volume of the added material and $K_1$ and $K_0$ corresponding co-efficients for the two cementing media. Further modifications are given for pozzuolanic additions. In all cases a series of tests should be made with mixtures with three variants, the results being plotted graphically by the triangular method.

*Pozzuolanic Additions.*—The addition of pozzuolanic material not only increases the strength of the mortar, but provides a material for the neutralisation of free lime. Some varieties of blast-furnace slag, when granulated, give a greater strength to the mortar than natural pozzuolanas. But if required to resist the chemical action of sea-water, the mortar would probably be better if a more silicious material than the highly aluminous blast-furnace slag were employed.—W. G. M.

### *Hydraulic Lime, Natural and Artificial; Theory of the Setting of.* K. Zulkowski. Chem. Ind. 21, 69 and 96.

Blast-furnace slag, to be successfully used as a material for hydraulic mortar, must be rich both in lime and in alumina. Granulation is also necessary, and the manner in which this is carried out is of importance, it being desirable that solidification should take place in the shortest possible time. The hotter and more fluid the slag, and the quicker it is cooled, the better the result. The author does not agree with Tetmaier, in supposing that the change which takes place on granulation is due partly to dissociation; but, on the contrary, is of opinion that dissociation is actually hindered thereby. Slag in which the lime rises above a certain limit is known to crumble to powder on cooling slowly in the air. A change appears to take place analogous to the devitrification of glass. Experiments carried out upon glass which had undergone this change led to the conclusion that the phenomenon was due to the separation of crystals of calcium monosilicate ($CaSiO_3$), and, according to the author's views, when slag is slowly cooled, the excess of lime becomes separated in a similar way. The hydraulic character then disappears, notwithstanding that the percentage composition of the whole mass remains the same. When, however, a suitable slag is cooled suddenly with water, it is found to possess hydraulic properties.

Portland cement clinker is also known to fall to powder sometimes on cooling, and when this takes place, the product has less hydraulic value. It has been proposed on this account to cool such clinker under a jet of steam, which the author suggests may accelerate the cooling, and thus produce somewhat the same effect as the granulating of slag. A clinker that falls to powder on cooling spontaneously in the air will become and remain hard if cooled in water.

The author suggests that molten or granulated slag contains a hydraulic body of the formula—

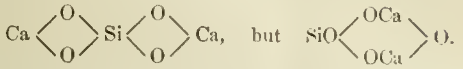
$$Al_2O_3,\ (m + n)SiO_2,\ (x + y)CaO,$$

capable of splitting up into two silicates of the formulæ—

$$m\,SiO_2 + Al_2O_3 + x\,CaO \text{ and } n\,SiO_2 + y\,CaO,$$

which harmonises with the fact that it is only slags and glasses rich in lime as well as in alumina that are liable to devitrification. Admitting, however, that in basic aluminic slag the alumina is present in the molten mass as a double silicate, which separates into two silicates upon slowly cooling, it still remains to be explained why such slag should possess hydraulic properties if cooled suddenly with water.

According to Kosmann, pure lime slag is not a neutral orthosilicate, but a basic metasilicate. The product obtained upon heating together silica and lime, in the requisite proportions, is not—

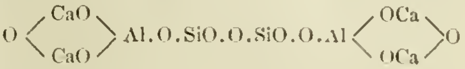


$$Ca\left\langle\begin{matrix}O\\O\end{matrix}\right\rangle Si\left\langle\begin{matrix}O\\O\end{matrix}\right\rangle Ca, \quad \text{but} \quad SiO\left\langle\begin{matrix}OCa\\OCa\end{matrix}\right\rangle O.$$

It is possible that at lower temperatures orthosilicate is formed, and at higher temperatures metasilicate, and the author thinks that setting is due more to the hydration of calcium metasilicate than to the formation of some other compound.

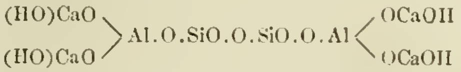
The behaviour of alumina and lime at high temperatures is quite analogous to that of silica and lime, a basic anhydrous lime compound being formed, which is capable of taking up water and setting.

In regard to the behaviour of alumina to lime, the author inclines to the belief that a basic silicate of the following composition is produced:—



$$O\left\langle\begin{matrix}CaO\\CaO\end{matrix}\right\rangle Al.O.SiO.O.SiO.O.Al\left\langle\begin{matrix}OCa\\OCa\end{matrix}\right\rangle O$$

which, upon hydration, becomes—



$$\left.\begin{matrix}(HO)CaO\\(HO)CaO\end{matrix}\right\rangle Al.O.SiO.O.SiO.O.Al\left\langle\begin{matrix}OCaOH\\OCaOH\end{matrix}\right.$$

Such a body would lose, on ignition, 7·45 per cent. of water, whilst a slag cement, which, after setting, showed nearly the same composition, yielded 6—8 per cent. of water of hydration.

Basic furnace slags suitable for the production of cement will, if highly calcareous, set without the further addition of lime, though such addition is advantageous. With slags of less basic character, however, the addition of lime is absolutely necessary. The author is of opinion that Portland cement, like slag cement, is a mixture of a hydraulic body with so much quick-lime as is left uncombined after the process of burning. Attempts were made to prove the presence of such free lime by extraction with alcoholic solutions of anhydrous and crystallised magnesium nitrate, as explained by the following equations:—

1. $Mg(NO_3)_2 + CaO = Ca(NO_3)_2 + MgO$
2. $Mg(NO_3)_2 + CaO_2H_2 = Ca(NO_3)_2 + MgO_2H_2$

The attempts, however, failed. It was found that whilst, in the first place, quick-lime was only partially attacked, in the second case the combined lime, as well as the free lime, was acted upon. An attempt was then made to solve the question in an indirect way by digesting cement in very dilute hydrochloric acid, collecting and drying the residue, and testing its setting power, after mixing with water and lime-water respectively. The author considered it reasonable to suppose that the free lime would be attacked and dissolved more readily than the combined, more especially as calcium chloride is a good solvent for free lime. On extracting more or less lime from Portland cement in this way it was found that its hydraulic properties were correspondingly diminished. Cement partially deprived of lime set quicker and more completely however, when lime and other alkaline substances were added to it, though without chemically uniting with them. The author considers that the presence of quick-lime in cement serves to render the water alkaline, and so to expedite and complete the setting.—H. H. B. S.

### *Cement, To make Waterproof.* Dingler's Polyt. J. 307, [7], 168.

Concrete may be rendered impervious to water by scrubbing and scraping the surface thoroughly clean, and then coating it with a layer—3 to 4 mm. thick—of caoutchouc glue, spread on with a trowel. When dry, an outer protective coating of cement can be applied, smoothed over with a steel blade; and when the operations have been carefully performed, the concrete will be impermeable by water.—C. S.

### *Slag Brick in Germany.* F. Luermann. Eng. and Mining J. 1898, 65, [11], 314.

It is stated that bricks manufactured from granulated blast-furnace slag are preferable to those produced directly from fluid slag, for use as building material for dwelling-houses, since the former are porous, whilst the latter, being impermeable to air and steam, would cause the aqueous vapour exhaled by the occupants to condense on cold days, and thus render the rooms damp and unhealthy. Granulated slag or slag gravel for brick-making was first produced by E. Langen by passing the fluid slag through water; disintegration ensues, and the silica is converted into a soluble condition, in which it easily hardens and readily combines with caustic lime. The grains of the slag gravel thus produced are, when subjected to pressure, bound by the soluble silica, and when ground to a fine dust, the binding by the silica is sufficient for the production of good building bricks. Bricks made from ground slag, however, require a longer time for hardening than those made from granulated slag and lime. Good bricks may be produced from granulated slag mixed with dust from slag which has crumbled in the air, but the hardening process is rather slow. Bricks produced from granulated slag and about 10 per cent. of slaked lime, require 6—8 days for hardening. Granulated slag retains, in the course of preparation, from 19 to 33 per cent. of water, and its weight ranges between 1,100 and 1,500 lb. per cb. m. (35·3 cb. ft.); that of ground slag is sometimes as high as 2,900 lb. per cb. m. Ground slag is added to the mixture of granulated slag and lime when specially dense and strong bricks are desired. The weight of the slag brick varies, according to dryness and to the kind of slag used, between 260 and 280 bricks per ton. Slag brick is stated to be quite as strong as ordinary brick, whilst it may be heated to the temperature at which carbonate of lime begins to decompose without injury to its strength, since even if a part of the carbonate of lime be decomposed, carbonic acid is again absorbed at a lower temperature. On account of this latter property, slag brick appears to be particularly adapted for the construction of chimneys, for lining lime-kilns, and for boiler setting. Slag brick is about five times as permeable as ordinary brick, and although it does not absorb water so quickly, yet it regains its permeability more rapidly than the latter.

To determine whether slag brick (or other kinds of brick) has sufficiently dried and hardened to be safely used, and especially to test its resistance to frost, a small piece of brick, fastened to a string, is dipped into a cold, concentrated solution of sulphate of soda, and hung up in a room. The crystallising out of the sodium sulphate, on evaporation of the water, resembles in its action the formation of ice; non frost proof brick is destroyed, each crystal-needle carrying a little cap of the material from which the brick was produced.

The slag of furnaces making "Thomas" or "basic pig" is preferred to that from Bessemer works for the manufacture of bricks. The cost of a slag-brick plant in connection with a blast furnace is estimated at about 5,000 dols.; and for the production of 1,000 bricks, 6,000—7,000 lb. of granulated slag and 450—700 lb. of lime are necessary.
—A. S.

### *Limestone, Freshwater, Utilisation of.* R. Burghardt. Thonind. Zeit. 22, [11], 106—107.

In place of the ordinary method of drying this lime, which consists in moulding the freshly dug, wet mass (containing up to 64 per cent. of moisture) into blocks, and drying in the open air—a very slow process, as well as wasteful, from the number of blocks that fall to pieces in wet weather—the author recommends leaving the lime to dry in small heaps for a few days, and then transporting it (preferably for some distance, to facilitate further drying), by means of a reciprocating carrier, like the Kreiss ore carrier, to presses, where it is compressed into large blocks. These are cut by a wire cutter into bricks, which are cohesive enough to be stood on edge. In consequence of this treatment, the output per kiln is considerably increased.—C. S.

### *Brick Earth rich in Lime: Burning in Continuous Kilns.* Thonind. Zeit. 22, [12], 113—115.

The presence of finely divided fine-grained calcium carbonate in many brick earths, imparts to the clay certain characteristics which increase the difficulties in the way of producing good, durable bricks therefrom. In the first place the bricks must be fired at nearly the clinkering temperature—*i.e.*, one closely approaching fusing point, obviously a temperature difficult to regulate. A more serious difficulty, however, is the prevention of discolorations, to which these bricks are peculiarly liable, the absorption of sulphur trioxide, from the furnace gases, by the lime, resulting in the production of red streaks due to gypsum. A means of preventing this exists in the employment of a reducing fire at intervals during the later stages of burning—a measure automatically accomplished in intermittent firing every time fresh fuel is fed to the furnace. On the other hand, various methods proposed for effecting the same purpose in continuous circular kilns have proved unsatisfactory from divers causes. However, the problem appears to have been solved in the Günther continuous kiln, which is divided into sections by chequer-work partition walls, which can be closed by means of sand run in from above, thus isolating any sections at will from the hot air flue, made by leaving a hollow space at the bottom of the sections when the bricks forming the charge are placed in the kiln. In the isolated sections there then ensues a deficiency of air for the perfect combustion of the fuel, and thus a reducing atmosphere is produced. As an instance of the alteration caused by shutting off the hot-air supply, the gases in one chamber when in circuit contained 3·2 per cent. of $CO_2$ and 14·2 per cent. of O, but when isolated, the proportions became 12·4 and 2·8 per cent. respectively. The isolated sections lose very little of their internal heat, cooling down beginning only when the sand has been withdrawn from the partitions.—C. S.

## PATENTS.

### *Artificial Marble and the like: Impts. in the Art, Method, or Process of Treating Gypsum Rock for the Production of.* T. Parker, Erdington, Warwick. From G. W. Parker, of Detroit, U.S.A. Eng. Pat. 7798, March 25, 1897.

Native gypsum rock, as taken from the mine, is shaped, by sawing, planing, &c., into the configuration required for the finished articles. After dehydration by exposure for 12 hours to air heated to 330° F., the blocks are cooled in a closed compartment, in which a proportion of ammonia gas is mixed with the air, and are finally immersed in a warm solution of aluminium sulphate. Effects in imitation of fancy marbles, &c., can be produced by applying mineral colours to the dehydrated gypsum before immersion in the aluminium sulphate.—H. H. B. S.

### *Blast-Furnace Slag or Scoria, An Improved Treatment of, for the Manufacture therefrom of Hydraulic or similar Cement, or for other Purposes requiring the Use of Dry Granulated Slag.* T. C. Hutchinson, Saltburn-by-the-Sea, York. Eng. Pat 9030, April 9, 1897.

Blast-furnace slag or scoria is disintegrated in such a way that a slag sand is produced sufficiently free from moisture to render subsequent drying unnecessary. The slag is run through a trough and cooled by a stream of water, the excess of which drains away beneath the slag before it has time to become absorbed. The disintegrated slag accumulates as a spongy cellular mass, which is removed by revolving rakes or other mechanical means.—H. H. B. S.

## X.—METALLURGY.

### *Australian [Gold] Slimes Agitation Vat.* F. C. Pengilly. Chem. and Metallurgical Soc. of S. Africa, Dec. 18, 1897, 9.

The author briefly describes Deeble's gold extractor, wherein refractory pyritic ores can be successfully treated with cyanide without the preliminary roasting. The chief point in the extractor is that the agitators are raised from the cyanide vat when the settling and decanting take place, and lowered again for the fresh solution. This prevents the slime settling on them, and saves digging out. The vat has a sliding door, by which, after settlement, all the liquid can be decanted from the top down to the level of the slimes. When the extraction is finished, the agitators are lowered and the sluice-valve opened, when the slimes gravitate out on to the dump heaps. The gold is precipitated by charcoal placed in several rows of boxes.

The result of a 10-cwt. trial of refractory ore with treatment of three cyanide solutions, of 0·318, 0·218, and 0·210 strength, showed an extraction of 86·5 per cent., the amount of cyanide used equalling 2·5 lb. per ton. The whole treatment took 23 hours. Large quantities, containing 4 dwt. of gold per ton, are practically treated at Bendigo with an extraction of 80 per cent. Against the advantage of dispensing with the calcination must be considered the extra cost of grinding the pyrites to a pulp.—A. W.

### *Gold Ores, Stamp Milling of, in its Relation to Cyaniding.* E. H. Johnson. Chem. and Metallurgical Soc. of S. Africa, Dec. 18, 1897, 6.

After pointing out the disadvantages of a high discharge from the stamps for ore in which the gold exists in the pyrites, thereby producing almost impervious tailings, the author deals with the question of using heated water from the condenser for milling purposes. He quotes Mr. W. Skey, who reports that some clean-looking gold direct from the reef would not amalgamate, owing to sulphur being present on its surface. This gold became capable of amalgamation when roasted, unless copper were present to the extent of about 7 per cent. or perhaps less, and also when treated with cyanide, nitric or chromic acid, and acidified calcium chloride. Sulphate of iron, with air and water, decomposes certain sulphides with the production of sulphuretted hydrogen, which readily attacks the gold surface, especially when warm, and is the cause of the non-amalgamation. As ferrous sulphate is present in nearly all mine water, it is consequently advisable not to use heated water.

Then, in its relation to the use of cyanide, it has been found that partial roasting yielded worse and worse extracting results until the ore was dead-roasted, owing partly to increasing withdrawal of oxygen for reaction with the sulphides. As roasting is only practicable with rich and highly pyritic ores, it is therefore advisable to treat the ore in its natural state as far as possible, and to avoid any preparation of it which tends to contribute to the decomposition of the sulphides.—A. W.

### *Chlorination and Cyaniding.* C. C. Burger. Eng. and Mining J. 1897, 64, [23], 663.

The author protests against the assertion of Recknagel, in his article on "Cyaniding Sulphide Gold Ores" (this Journal, 1898, 50), that "unlike the chlorination process, cyaniding of roasted ores is always cheap." It is stated that, comparing the modern cyaniding and barrel chlorination processes, the latter costs 30 c. per ton more for chemicals, but 10 per cent. more gold is saved than with cyanide, and the cost of crushing is less than one-half. At some milling plants in the U.S.A., both processes are employed, the oxidised ores going to the cyanide department and the unoxidised to the chlorination section. It is also stated that distance from a railroad does not make chlorination excessively costly.—A. S.

### *Chlorination and Cyaniding.* R. Recknagel. Eng. and Mining J., 1898, 65, [11], 308.

The author adheres to his statement (see preceding abstract) with regard to the cheapness of cyaniding roasted gold ores, as compared with the chlorination method, more especially in the case of lower grade ore. He takes exception to Burger's statement that chlorination costs 30 c. more per ton for chemicals, but 10 per cent. more gold is saved than with cyanide, and the cost of crushing is less than one-half; and states that when, in both cases, roasted ore of the same fineness is used, only 2—3 per cent. better extraction is obtained by chlorination than by cyaniding. The author also considers that cyaniding is preferable to chlorination, other conditions being equal, on account of the simplicity of the requisite plant, and also from the fact that whilst a modern barrel chlorination plant is only suitable for the treatment of large quantities, a cyanide plant may be fitted up equally well for any quantity, large or small; also the difference in working expenses is never so high as with chlorination. Finally, it is contended that the necessity of roasting previous to cyaniding does not prevent the successful application of the latter process.—A. S.

### *Gold and Silver, The Melting Points of.* D. Berthelot. Comptes Rend. 1898, 126, 473—476.

A gold or silver wire a few millimetres long completes a circuit formed by two platinum wires, and is slowly heated till the current is interrupted by the fusion of the wire. A platinum-platiniridium thermo-couple closely adjacent to the gold or silver wire measures the temperatures. Six observations on silver, varying from 959°·2 to 966°·2 C., gave as the average 962° C. for the melting-point of silver; while the average of five observations, lying between 1,062°·1 and 1,066°·7, gave 1,064° C. for that of gold. The author collects the figures given by former observers, lying between 954° and 971° for the melting point of silver, but varying between much wider limits for that of gold. He points out, however, that recent observers, working by differential methods, all place the melting point of gold at 100°—105° above that of silver, which affords confirmation of the accuracy of the figures just given.—J. T. D.

### *Nickel Steels: Variations of Volume in Irreversible Alloys.* C. E. Guillaume. Comptes Rend. 126, [10], 738—740.

A bar of the steel to be examined was heated to redness, clamped to a similar bar of German silver, plunged into a bath of oil at 200° C., and allowed to cool very slowly, while the difference in the lengths of the two bars was carefully determined from time to time. With steel containing 15 per cent. of nickel, contraction takes place down to 130°, when the bar begins to expand, and continues to do so to −60°: the experiments were not continued below this temperature, but the curve representing the variation of length of the bar, showed that the transformation was then nearly complete. If, below 130°, such a bar be heated for a few degrees, it expands like an ordinary metal, and on again cooling, contracts correspondingly; but as the temperature at which the heating began is reached and passed, the bar again expands, and its variations of length follow the curve, which was interrupted when the heating began. Sometimes (in particular with a steel containing 24 per cent. of nickel) a state resembling superfusion occurs, and the contraction during the cooling just referred to, continues for some degrees below the point at which the heating began, and then there is a sudden elongation, the length of the bar becoming what it would have been at this temperature had the curve of elongation been followed all along.—J. T. D.

### *Nickel Steel, The Magnetic Properties of.* E. Dumont. Comptes Rend. 126, [10], 741—744.

The author has determined the magnetic permeability for fields lying between 14 and 50 C.G.S. units, and for temperatures between −78° and 250° C., of reversible nickel steels, containing from 27 to 44 per cent. of nickel. The variations of permeability with temperature in the maximum field are continuous, and the curves representing them are roughly parallel to each other, so that at equal distances (of temperature) from the point of total loss of magnetism, all the alloys have the same permeability. At a temperature of 20°, all the alloys show similar changes of permeability, with variations in the magnetising field, rising slowly as the field is strengthened from 14 to 25 units, then rapidly increasing to a maximum in a field of 35 units, and slowly diminishing as the strength of the field is further increased. Under the same circumstances of temperature and strength of field, the permeability increases with the content of nickel.
—J. T. D.

### *Gold Bullion, Assaying of.* C. Whitehead and T. Ulke. Eng. and Mining J. 1898, 65, [7], 189.

*See under* XXIII., *page* 379.

*Phosphorus, Rapid Estimation of.* O. Herting. Stahl u. Eisen, 1897, 1005; Chem. Centr. 1898, 1, [3], 219.

*See under* XXIII., *page* 377.

## PATENTS.

*Lead Ores, Treatment of; Impts. in.* A. Pigott, St. Leonards, Sussex. Eng. Pat. 142, Jan 3, 1898.

This patent relates to modifications of Eng. Pat. 2883 of 1893 (this Journal, 1894, **13**, 401). Galena (alone or mixed with lead) is fused, and is then volatilised by passing heated air or neutral or reducing gases through it. Air or steam is then injected into the galena vapour, whereby the "oxysulphides" $Pb_3S_2O_8$ and $Pb_3S_2O_9$ are formed. Any silver in the lead is left behind. If air be used to vaporise the galena, some lead is reduced, which is to be resulphurised in order to separate it from the silver. The "oxysulphides" are used to reduce galena, according to the equations—

$$2PbS + Pb_3S_2O_8 = 5Pb + 4SO_2.$$
$$5PbS + 2Pb_3S_2O_9 = 11Pb + 9SO_2.$$

By using a slight excess of the "oxysulphide," a very soft lead and a little litharge are produced. Metallic lead and $SO_2$ are also to be produced by volatilising the galena with the aid of non-oxidising gas, and passing the vapour through molten litharge.—W. G. M.

*Metallurgical Furnaces and the like; Impts. in or relating to the Utilisation of Waste Products from.* A. J. Boult, London. From H. L. Hartenstein, Bellaire, Ohio, and G. A. Weber, Chicago, Ill., U.S.A. Eng. Pat. 224, Jan. 4, 1898.

Molten slag is mixed with very finely divided carbon (coke dust), and subjected to the action of a powerful electric current, whereby the carbon and the oxides in the slag react to form carbides of calcium, aluminium, and silicon. The mixture may be made cold, and reducing gas may be injected whilst it is being heated electrically. Or the carbon, suspended in the waste gases from the blast furnace, may be injected into the molten slag. Three parts of slag should be incorporated with one part of carbon. The operation is conducted in a rotatory converter, mounted on hollow trunnions, and divided into two compartments, so arranged that by the rotation of the vessel, the fluid charge may be run from one to the other. One of these compartments is provided with tuyères communicating with the trunnions; the other has electrodes, through which the current is introduced. The blast-pipe outside the trunnions is fitted with a hopper and a rotating feed for introducing the coke-powder into the blast. One trunnion may be used with reducing gas and one with air, the gases being mixed at the tuyères. The molten slag is introduced into, and the fluid carbides are poured from the converter through the nose.—W. G. M.

*Ores, Impts. in the Treatment of.* D. M. Hyman, Denver, U.S.A. Eng. Pat. 17,542, July 26, 1897.

The process is especially applicable to zinc-lead ores. The ore is crushed to the size of the smallest crystal of the constituent which has the highest specific gravity. The actual degree of fineness is therefore decided after careful examination of the ore. A mechanical dressing or separation is then effected. In carrying this out the ore is dumped into bins at a considerable elevation, thence it passes through a crusher and over grizzlies to crushing rolls capable of crushing it to from ½ to ⅛ in. in diameter. It is next raised by an elevator to a bin large enough to hold one day's charge. It is then fed (still dry) to (three) Chilian mills, where it is mixed with water and crushed to the required size, after which it escapes through adjustable screens to classifiers, where, meeting an ascending current of water, it is separated into sizes. Each class of ore is fed on to a separate concentrating table, by which the constituents are separated; and like constituents from all the tables, are ultimately collected in the same receptacle. The slimes and dirty water from the classifier pass through a spitzkasten (settler, or separating apparatus), and the dirty water from the whole mill is run through a second spitzkasten, and may then be run to waste or may undergo further treatment, according to circumstances. The separated minerals are roughly dried and conveyed away in cars.
—W. G. M.

*Iron and Steel, and Alloys of same; Impts. in the Manufacture of, direct from the Ore.* H. Niewerth, Berlin. Eng. Pat. 3926, Feb. 13, 1897.

The ore (or mixture of ores suitable for forming the required alloy) is crushed to powder and mixed into a paste with tar and coal-dust and water. The paste is then moulded into the shape of the object to be made, tubes or rectilinear objects being forced out of nozzles under pressure, and the pieces are then heated in muffles or ovens until the metal is reduced and the particles have become consolidated by welding themselves together. The grade (carbon-percentage) of the resulting pieces depends upon the amount of coal-dust and the nature of the gas used in firing. For common goods the coal-dust is omitted.—W. G. M.

*Ores [Gold], Treatment of, with Solvents; Impts. in or relating to.* W. A. Koneman and W. H. Hartley, both of London. Eng. Pat. 4857, Feb. 23, 1897.

This process is intended chiefly for the treatment of slimes, or of ore containing a large proportion of slimes. A cylindrical plate-iron vessel, of from 5 to 40 tons capacity, is supported on masonry walls and provided with a removable top and bottom, which are supported by bolts held in place by collars. The bottom rests on a carrier, the height of which may be adjusted by a screw-jack, and the carrier is mounted on wheels running upon a track which passes between the walls and under the tank. The top may be raised by means of jacks, and may then be run similarly along a lateral track. The bottom is provided with movable connections through which air or water may be forced at will, under control by means of suitable cocks. At the top is a pressure gauge and an escape valve. Within is a wide float of cane or bass-wood, with a flexible tube, so attached that one end projects just below the level of the liquid upon which the float is resting, whilst the other is connected with a pipe passing through the wall of the vessel and provided with a cock outside. The ore and solvent, after preliminary agitation and contact outside, are introduced into this vessel; air is then pumped in under a pressure of about 100 lb. per sq. in. until the solid particles are caked into a compact mass at the bottom; the solution is next expelled through the flexible tube, and wash water is forced through the mass until the cake is reduced to a sludge, when the water is shut off and air is once more let in under pressure. In this way the gold solution is removed completely to all intents and purposes. After the washings have been taken off, the bottom of the vessel is removed, another trolley is brought up in place of the first, and air is forced into the top of the vessel, so that the cake is pressed bodily out into the trolley and removed. The apparatus is then ready for use again.—W. G. M.

*Iron and Steel, Impts. in Treating.* T. Korovine, N. Klatschko, and J. Berthenson, Moscow. Eng. Pat. 5796, March 4, 1897.

Soft steel is faced with hard steel by shaping the former, heating it to redness, plunging it into a warm mixture of 4 parts of rosin, 1 of turpentine, and 1 of hemp-seed oil (heated until the rosin is just melted), and then applying the hard steel, and heating the two in contact up to the welding point, and consolidating the weld by pressure. The material will thus have been overheated, and it must therefore be plunged again into the warm rosin mixture, and kept there for at least five minutes. After the surface has been worked as required, the article is heated to redness, and plunged into the same bath diluted with enough naphtha or turpentine to make it liquid at ordinary temperatures. In this way great hardness is imparted. Worn parts of machinery may thus be restored with hard faces.
—W. G. M.

*Metals, Impts. in Liquid Fuel Furnaces for Heating or Smelting or Refining.* J. Davies, Wednesbury, and A. Sanders, Birmingham. Eng. Pat. 6489, March 12, 1897.

Apparently the oil is to be heated and then pumped in the form of fine spray into the vaporiser, whence it is to be blown into the furnace through tuyères by means of a blast of hot air or superheated steam. The claims are—"First: The heating of air, steam or gas, and oil before entering tuyères, by waste heat or other suitable means. Second: Use of oil for smelting or refining metals."

—H. B.

*Furnaces, Muffle-, Impts. in.* E. Edwards, London. From C. Schaaf, Baden, Germany. Eng. Pat. 9522, April 14, 1897.

Two muffles, each with a door at either end, are set side by side, but with a thick partition wall dividing the central portions. Lateral communication is provided between the corresponding ends of the muffles. Beneath the centre of one muffle is a grate, the products of combustion from which pass first through flues surrounding this portion of the muffle, thence around and under one end only of the same muffle, and finally to a descending flue provided with a damper. Rising partitions or dampers are provided on either side of the hot section of this muffle, and a similar partition closes the channel communication between the hotter end thereof and the adjoining muffle. In the centre of the latter is a door through which are introduced the trays containing the pottery to be fired. These trays are then gradually moved towards the partition separating the two muffles, the heat being controlled by means of the partition. They are next passed into the firing muffle and by degrees into the hottest part thereof, whence, being sufficiently burnt, they are shifted, first into the cooler section of the heated muffle, and then through the back communication to the one into which they were first admitted. The trays are manipulated through the end doors, and also through openings provided on the long side of the cool muffle, opposite the connecting passages.

—W. G. M.

*Furnaces, Regenerative, Suitable for Melting Steel in Crucibles or Pots; Impts. in.* B. Dawson, Malvern Link, and S. Pope and S. J. Robinson, both of Sheffield. Eng. Pat. 9818, April 17, 1897.

A row of crucible furnaces is served by one set of regenerators, situated at some distance from them, so that the workman may not be exposed unnecessarily to heat. Each furnace, with its pair of pots, has four horizontal flues connected respectively with the two gas and the two air regenerators. The flues are provided with dampers, so that the temperature of every furnace is under perfect control, independently of the others; and the direction as well as the quantity of gases supplied may be regulated at will in each furnace.—W. G. M.

# XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## (*A.*)—ELECTRO-CHEMISTRY.

### PATENTS.

*Electric Batteries [Fused Electrolytes, &c.], Impts. in and connected with.* J. L. Dobell, Harlesden, Middlesex. Eng. Pat. 25,036, Nov. 7, 1896.

Claim: "An electric battery, comprising a cell, a porous pot [see abstracts of later patents following] suspended within the cell, a quantity of nitrate of a suitable metal within the cell, a quantity of pure lead within the porous pot, a negative electrode within or acting upon the nitrate, a positive electrode immersed in the lead, a furnace for keeping the nitrate and lead in a molten state, and a jet of air or other oxidising agent continuously injected into the fused nitrate, &c."—J. C. R.

*Electrical Insulation Conduits, Impts. in, and in the Method of and Apparatus for Making same.* P. M. Justice, London. From The Lithosite Manufacturing Company, St. Louis, U.S.A. Eng. Pat. 1379, Jan. 18, 1898.

"This invention relates to a new and useful improvement in methods of, and apparatus for, making electrical insulation conduits, the object being to make a conduit which will be light, strong, and an effective insulator."

Ten claims. Claim 1. . . . "Method of making insulation conduits, the same consisting in treating a fibrous material (preferably in the form of burlap or other fibrous material) with a cementitious substance, magnesium chloride, 30° B., one pint, oxalic acid (to neutralise calcium chloride), magnesium oxide, $1\frac{1}{3}$ lb., and, to hasten drying, 5 per cent. of sal-ammoniac with the oxide, &c. As fillers, it is proposed to use in 'proportions of about one to one of the mass, composed of magnesium chloride and oxide of magnesium,' the following: 'graphite, powdered talc, slate dust, pyrites, powdered mica or other suitable material,' winding the fibrous material so treated on a mandrel, and then removing the mandrel," &c.

It is also proposed to "immerse the fibrous material, after being so treated, in a bath of chloride of zinc, to close the pores thereof, and then removing said conduit from the mandrel."—J. C. R.

*Electric Batteries [Fused Electrolytes, &c.], Impts. in or connected with.* J. L. Dobell, Harlesden, Middlesex. Eng. Pat. 2272, Jan. 28, 1897.

"Relates to that class of electric batteries in which electrical energy is obtained by the consumption or combustion of carbon or carbonaceous matter."

The electrolyte is fused litharge, &c. "So long as the battery is fed with fuel, that is to say, with the carbonaceous material, and with a continuous supply of air or other oxidising agent, a constant current of electricity will be maintained at the expense of such fuel."

Claim 1. "An electric battery, comprising a cell or containing vessel, a quantity of lead within the cell or containing vessel, fed with solid carbonaceous material, a porous pot suspended within the cell or containing vessel and descending into the lead, a quantity of suitable metallic oxide within the porous pot, supplied with air or other suitable oxidising agent, a gas or other fire for keeping the lead and oxides in a fused condition; means for feeding the carbonaceous material to the fused lead, and means for injecting air or other oxidising agent into the fused oxide, substantially as herein shown and described," &c.

—J. C. R.

*Electric Batteries, Impts. in or connected with.* J. Laskey, Dobell, Harlesden, Middlesex. Eng. Pat. 4442, Feb. 18, 1897.

The object is to obtain a porous pot which will resist the action of fused oxides, "litharge, caustic soda, caustic potash, or like oxides." Claim 1. "A porous pot or other article formed of calcined magnesite, reduced to a fine state of division, moistened with a solution of boric acid and water, or spirit and water, or with a quantity of boric acid dissolved in spirit, then made into a stiff paste or dough, and shaped to the required form of the article to be produced, and afterwards dried, and, if desired, baked or fired," &c.—J. C. R.

*Galvanic Batteries [Zinc-Carbon], Impts. in Primary.* S. F. Walker, Cardiff. Eng. Pat. 5383, March 1, 1897.

1. "The use of a solution, consisting of chromic and hydrochloric acids, in a zinc-carbon galvanic cell, in proportions according to the work to be done by the battery." 2. . . . . "chromic acid, hydrochloric acid, and sodium sulphate. . . ." 3. . . . "carbon electrode, the pores of which have been filled up with sulphur or other material . . . to resist chromic acid," &c. 4. . . "a carbon cylinder perforated with holes . . ." Other patents referred to, 24,443 of 1895, and 12,028 of 1897.—J. C. R.

*Secondary Batteries, Impts. in the Active Material of.* A. Sebanschieff, West Dulwich, Surrey. Eng. Pat. 5826, March 5, 1897.

Claim 1. "A process for the production of active material compound for battery elements consisting in intimately mixing pure carbon with organic hydrocarbon acids, sulphuric acid and lead material, and then causing such a decomposition of the organic acids, by means of an electric current, that a finely porous gas-absorbing conducting substance of peroxide of lead and carbon, is produced . . ."

2. "A process" &c., "consisting in intimately mixing pure carbon ["charcoal"] with tartaric acid, formic acid, sulphuric acid and lead material [litharge, &c.] in or about the proportion stated [in grms. 60 to 80, 20, 10, 100 to 180, 700 to 1,000, respectively, care being exercised in mixing] and decomposing the acids by electric current," &c.—J. C. R.

*Sodium Chloride or other Salts, Impts. in Apparatus for Electrolysing.* F. Taylor, A. Cooke, and B. W D. Montgomery, Belfast. Eng. Pat. 6929, March 17, 1897.

The anodes are formed of hard, round carbon, and the cathodes of sheet iron, which are surrounded by strips of glass in louvre form, opening from the cathode outward. These inclined strips of glass facilitate the descent of caustic soda, and the ascent of the gaseous products, which are removed, by means of perforated pipes dipping into each anode compartment, to a hollow agitator spindle and arms rotated at a medium speed in a solution of lime or magnesia, which absorbs the chlorine.—G. H. R.

*Secondary Batteries or Accumulators [Lead Plates, &c.], Impts. in and relating to, and the Manufacture thereof.* A. J. Marquand, Cardiff. Eng. Pat. 7027, March 17, 1897.

Claim 1. "The improved method of producing plates for secondary or storage batteries, consisting of the protection of certain parts of lead plates or foundations against the action of sulphur, the treatment of such partly protected lead plates or foundations in a closed box, with sulphur at or near the melting point of lead, and the subsequent reduction or peroxidation, by electrolytic means, of the prepared surfaces, . ." The following composition is found to be a suitable enamel for the purpose: calcium fluoride, 4·9 per cent.; felspar, 15·6; sodium silicate, 25·3; silicon dioxide, 1·9; barium sulphate, 19·3; fused by a blow-pipe or oxyhydrogen flame without melting the metallic lead underneath.

Claim 1. "An improved electrolyte for use with lead plates in secondary or storage batteries, composed of dilute sulphuric acid containing other sulphur compounds, such as the thionic acids or salts of the same produced by electrolytically reducing sulphide of lead in dilute sulphuric acid, &c. . . ."—J. C. R.

*Electrodes [Differing], in Electrolytic Processes, Impts. in or relating to the Application of.* O. C. and H. H. Strecker, Cologne, Germany. Eng. Pat. 26,919, Nov. 17, 1897.

The object of the present invention is the reduction of the difference of potential necessary in electrolytic processes, in which the anode enters into the desired combination. The results are given in a series of experiments with aluminium, copper, zinc, cadmium, tin, lead, antimony, bismuth, cobalt, iron, and nickel, using 15 per cent. solutions of sodium chlorate, and 26 per cent. of sodium chloride, and their utilisation is claimed for in the choice of electrodes which are different one from another in their metal, for the electrolytic production of those oxides or salts in which the anode metal enters into the combination to be formed.
—G. H. R.

*Insoluble or Hardly Soluble Salts from Metallic Anodes by Electrolysis, or Aqueous Solutions containing Two Salts; Improved Process for.* O. C. and H. H. Strecker, Cologne, Germany. Eng. Pat. 26,921, Nov. 17, 1897.

This is a process for the preparation of salts, which are insoluble or hardly soluble in water or salt solutions, by means of electrolysis from metallic anodes, characterised by the mixing of the solutions of the two salts used as the electrolyte, in such a manner that the electrolyte contains the dissolving salt in the proportion of its greatest conductivity, and the other or precipitating salt, in the proportion of the electro-chemical equivalent of the current density employed. The principle is illustrated by a description of the manufacture of white lead by this method, using lead anodes and an electrolyte containing a 7 per cent. solution of chlorate of sodium, and 0·011 per cent. of carbonate of sodium, the current density employed being 0·5 ampère per sq. cm. of anode surface.—G. H. R.

*Protoxides and Oxides or Hydroprotoxides and Hydroxides, Impts. in or relating to the Production of, from Metallic Anodes by Electrolysis of Alkaline Salt Solutions.* O. C. and H. H. Strecker, Cologne, Germany, Eng. Pat. 26,923, Nov. 17, 1893.

This invention relates to a process for the preparation of protoxides, oxides, or of hydroprotoxides and hydroxides, from the metallic anodes employed, by electrolysis of salts of the alkalis, characterised by the utilisation of the division of the salt employed into its acid ion and alkali hydrate, for the purpose of dissolving and reprecipitating the anode metal employed. It is illustrated by examples of the use as anodes, of zinc, cadmium, tin, copper, iron, nickel, cobalt, antimony, lead, bismuth, and silver in solutions of sodium chloride, sodium chlorate, and sodium sulphate at a concentration showing best conductivity.—G. H. R.

## (*B.*)—ELECTRO-METALLURGY.

### PATENT.

*Electro-plating Apparatus, Certain Impts. in. [Revolving Barrel.]* J. Guise, Redditch. Eng. Pat. 6962, March 17, 1897.

The apparatus consists of a watertight barrel, having one end removable, against which the anode is fixed, whilst the cathode is formed by a copper or other suitable wire wound spirally or in zig-zag fashion on the inside of the barrel, and connected to a copper band encircling it, and against which presses a brush connected to the negative terminal of the dynamo. The barrel is provided with a central divided axle connected to the positive terminal, and contains the electrolyte and the articles to be plated, which come into contact with the cathode wires as the barrel revolves.
—G. H. R.

# XII.—FATS, OILS, AND SOAP.

*Illipé-Fat in the Manufacture of Stearine.* A. Z. Seifensieder Zeit. 25, [1]. 13–14.

This fat is well adapted for the saponification method of stearine manufacture. In the autoclave it yields about $9\frac{1}{2}$ per cent. of fairly pale glycerin of 28° B. The fatty acids are greenish-white and crystallise in prisms, but the stearine obtained by cold- and subsequently warm-pressing is deficient in whiteness and transparency. To overcome this defect, fatty acids of low melting point should be added in sufficient quantity to ensure the expression of a considerable amount of olein under cold pressure, the colouring matter being thereby removed. For this purpose, good bone-fat is said to be the most suitable, although attended with the difficulty that the fatty acids, which crystallise well by themselves, do so no longer when mixed. This peculiarity, which often causes trouble in stearine works, is probably due to the ratio between the palmitic and stearic acids present; it has not as yet, however, been scientifically investigated.

By the distillation method, also, Illipé fat can be worked up with ease, and yields pure white fatty acids which crystallise well, and are therefore suitable for direct pressing.—C. S.

*Seal Fat, Liquid Fatty Acids of.* E. Ljubarsky. Journ. prakt. Chem. 1898, 57, [1], 19—27.

By treating the lead salts of the fatty acids of Caspian seal oil with ether, the author obtained 17 per cent. of a saturated fatty acid, which, from its melting point, appeared to be palmitic acid, and 83 per cent. of liquid fatty acids, which were found to consist of oleic acid, $C_{18}H_{34}O_2$, and physetoleic acid, $C_{16}H_{30}O_2$, the latter being the more abundant constituent, and yielding dihydroxypalmitic acid on oxidation with permanganate. In connection with the examination of the oxidation products of the liquid fatty acids, it is stated that dihydroxypalmitic and dihydroxystearic acids form a molecular compound with physical properties quite distinct from those of the individual components. Thus, it melts at 124°—125° C., solidifies at 120° C., and is soluble in hot water, whereas dihydroxystearic acid is nearly insoluble. Attempts to obtain margaric acid (an acid with 17 carbon atoms) by the action of phosphorus iodide in the presence of water on this body, apparently dihydroxymargaric acid, and reduction of the fatty iodide with nascent hydrogen, confirmed the author's views, as the reduction product was found to consist of a mixture of palmitic and stearic acids.—C. A. M.

*Elaïdic Acid, Action of Sulphuric Acid on.* A. Tscherbakoff and A. Saytzeff. Journ. prakt. Chem. 1898, **57**, [1], 27—35.

Elaïdic acid (100 grms.) was treated with 35 grms. of sulphuric acid (65·5° B.) at a temperature below 50° C. The product, after standing, was diluted with twice its volume of water, and the oily layer separated and treated with successive portions of boiling water until the aqueous layer beneath became clear. The oily layer then became very viscid on cooling, and contained masses of crystals, which showed that anhydrides were present in large quantity. It was warmed on the water-bath for several days with an excess of alcoholic potash, the soap dissolved in water, and the fatty acids liberated and crystallised from alcohol. On saponifying the mother-liquor and again crystallising the liberated acids from alcohol, a further crop of crystals was obtained, which showed that anhydrides were still present after the first treatment with alkali. The two deposits were united and purified by recrystallisation from alcohol (hexagonal plates) and ether (crystalline powder). They were identified as hydroxystearic acid ($C_{18}H_{36}O_3$). Their melting point (82°—85°) and solidifying point (66°—62°) and their solubility in alcohol and ether were practically identical with the corresponding figures for the hydroxystearic acid obtained from oleic acid; and, like that acid, they decomposed when distilled under reduced pressure, with the formation of water and acids of the oleic series. The authors conclude that the oxystearic acid obtained from elaidic acid by the action of sulphuric acid is identical with that yielded by oleic acid.—C. A. M.

*Fats, Rancidity of.* A. Scala. Staz. sperim. agric. ital. **30**, 613—630; Chem. Centr. 1898, **1**, [7], 439.

The author submitted three fats to the action of light and air, and found that the largest increase in weight took place in the case of the fat containing the largest quantity of fatty anhydride, and the least increase in the one containing the smallest amount. Stearic and palmitic acids in pure preparations were found to undergo no alteration in weight after 4—6 months' action of light and air; some fatty acids, under these conditions, increased considerably in weight. The author therefore concludes that the development of rancidity in fats proceeds from the oxidation of the fatty acids. In order to identify the substances formed during the change, the author examined a large quantity of very rancid olive oil, and isolated: œnanthylic aldehyde, which gives to rancid fats their taste and odour, formic, acetic, butyric, and œnanthylic acids; and azelaic and sebacic acids. The same compounds were isolated from a rancid fatty acid, together with another compound which is designated dihydroxystearic acid.
—A. S.

*Drying Oils, Varnishes, and Dégras Oils; Testing.* A. Gawalowski. Chem. Rev. Fett- u. Harz-Ind. **5**, [1], 9.

*See under* XXIII., *page* 383.

*Bone-Fat, Technical Analysis of.* A. A. Shukoff and P. J. Schestakoff. Chem. Rev. Fett- u. Harz-Ind. **5**, [1], 5—1; [2], 21.

*See under* XXIII., *page* 383.

*Spermaceti, Examination of.* L. F. Kebler. Rev. intern. falsific. **10**, 208; Chem. Centr. 1898, **1**, [3], 223.

*See under* XXIII., *page* 383.

*Wax, Examination of.* G. Buchner. Zeits. für öffentl. Chem. **3**, 570; Chem. Centr. 1898, **1**, [2], 151.

*See under* XXIII., *page* 383.

*Edible Fats, Delicate Test for the Detection of a Yellow Azo Dyestuff used for the Artificial Colouring of.* J. F. Geisler. J. Amer. Chem. Soc. 1898, **20**, [2], 110.

*See under* XXIII., *page* 380.

### PATENT.

*Saponification of Fatty Matters, Impts. in and connected with the, and in the Manufacture of Glycerin.* E. G. Scott, Liverpool. Eng. Pat. 8796, April 6, 1897.

When fats are saponified with lime, the insoluble soap retains much of the glycerin within its cavities; and when it is decomposed with sulphuric acid, the resulting sulphate of lime renders the concentration of the aqueous liquors very troublesome. To remove the dilute glycerin solution from lime-soap obtained in lime saponification, the inventor grinds the soap in a roller mill fitted with plain or toothed rollers, or cuts it up in a cutting machine, and afterwards places it in a vacuum filter or centrifugal machine, and washes it, if necessary, with water, in order to recover the last traces of glycerin.—F. H. L.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (*A.*)—PIGMENTS, PAINTS.

*"Blanc Fixe" and Barium Chloride, Manufacture of.* K. W. Jurisch. Zeits. f. angew. Chem. 1898, [9], 198—199.

In 1882 these substances were prepared in a factory in the north of France by dissolving *witherite* in hydrochloric acid, freeing the solution as completely as possible from iron, and evaporating for the crystallisation of barium chloride, or adding sulphuric acid for the manufacture of blanc fixe. The analysis of three samples of the *witherite* showed that it contained from 77·61 to 88·7 per cent. of barium carbonate, 0·68 to 3·91 per cent. of calcium carbonate, 0·25 to 0·95 per cent. of water, and 0·012 to 8·41 per cent. of alumina and silica: whilst one sample contained 11·27 per cent. of insoluble matter not included in the above. The crystalline barium chloride obtained, had the following percentage composition:—Barium chloride, 81·58; calcium chloride, 1·90; water, 16·22; whilst the "Blanc Fixe" paste as received from the filter-press contained: barium sulphate, 80·25; water, 19·75 per. cent., with a trace of calcium sulphate. It is stated that attempts are again being made to substitute heavy spar for the more costly *witherite*.—C. A. M.

*Oil Paints, Testing.* H. Loesner. Chem. Rev. Fett- u. Harz-Ind. **4**, [24], 329—330.

To determine the permeability of paints by water and gases, the author proposes to apply the following tests:—Small strips of sheet iron (55 × 100 mm.) are carefully brightened on one side with sand- or emery-paper, and then thoroughly coated with two thin layers of the paint under examination, being left to dry for four days after each application. They are then laid face downwards over the apertures of a constant-level water-bath, at a height of 50 mm. above the water, and supported on strips of wood at each end to prevent abrasion. After 15 hours' exposure to the ascending steam, the paint is dried at a maximum temperature of 100° C., and removed by the aid of aniline and a soft brush, the under surface of the metal being examined for rust, which should not be present if the paint be a good one.

Resistance to acids is also tested in a similar manner, except that concentrated (33 per cent.) hydrochloric acid is employed instead of water, and that the protective incrustation formed on the surface of the paint must be rinsed off every hour, the temperature of the experiment being in this case 20° C., and the duration of exposure 12 hours.

The same methods may also be applied to fats, varnishes, &c. employed for protecting iron from rusting.

Considerable difference in the penetrability of these media is observed, some samples of vaseline standing the steam test for 1—2 hours, whilst others allow rust to form in half an hour. Fats may be considered satisfactory if they withstand 4—5 hours' steaming, whilst 12 hours is the minimum for a good oil paint.—C. S.

## PATENTS.

*White Lead, Impts. in Making.* J. S. MacArthur, Glasgow. Eng. Pat. 9775, April 17, 1897.

In this specification an improvement on the process for making white lead detailed in Eng. Pat. 9655, 1894 (this Journal, 1895, 587), is described. The solution of lead tartrate is mixed with a slight excess of ammonia before being treated with carbonic acid, which allows the precipitation to be conducted at about 82° C. (instead of the boiling point), and reduces the amount of tartrate present in the finished pigment. The current of gas should be stopped immediately or just before the liquor is saturated, as too much of it interferes with the solution of fresh quantities of lead oxide.—F. H. L.

*Carbonate or Sulphate of Lead, Impts. in the Manufacture of.* A. H. Eyles, H. S. Rapelye, and A. Applegate, Mount Vernon, New York, U.S.A. Eng. Pat. 30,242, Dec. 21, 1897.

Metallic lead is melted in a crucible and raised to a temperature just below its point of volatilisation, then sprayed by means of superheated steam or air, into a hot chamber which has a layer of weak nitric or acetic acid about 6 or 8 ins. deep, at the bottom. In this manner a mixture of finely-divided metal and oxides is produced, the latter varying from $Pb_2O$ to $PbO_2$. The acid is neutralised with ammonia, the solid with five times its volume of hot water placed in a revolving drum, such as that described in Eng. Pat. 19,030. 1897 (this Journal, 1897, 924), and treated with a current of hot air. The metal soon oxidises, the peroxide loses oxygen, and, finally, the whole mass becomes yellow and homogeneous, and consists entirely of PbO. It is then run into another similar vessel and exposed to the action of carbonic acid; or, if preferred, it may be agitated with sulphuric acid to convert it into lead sulphate.—F. H. L.

## (*B.*)—RESINS, VARNISHES.

*Chemistry of Xanthorrhœa Resins.* Chem. and Druggist, March 26, 1898, 495.

W. Finselbach writes *apropos* of the note on this subject (this Journal, 1898, 163), to the following effect:—

Tschirch and Hildebrandt have lately published on the same subject, and Karl Dieterich (Helfenberg) is working now on the acaroid resins, after having scientifically investigated (vide *Helfenberger's Annalen*, 1896) "resins and gum resins used in therapeutics." On November 9 last, he showed to the German Pharmaceutical Association samples of different acaroid resins, pointing out the inconvenience produced because the correct species of *Xanthorrhœa* furnishing a certain resin is hardly ever mentioned. The same applies to our kinos and eucalyptus oil. Dieterich considers that it is mainly *X. quadrangularis* which produces the gum acaroids of commerce. Lately this resin has been used as varnish for metal instead of shellac. Potassium and sodium soaps of our *X.* resins are used instead of colophonium, for sizing papers in the United States of America. There cannot be any doubt that Australian vegetable raw products would have found before now larger use in chemistry and industry if unfortunately the products of different species were not indiscriminately mixed together, and so brought into the market.

*Resins, Characterisation of some Rare.* K. Dieterich. Chem. Rev. Fett- u. Harz-Ind. 4, [24], 327—329.

*Crude Chicle Gum* from Achras Sapota—used in America for the preparation of "chewing gum" and as a cementing material—contains 75 per cent. of resin, 10 per cent. of gum, 9 per cent. of calcium oxalate with 5 per cent. of sugar and inorganic salts. When purified—for which purpose carbon bisulphide is the best solvent to employ—it is plastic, can be vulcanised, and is similar in general properties to gutta-percha.

*Guaiacum in lacrymis* gives, when purified, an acid number of 72·8—75·6 (for the author's method, see this Journal, 1897, 829, 830); and as the acid number of ordinary commercial qualities of guaiacum resin ranges from 89·6 to 92·5, they do not conform to the normal standard of purity.

*Socotra Dragon's Blood.*—The pure red resin obtained from this grade is a mixture of several resins. The crude product greatly resembles palm dragon's blood in melting point, the absence of free acids, and in the ester number and saponification value, but differs therefrom in solubility and in being destitute of the characteristic draco-alban.

*Bisabol Myrrh.*—To avoid the errors of previous workers whose experiments were conducted with alcoholic extracts and not with the natural drug, the author devised the following methods of examination:—

Acid Number.—30 c.c. of water are poured over 1 grm. of the finely-ground and thoroughly representative average sample, and warmed for ½ hour under a reflux condenser, being afterwards boiled for an equal time with 50 c.c. of strong alcohol, and titrated, when cold, with ½ N alcoholic potash and phenolphthalein, this strong alkali facilitating the detection of the end reaction.

Saponification Value.—Similar quantities of myrrh and water are mixed with 30 c.c. of ½ N alcoholic potash after half an hour's standing, and are then boiled for another ½ hour under a reflux condenser, cooled, diluted with alcohol and titrated back. The *ester number* is ascertained by subtraction as usual.

Solubility in alcohol is determined by extraction in a Soxhlet apparatus.

By these methods the following average constants were obtained:—

| — | Acid Number. | Saponification Value. | Ester Number. | Solubility in Alcohol. |
|---|---|---|---|---|
| | | | | Per Cent. |
| Bisabol myrrh . | 20·06 | 145·6 | 125·54 | 50 |
| Herabol myrrh | 25·48 | 226·6 | 201·12 | 20 |

*Xanthorrhœa Resin.*—From authentic Australian samples received by the author, it would appear that *Xanthorrhœa quadrangularis* (instead of *Xanthorrhœa australis*) is the chief source of the red Acaroid resin. —C. S.

*Driers and Varnishes.* M. Weger. Chem. Rev. Fett- u. Harz-Ind. 5, [1], 1—5. (See also this Journal, 1897, 1024.)

Apart from the influence exerted by light and warmth, the drying of varnishes is affected by the percentage of moisture in the air, the author's experiments going to show that the lead- and lead-manganese varnishes dry more quickly, whilst the drying of manganese varnishes is retarded, in a moist atmosphere; though the results obtained under apparently identical conditions are not always constant.

The age of the varnish also influences the rate of drying. Generally speaking, those prepared with lead-manganese compounds (irrespective of their composition) suffer diminution in this respect, whereas manganese varnishes remain constant, unless the quantity of drier used is so large, that a portion separates out during storage.

In the author's experiments, wherein the rapidity of drying was taken as the basis for comparison, as being of greater practical utility than the amount of oxygen absorbed during the operation, simple drying oils composed of linseed oil and solid driers were employed. The results show that the drying power increases with the percentage of driers used, up to a maximum, and then remains constant. The maximum effect is produced with a (metal) content of 0·2 per cent. of manganese, or 0·6 per cent. of lead-manganese (in the ratio of 5 : 1). The reported decrease in drying power resulting from increased additions of driers is probably due to deposition of the siccative, or to neglect of the necessary precautions in experimenting.

With regard to commercial driers, fineness of division is a great factor in the efficiency of the manganese oxides and borates, and the lead oxides; manganese borate and lead acetate are preferably employed in the anhydrous condition; the various preparations of lead-manganese resinate (dry siccatives) chiefly contain a preponderance of the former metal (5 : 1), the converse being of less frequent occurrence. Lead-manganese linolate is mostly sold dissolved in oil of turpentine (2 : 3 or 4 : 5), the resinates being more rarely employed in this condition, and so constitutes an intermediate grade leading to the lacquer varnishes.

—C. S.

### PATENT.

*Preservative Compositions for Coating Metal, Canvas, or the like, Impts. in.* H. L. Bates, Portsmouth, New Hampshire, U.S.A. Eng. Pat. 22,867, Oct. 5, 1897.

This material dries quickly, gives a transparent film, and does not crack if the surface to which it is applied is bent. It consists of: acetic acid, 1 quart; glucose, 1 oz.; white-grape juice, 1 oz.; and ⅛ oz. each of powdered borax, sodium carbonate, paraffin wax, citronella, fine salt, alum, and nitre.

## (C.)—INDIA-RUBBER, &c.

### PATENT.

*Rubber or other Gum, and the like, dissolved in a Solvent; Impts. in or connected with the Manufacture of Articles [Pneumatic Tyres, &c.] wholly or partly of.* F. King, London. Eng. Pat. 9538, April 14, 1897.

When a fabric, such as canvas, is coated with rubber in solution, the evaporation of the liquid occupies a considerable time; and, if the operation be hastened by the application of heat, the drying is not uniform, and the fabric is apt to be injured. The inventor, accordingly, places the articles in a vacuum chamber, thus expediting the process and recovering the solvent.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Bates and New Bating Materials.* Der Gerber, **24**, [563], [564], and [565].

Notwithstanding the many changes which have of late years been made in the processes of leather manufacture, the "bate" is still used to prepare the pelt, and is one of the most subtle, difficult, and least-understood processes connected with the production of leather, the importance and necessity of which, in spite of the many methods suggested to do away with its use, are still generally acknowledged.

The "dung" and "bran" bate are still, by some, looked upon merely as methods of cleansing the pelt, by which the lime is removed from the unhaired hide, and also as a means of loosening the scud and dirt; but the functions of the bate are much more important, for it is in the bates that the character and quality of the leather to be produced from the skins, is determined. Where the dung or bran bate are used merely for the removal of lime and dirt only, they should be substituted by some of the many other materials, cheaper and better suited for the purpose. The chief action of proper bating is the opening up of the fibre of the skin, by which the leather is made pliable, soft and mild, and the grain finer and less liable to crack, and, at the same time, by opening the fibres of the pelt it becomes possible to remove the scud, hair sacs, and other extraneous matters by mechanical means. Before subjecting the pelt to the bating process, one must consider the result desired, whether it be—

(1.) The removal of lime from the pelt.

(2.) Removal of lime, together with the scud and dirt, from the pelt.

(3.) The partial liquefaction of the pelt, as preparation for the production of pliable and mild leather.

(4.) The raising of the pelt and opening of the fibres and pores in order to prepare the skin to take up the tannins.

*Bates for the Removal of the Lime only.*—The thorough removal of the lime previous to putting the pelt in the tan liquors is advantageous for all classes of leather, although in sole leather yards it is customary to remove only part of the lime, by washing the hides in water, and trusting to the natural acids in the liquors for the removal of the remainder. This is accomplished by washing or drumming the hides in water. If the water be soft, a certain percentage of the lime is dissolved out, as indicated usually in a loss of plumpness by the hides; but if the hides be washed in hard water containing bicarbonates, instead of the lime being removed, it is transformed into carbonate of lime, and fixed more firmly than before. The minute cavities of the hide becoming filled with this substance, the removal of the lime and dirt on the scudding beam is thus prevented, and at the same time the grain is rendered brittle.

To remove the lime wholly from the hides, it is usual to put them into water containing some material which will form normal and soluble salts with the lime, which can be easily dissolved out; and many materials have been suggested and used with successful results, among which the following are the most important:—

*The addition of sugary matters, as glucose and molasses.*—These form soluble calcium saccharates and glucosides, and were used centuries ago by the Moors to remove the lime from the skins in the production of Morocco leather.

*The use of carbon dioxide (Nesbit's process).*—The hides are put in water, which is saturated with this gas; carbonate of lime is formed in the first instance, and then re-dissolved in excess of the gas. This, however, was not found satisfactory. Experiments showed that the lime in various packs so treated was only reduced from 2·45 per cent. to 2·14 per cent.

Other de-liming agents are "cresotinic acid," "anticalcium" (cresol sulphonic acid), Borol (consisting chiefly of sodium hydrogen sulphate with small quantities of boric acid).

*Sulphuric acid* has also been proposed. This, however, was only used because it kept the hide "plump"; it did not remove lime.

*Acetic acid* gives good results, although it produces a coarse and rough grain, and the commercial pyroligneous acid always contains products which have an affinity for the fibre, and prevent tannage.

*Lactic acid* has the objection of being too costly.

The best material is undoubtedly *hydrochloric acid*, which, if used as follows, always gives satisfactory results. The hides are suspended in water, or, better, placed in a revolving latticed drum, sunk in an outer pit of water. For every 50 average hides about 5 galls. of commercial acid free from iron, should be added, at intervals, in five successive portions. This quantity will not be suited for every tan-yard, as the quantity of lime varies, according to the process of preparation. The most reliable method is to continue the addition of acid until the water gives a permanent sherry colour to blue litmus paper. After 2—5 hours the lime will be dissolved out, and the hides may then be scudded and passed on into the liquor pits. The hides thus freed from lime will have lost their plumpness, which was due to the action of the lime on the raw pelt, but will soon regain their substance in the acid tanning liquors. They also appear dark in colour; this will likewise disappear as the tanning advances, and the finished leather made from pelt properly de-limed will be brighter in colour, better in quality, finer grained, and have a pleasant mild feel.

—J. G. P.

*Glue, The Testing of Commercial.* R. Kissling. Chem. Zeit. **21**, 172.

*See under* XXIII., *page* 384.

*Suède or Glove Leather, The Dyeing of.* Der Gerber, **24**, [562], [563], [564], [565], 30; 43; 55; 67.

*See under VI., page 345.*

### PATENTS.

*Leather for Photographic Purposes, Manufacture of Prepared Sensitised.* O. Imray, London. From the Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main. Eng. Pat. 6769, March 15, 1897.

*See under XXI., page 371.*

*Gelatinous Substances, Skins, and the like, Impts. in the Treatment of, in order to obtain Superior Elasticity, Durability, and Resistance to Air, Water, Grease, and Wear.* A. Zimmermann, London. Eng. Pat. 8391, April 1, 1897.

The use of formalin for tanning skins is rendered practically impossible, on account of the hardness of the product. By using glycerin, vaselin, oil, or yolk of eggs alone, or mixed with flour at the same time as the formalin is being applied, this hardening action is prevented, and animal skins or gelatin films of superior elasticity are obtained.—J. T. W.

*Leather for Gloves, Impts. in the Manufacture of.* Alois Chiba, Carlsbad. Eng. Pat. 9820, April 17, 1897.

The skins are tawed with alum in the usual way, and allowed to age for 4—6 weeks; they are then washed in warm water (25° C.), and finally tanned in a one-bath chrome liquor; this is made by dissolving hydrated oxide of chromium in hydrochloric acid, and rendering the solution basic by the addition of soda. It is then diluted to 22° B. One hundred skins, weighing about 25 kilos., require 3½ litres of solution in 30 litres of water, which is worked into them in a fulling vat. After this operation the skins are treated for an hour with 4 litres of egg yolk (10 per cent. of salt), as little water as possible being used. The glove leather thus produced is said to be more durable than alum leather, and may also be washed in cold or warm water without detriment to quality or colour.
—J. T. W.

*Vegetable Glue, A New and Improved, and a Process for Manufacturing the same.* S. Herzberg, Berlin. Eng. Pat, 12,326, May 13, 1897.

Soft water, acidulated with from 2—6 per cent. of sulphuric acid, is warmed; from 5—30 per cent. of potato flour is then added, with constant stirring until perfectly dissolved, the quantity depending on the purpose for which the glue is required. From 2—5 per cent. of sodium hydroxide is now added, and the glue is ready for use.—J. T. W.

*Tanning Liquids, Impts. in the Treatment of, for Clarifying, Bleaching, or Decolorising them.* P. G. Sanford, London. Eng. Pat. 12,450, May 20, 1897.

The extract before concentration is treated with a vegetable or blood albumin, together with an alkali fluoride or borofluoride. The albumin is subsequently coagulated by means of heat or the action of *picro-citric acid. As an example, a decolorising solution may be made as follows:—One part of ammonium fluoride is dissolved in water at 25°—35° C., to this is added 150 parts of bran, and the whole is placed in a dialyser. The vegetable matter is then pressed, and the liquid filtered. Boric acid may be added after dialysis is complete. This solution is then added to the solution of tannin, stirring so as to cause complete mixture. The temperature is then raised to 65° C., when coagulation takes place. In the case of liquids decolorised with blood albumin, there is added a strong solution of picro-citric acid, made by dissolving 10 parts of picro-citric acid and 20 parts of citric acid in 770 parts of water. The quantity of this solution necessary, is one-eighth to one-quarter part of the total quantity of active material present. If the liquid, prior to decolorising, be acid, it may be neutralised by adding the necessary quantity of a solution of sodium borate, 37·5 grms.; sodium hydrate, 0·9 grm.
—J. T. W.

## XV.—MANURES, Etc.

### PATENT.

*Artificial Manures or Fertilisers, Impts. in or relating to.* A. Gladstone, Seaton Carew, Durham. Eng. Pat. 9369, April 13, 1897.

Common slag, or the by-product or waste from blast furnaces, is powdered, and mixed with any of the usual fertilisers, for application as a manure.—E. S.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar-Cane Juice and Raw Sugar, Chemical Composition of.* J. W. Leather. Agricult. Ledger (Govt. of India), Calcutta, 1895, [13], 1—7.

The results are given of a number of analyses of sugar-cane juice and of raw sugar made at the Cawnpur and Poona farms during 1894–95, and several analyses of the whole cane are referred to, the object being to compare the differences in the quality of the juice and of the *gur*.

At Cawnpur many of the plots were "laid" by excessive rain, and gave considerably lower results than the standing cane. Omitting these, the average amount of sugar in 100 parts by weight of the juice taken immediately after pressing, was from 13·5 to 15·5.

The Poona crop, not being "laid" by rain to any extent, showed much greater uniformity, although a sharp distinction may be drawn between individual plots. Plots 5 to 15 showed 15·18 to 17·33 per cent.; plots 18 to 24, 12·87 to 15·11 per cent. The differences in the quality of the juice of these plots cannot be referred to different methods of manuring, or to the different amounts of nitrogen and of phosphoric acid applied in the manures. The most plausible explanation is that the slope of the land may have indirectly caused the difference, there being a gentle slope of black open soil, and soft rock (murrum) within about two feet of the surface, so that the drainage water possibly assisted the cane on the lower-lying land to produce more concentrated juice than that on adjacent plots lying higher.

Samples of raw sugar were analysed from both farms, as well as some of the *gur* prepared by the cultivators.

That made from cane on the Cawnpur plots 1 to 9 (excluding those of "laid" cane) contained from 70 to 76·5 per cent. of cane-sugar, 7·9 to 10·9 of glucose (reducing sugar), 10·9 to 14·9 of water, and 1·16 to 3·26 of mineral matter. The "laid" cane showed less cane-sugar and more glucose, so that both as regards the juice obtained and the *gur*, at least for this variety of cane, the "matna," it is important to prevent it from being laid by heavy rain and wind.

Four other varieties of cane were grown on waterlogged fields, the cane being so much damaged that the weight of the sugar obtained was far lower than it ought to have been; still it gave a *gur* with a higher percentage of cane-sugar and a lower percentage of glucose than in that from adjacent crops of the *matna* cane.

Reference is made to one or two points in the qualities of the Cawnpur farm *gur* and the Poona *gul*. Both these names apply to the raw sugar obtained by boiling down the juice until it solidifies on cooling. The cultivators apparently endeavour to produce as great a weight of sugar as possible irrespective of purity; but although the *gur* prepared on the farms was all passed through a cloth before being boiled down, and that of the cultivators contained much dirt, bits of cane and earth, still judging by analysis, they appear to be of about equal quality.

The cane-sugar in the samples of Poona *gur* was higher than in that prepared at Cawnpur, but the glucose was higher also, whilst the moisture and ash were both less. When, therefore, it is found that from three to four times as much raw sugar per acre is produced at Poona as at Cawnpur, it is evident that this result must be of more importance than the composition of the *gur*. Refined sugar being now made on a large scale in the neighbourhood of both farms, attention is drawn to the amounts of glucose and mineral matters as affecting the suitability of the raw sugar for refinery purposes. The smaller amount of glucose and higher ash in the Cawnpur samples over those from Poona may be referred to some inherent quality of the juice, but is more probably due to the fact that at Cawnpur, a mixture of potash and a mucilaginous substance was added to the juice to neutralise acidity and prevent inversion, and to precipitate albuminoids. As it is the soluble ash which refiners consider prevents crystallisation of sugar, it may be advantageous to add caustic lime, or carbonate of lime, instead of potash.

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* Esbach's solution, composed of 10 grms. of picric acid and 20 grms. of citric acid dissolved in water to form 1 litre of solution.

When the cane is crushed in an ordinary mill, only a portion of the juice, and therefore of the sugar, is extracted. By an indirect method, it was estimated that at Poona from 2 to 2·5 per cent. of sugar was thus lost, and at Cawnpur no less than 5 per cent., the latter representing fully two-fifths of the total sugar in the cane. This must not be hastily attributed to the mills, but may be due to the quality or hardness of the cane itself; and if this should prove to be the case, it shows the desirability of growing a cane from which a higher proportion of juice may be extracted. From the *matna* cane at Cawnpur, 50 per cent. of juice was extracted, whilst no less than 69 per cent. was obtained from *pounda* cane crushed by Sabbiah.

The sugar-cane crop is generally supposed to be very exhausting to the soil. An attempt was made to determine the nitrogen and phosphoric acid, but difficulty was experienced in getting representative samples. Assuming 40,000 lb. of cane and 4,000 lb. of tops per acre to represent the Cawnpur crop, the phosphoric acid was about 24 lb. and the nitrogen 34 lb. per acre. The Poona crop, with 100,000 lb. of cane and 14,000 lb. of tops, gave 50 lb. of phosphoric acid and 94 lb. of nitrogen per acre. A crop of wheat of 600 lb. of grain per acre will (including the straw) remove about 6 lb. of phosphoric acid and about 13 lb. of nitrogen per acre.—L. J. de W.

*Sugar-Cane and Raw Sugars, Chemical Composition of.* J. W. Leather. The Agricult. Ledger (Govt. of India), Calcutta, 1896, [19], 1—21.

The experiments on the growth of sugar-cane carried out at Poona, Cawnpur, Dumraon, and Burdwan during 1895–96, lead to the following conclusions :—

(1.) It is evident that the juice of different varieties of cane contain very different proportions of sugar; further, that this proportion of sugar is not materially affected in any one year by any description of manure or its amount. This proportion of sugar in a cane may be affected seriously by a change of climate.

(2.) The proportion of glucose (reducing sugar) in the juice of different varieties varies considerably; this proportion is increased, in some cases, largely during the boiling process; but *inversion* may be prevented in a great measure by the addition of lime. It is also probable that cultivators could easily be taught to *lime* their juice, if there were any call for it.

(3.) During the boiling-down process there is a loss of about 10 per cent. of the sugar present in the juice, most of which is carried away in the scum. This sugar need not, however, be lost in the economic sense. Cattle may be fed with it, or (as at Poona) some at least of it may be recovered.

(4.) The amount of juice, and consequently of sugar also, which remains unexpressed from the cane, depends on the proportion of crude fibre in the cane. Therefore, it is desirable to grow varieties of cane containing a low proportion of crude fibre. The amount of sugar remaining in the crushed cane may be as much as nearly one-half that in the cane, or it may fall to as low a proportion as one-fifth.

(5.) The amounts of nitrogen and of phosphoric acid taken up per acre by the sugar-cane crop vary from some 40 lb. each (or less in poor crops) to about 100 lb. in such heavy crops as those grown at Poona.—L. J. de W.

*Sugar-Cane and Raw Sugars, Chemical Composition of.* J. W. Leather. The Agricult. Ledger (Govt. of India), Calcutta, 1897, [3], 1—35.

The experiments on the growth of the sugar-cane at the four farms, Poona, Cawnpur, Dumraon, and Burdwan, were continued during 1896–97.

To avoid loss of sugar in the scum, it is desirable to remove this scum as quickly as possible, whilst the juice is dilute.

The amount of sugar in the juice of fallen canes is smaller than that in standing canes, but the deficiency is not so marked as in the case of canes laid by rain. When the crops grow very tall, they are liable to fall down considerably unless tied up with the dry leaves, a method commonly practised in the Burdwan district, where tying up commences when the crop is only a couple of feet high. Canes, even when 14 to 16 feet high, will remain erect if properly supported in this manner.

Canes transported from long distances gave, in some cases, much lower results than in their original districts, in others slightly higher, and in one case the yield remained stationary. A second year's crop shows improvement in some cases, in others none. Where heavy manuring had increased the quantity of juice, the proportion of sugar had fallen very seriously, but it must not be inferred that heavy manuring is to be deprecated generally.

The amount of juice from the top end of the cane is very much less than that from the main portion of the stem, and the amount of cane-sugar and of total sugar is also much higher in the juice of the main part of the stem than in that of the top, but the amount of glucose is lower in the former than in the latter. Assuming that just as good a crop will be obtained from planting with tops only, instead of cutting up the whole cane, and, further, that cane will not deteriorate by being thus propagated, it will be evident that it is much more economical to grow from tops only.

In estimating the sugar contained in the juice by the hydrometer, if 2·0 be deducted from the percentage of sugar calculated from the density, the error lies between very narrow limits, and a very fairly accurate idea would be obtained of the amount of sugar present in the juice of any variety to be examined.

In some localities a description of raw sugar is made, which consists of a semi-liquid mass obtained by stopping the boiling process a little sooner than is the case when hard *gur* is wanted. It is transferred from the boiling pan to an earthen vessel and allowed to cool slowly, when a considerable amount of cane-sugar crystallises out. At Burdwan this substance goes by the name of *gur*, but at Behea it is called *rab*. By hand centrifugals, about 50 per cent. of the *rab* may be obtained as a semi-refined sugar called "turbine-sugar," and the rest is boiled down to *gur*.

It is found that very little inversion takes place during the boiling process if nine-tenths of the acidity of the juice is neutralised with lime, litmus paper being used as indicator. The yellow colour of the cane juice itself, when made alkaline, may also serve as indicator. When, however, this yellow colour becomes apparent, the juice is strongly alkaline, so that it has been deemed advisable to only add lime to half the juice until it became yellow, and then to add the other half to render it all acid again. If an excess of lime were employed, the resulting *gur* would be black.—L. J. de W.

*Sugar Cane, The Lecithins of.* E. C. Shorey. J. Amer. Chem. Soc. 1898, **20**, [2], 113—118.

After the precipitation of the albuminoids in raw cane-juice, syrup, or molasses by copper hydroxide, phosphotungstic acid added to the filtrate produces a precipitate, chocolate in colour, flocculent, and easily washed, and invariably containing nitrogen. To determine the character of this nitrogenous compound, it was decomposed by digesting it with barium hydroxide or carbonate, or milk of lime. Salts of a fatty acid were obtained, and in order to isolate the body from which the fatty acids were evidently formed by decomposition, the following method was adopted :—The precipitate produced by phosphotungstate of sodium was nearly dried over sulphuric acid, mixed to a paste with dry sodium carbonate, and extracted with 90 per cent. alcohol, which resulted in a dark red solution. The alcohol was removed by distilling in a vacuum, and the residue taken up with a small quantity of water. The watery solution was agitated several times with ether. After evaporation of the ether from the ethereal solution, there was left a light yellow, translucent, wax-like solid, which appeared to be a lecithin.

Sugar-cane lecithin was proved to give more than one fatty acid, showing it to be a mixture of several lecithins. The alkaloid resulting from the decomposition of sugar-cane lecithin was found to be a mixture of betaine and choline.

The ease with which lecithin is decomposed has suggested one point of interest to the sugar manufacturer. The grease found in the first cell of a multiple effect is, of course, derived from the oil in the exhaust steam, but that in the second and third can be derived only from the boiling juice. It is no doubt derived in part from fats and wax naturally occurring in sugar cane, but also, no doubt, is due in part to the decomposition of lecithin and the carrying over of the freed fatty acids with the steam. The decomposition takes place slowly in boiling neutral solution; in alkaline solutions it is very rapid.—L. J. de W.

*Sugar-Cane Amide, Additional Notes on the.* E. C. Shorey. J. Amer. Chem. Soc. 1898, **20**, [2], 133—137.

The author has obtained additional proof of the correctness of the conclusion reached in a former paper that the amide of sugar cane is amido-acetic acid and not asparagin.

A much more satisfactory and conclusive test of the character of the sugar-cane amide is the formation of hippuric acid from it and benzoic acid on heating in a sealed tube at 160° C. for ten or twelve hours, or more simply by the action of benzoyl chloride on the amide. This reaction promises a simple and reliable method for its estimation.
—L. J. de W.

*Glucose, Turbidity in Starch.* D. Saare. Zeits. für Spiritusind. 1898, **21**, [9], 79—80.

During the preparation of glucose from starch, as well as in the finished syrup ("Stärke-syrup"), there appears at times a turbidity which seriously impairs its quality, or necessitates reworking, with incidental increase of cost on the finished product in bright condition.

An insufficient conversion of the starch, as also when it is under-boiled, causes the juice to remain dull or bluish; it then gives a violet colour with iodine solution.

When over-boiled, too much dextrose is formed, and the dextrin present is insufficient to prevent its crystallisation; it therefore appears as specks, and on shock or agitation the syrup is apt to become solid.

When sulphuric acid is used, crystals of sulphate of lime often separate in the syrup if it is insufficiently concentrated in the first stage. In this case it should be thickened to 31° or 33° B., and filtered as hot as possible. The finished syrup should be diluted and returned to the liquor.

One case is recorded (*ibid.* 1884, 711) where a turbidity was due to sulphate of lead, possibly derived from the sulphuric acid used for conversion.

Glucose syrups have frequently been received which are dull from separation of phosphate of iron. Dull syrup contained 0·051 per cent. of phosphate of iron; clear syrup showed only traces, or a maximum of 0·006 per cent.

One very fine syrup, almost free from phosphate of iron, showed, however, 0·0113 per cent. of oxide of iron in some other combination.

In isolated cases, syrups have been found dull from the presence of organisms which must have found their way in during cooling, or from the casks. In one striking case, the syrup, containing about 20 per cent. of water, showed budding yeast cells. The percentage of sugar was about 40 per cent.—L. J. de W.

*Phosphates, Estimation of the "Citrate-soluble" Phosphoric Acid.* O. Böttcher. Chem. Zeit. 1898, **22**, [21], 201.

*See under* XXIII., *page* 380.

*Glucose, Quantitative Analysis of.* E. Pflüger. Pflüger's Arch. **69**, 399; Chem. Centr. 1898, **1**, [6], 420.

*See under* XXIII., *page* 381.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Bavarian Beer, Residue and Sediment, Filtering.* G. R. Allgem. Anzeig. fur Brauereien, **14**, [5], 136.

In consequence of the thicker character of Bavarian and brown beers, due to the higher kilning of the malt, they are more difficult to filter than paler beers, and though this difficulty is less apparent in the hot wort passing through the grains, it becomes inconvenient in the recovery of the wort retained by the sediment and cooler-sludge, the liquid setting to a jelly in the filter bags on cooling, and thus resulting in a loss of the unfilterable wort. This difficulty can, however, be surmounted by the aid of suitable filter presses, the whole of the contained wort being quickly expressed without risk of souring or infection by exposure to the air.—C. S.

*Diastatic Ferments, Influence of Heating on.* A. Pugliese. Pflüger's Arch. **69**, 115—131; Chem. Centr. 1898, **1**, [4], 265.

Diastatic ferments of different origin, as ptyalin and diastase, are not identical, as different temperatures have been observed as optima of their activity. It is shown also that the activity of the ferments, and especially their behaviour towards the influence of temperature, depends very largely on the concentration, reaction, and salt- and albumin-content of the solutions. Thus the optimum of activity of one ferment is sometimes higher, sometimes lower than that of the other, according to the concentration of the solution. Toluene does not injure the action of the ferment of the saliva at all, but thymol does, proportionally to the dilution.—A. S.

*Wines ["Boiled"], Some Analyses of.* G. Paris. Zeits. f. Untersuchung. d. Nahr.- u. Genussmittel, 1898, **3**, 164—167.

The so-called boiled wines (vini cotti) are those obtained by fermenting grape juice which has been evaporated to half its volume, and to which a little fresh must has been added. A tabular statement of the results obtained from the analyses of 11 of these wines is given in the original paper.

Only two of the samples gave more than 0·2 per cent. of volatile acids, whilst all contained more levulose than dextrose. The proportion between the sugar-free extract and the ash was not the same in the different samples. None of the samples contained free tartaric acid, and the amount of sulphates (as potassium sulphate) was less than 0·2 per cent. The proportion of glycerin to alcohol (by weight) varied between 6·8 and 13·9. The quantity of phosphoric acid in the wines was 0·036 to 0·192 per cent., and of ferric oxide from 0·00236 to 0·00714 per cent.
—W. P. S.

*Wine Fermentation.* C. Boettinger. Chem. Zeit. 1898, **22**, [16], 138—139.

The author has made experiments on the fermentation of grape juice on the small scale, the grapes used having been damaged by birds and insects in the first experiments, left in a heap for some days in the second, and plucked in very unripe condition in the third. After 43 and 16 days respectively, the fermentation in the first two flasks had practically ceased, the amount of alcohol being about 6 per cent. There was still present a considerable quantity of reducing sugars (0·153 and 0·139 grm. per 100 c.c.), and this raised the question of the cause of the cessation of fermentation, and whether what is known as "after-fermentation" is accompanied by the decomposition of the products of the first fermentation. With the object of possibly throwing light upon these points, sugar was added in proportions in accordance with Pasteur's rule with regard to the yield of alcohol. After the addition, the extracts showed an increase of 1·2141, 3·5191, and 3·8250 grms. respectively. Wine yeast was added, and, after 15 days, when the fermentation had again ceased, it was found that the following quantities of extract had been fermented: 1·209, 3·4514, and 4·814 grms.—amounts closely corresponding with the quantities added, except in the case of the third flask, in which the fermentation had not ceased before the addition of the sugar. In each case the total acidity had decreased by 0·03 to 0·1 grm. per 100 c.c., and the phosphoric acid in the first and second flask by about 0·01 grm. A subsequent examination, 11 days later, of the wine in the second flask, on which a mould had formed, showed a further decrease in the total acidity and phosphoric acid, as also in the amount of extract, ash, and alcohol. This the author attributed to the action of the mould. The wine in flasks 1 and 3 was again examined after 22 days, during which "after-fermentation" had been slowly proceeding, and showed a decrease both in the extract and the ash, the relative proportions of each being almost as before. After another three weeks, the wine had begun to clear, the yeast falling, and on examination showed a further considerable decrease in the total acidity, and a slight decrease in the extract, ash, and phosphoric acid.—C. A. M.

*Spirit from Cellulose and Wood.* E. Simonsen. Zeits. für angew. Chem. 1898, [9], 195—196.

The author gives the following list of references to this subject in scientific literature:—Ann. de Chim. Phys. **12** (1819), 172; Dingler, **1** (1820), 312; **25** (1827), 81; **134**, 219 and 316; **136**, 187; Annales Mensuelles, April, 103; Erdmann J., **5** (1835), 217—228; Comptes Rend. 1854; Repert. of Pat. Invention (1855), 436; Dingler, **138** (1855), 79, 80, and 426; **151** (1859), 394; **181** (1866), 233; **185** (1867), 308; **273**, 276; **287**, 91; Wagner J. (1855), 200 and 220; (1856), 225 and 242; (1859), 40 and 401; Zeits. f. Spirit. 1883 [7]; Lindsay, Inaug. Dissertation, 1891; Muspratt, Techn. Chem., 4th Ed. 1. 601. He considers that the literature contains many inaccurate and impossible statements, and that it is extremely defective. Thus, for example, there is no record of any systematic investigation as to the effect of variations in the factors, such as amount of water, pressure, amount of acid, and time, in high pressure inversion; nor are there any comparative experiments on the inversion of cellulose and wood. That these investigations may have been made and their results kept secret is not impossible, since factories have been established; but such researches could hardly have dealt with high-pressure inversion, which has only been practically carried out on a large scale within the last 20 years. Yet the manufacture of spirit from cellulose material by means of inversion under such unfavourable conditions as that over 100 per cent. of sulphuric acid was required for the dry wood and a corresponding quantity of calcium carbonate (and taking into account the high price of the materials at that time and the length of time required for the process), seems to point to the fact that the inversion of wood will be the method of the future, if only a satisfactory process can be found.

It is highly probable that the time required for the inversion of cellulose could be considerably shortened by use of the modern high-pressure boiler, if the right conditions were present, and that a considerable yield of sugar might be obtained from sawdust, concurrently with its manufacture from potatoes—a material 50 times more expensive than sawdust. At the same time it must be remembered that sawdust is about seven times as bulky as potatoes. The amount of water in potatoes (about 75 per cent.) is a drawback to their employment, although the starch is easily converted, and a comparison of the composition of the potato with that of ordinary wood, such as pine and fir, shows that the latter would be far preferable as a raw material, provided that the cellulose and lignin (together about 83·3 per cent.) could be converted into sugar. But it is not known yet whether the exterior envelope (lignin) is capable of inversion, or to what extent the cellulose can be inverted.

With the object of throwing light upon these points, the author has made systematic experiments with cellulose (sulphite cellulose) and wood (sawdust), taking uniformly 40 grms. of the former and 100 grms. of the latter. The inversions were made in an ordinary Muencke autoclave, in which the effects of direct heating by fire are obviated, and the time was reckoned from the moment when the desired pressure was attained. When cold, the mass was pressed, washed with water, and the solution titrated with Fehling's solution. The liquids obtained, were yellow to brown, and had a pleasant odour, recalling that of vanillin in the case of cellulose, whilst a smell of furfural was noticeable in the product obtained from wood, especially when the pressure had been high. As a rule, precipitates formed in the filtered solutions after standing for some time, and in some cases where the liquid became quite turbid in a few minutes. On adding soda, the solutions became nearly black, the original yellow colour being restored by acids. It was observed that the liquid obtained from sulphite cellulose gave a flocculent precipitate on adding concentrated hydrochloric acid, but that this did not take place when filter-paper had been used as the raw material.
—C. A. M.

*Wine, Estimation of Potassium Bitartrate in.* L. Magnier de la Source. Rev. intern. falsific. **10**, 195; Chem. Centr. 1898, **1**, [2], 149.

*See under* XXIII., *page* 385.

*Methyl Alcohol, Determination of, in Mixtures with Ethyl Alcohol.* A. Lam. Zeits. f. angew. Chem. 1898, [6], 125.

*See under* XXIII., *page* 385.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Jamaica Ginger.* F. B. Kilmer. Chem. and Druggist, 1898, **52**, [932], 352. From Amer. J. Pharm.

The ginger harvest is from March to January, and as soon as the stalk withers and the bloom has departed, the rhizomes are twisted out of the ground with a fork, the soil and fibrous roots removed, and the root stalks thrown into water ready for peeling. The peeling is mostly done by experts by the aid of a knife, peeling machinery having proved unsuccessful. The peeling has to be done very carefully, as, under the outer striated skin, are numerous layers of thin-walled cork cells, the oil contained in which is, in fresh specimens, very pungent, exceedingly aromatic, and almost colourless. This corky layer contains the greatest amount of oil and resin cells, and thus the deeper the peeling is made, so much more of these substances are carried away with the epidermis. After peeling, the roots are thrown into water and washed, the water used being as pure as possible. If lime juice be used in the wash-water, a whiter product is obtained, but the saccharine matter and pectose contained in the lime juice prevent drying, and mildew follows. The rhizomes are finally dried for 6—8 days in the sun. The amount of ginger exported from Jamaica during the past 10 years was as follows, half of the total amount going to the United States:—

| | Lb. | | Lb. |
|---|---|---|---|
| 1887 .......... | 1,121.827 | 1892 .......... | 1,822,531 |
| 1888 .......... | 1,141,877 | 1893 .......... | 1,526,884 |
| 1889 .......... | 1,002.653 | 1894 .......... | 1,672,384 |
| 1890 (half year) | 554,193 | 1895 .......... | 1,736,460 |
| 1891 .......... | 1,219,197 | 1896 .......... | 1,960,609 |

—A. S.

*Butter, Glucose in.* C. A. Crampton. J. Amer. Chem. Soc. 1898, **20**, [3], 201—206.

It is stated that for the purpose of preserving butter for export to tropical countries, the manufacturers, especially in France, have found glucose the most satisfactory agent, when used in conjunction with a large amount of common salt.

The figures obtained by the analysis of three samples of such butter are given on the next page.

Butter No. 2,443 was condemned by the customs authorities, on account of the Reichert figure (25·1) being considered too low, but the presence of glucose was not detected.

The author examined a sample of glucose as used by the makers, and found it to consist of the ordinary syrup known as "confectioner's glucose"; it contained 16·5 per cent. of water, and gave $[a]_D$ = 119·3.

| — | No. 2,131. Butter sent from U.S. to Brazil. | No. 2,443. Butter sent to Brazil and returned to U.S. | No. 2,400. Beurre rouge sent to Guadeloupe. |
|---|---|---|---|
| Water | 16·29 | 18·93 | 21·60 |
| Casein (N × 6·25) | 1·19 | 1·06 | 0·81 |
| Ash | 7·00 | 6·19 | 16·42 |
| Fat | 72·16 | 69·67 | 51·15 |
| Glucose (by difference) | 3·36 | 4·15 | 10·02 |
| | 100·00 | 100·00 | 100·00 |

For a direct test, 10 grms. of the butter are extracted with successive portions of hot water, and the solution made up to 250 c.c. Any considerable reduction of Fehling's solution by this liquid would point to the presence of glucose or sugar.—C. A. M.

*Sesamé Oil in Butter and Margarine, Detection of, in the Presence of Artificial Colouring Matters.* C. A. Neufeld. Zeits. f. Untersuchung d. Nahr. u. Genussmittel, 1898, **3**, 156.

*See under* XXIII., *page* 381.

*Coffee, Artificial Colouring of, and Means for its Detection.* G. Morpurgo. L'Orosi, 397; Chem. Centr. 1898, **1**, [7], 472.

*See under* XXIII., *page* 381.

*Starch, &c., in Chocolate, Detection of.* G. Possetto. Giorn. Farm. Chim. **48**, 5; Chem. Centr. 1898, **1**, [6], 418.

*See under* XXIII., *page* 381.

## PATENTS.

*Alimentary Substances and Beverages, Impts. in and relating to the Treatment of.* J. Hogarth, Kirkcaldy, Fifeshire. Eng. Pat. 4695, Feb. 22, 1897.

The claims are for processes in which grain, flour, fermented or fermentable liquors are subjected to the action of electricity.

In the case of grain or flour, the substance is passed between plates or wires electrified with "Tesla" currents, the current passing across the material from one plate to the other. Other electric currents may be used instead of the above, or the grain may be subjected to the rays of incandescent lamps. In applying the process to liquors, the patentee uses the Röntgen rays, but "Tesla" or other currents may be used.—W. P. S.

*Preservation of Articles of Food, Impts. in the, and in Compositions for Same.* E. May and E. Kühne, Vienna. Eng. Pat. 7384, March 22, 1897.

The process claimed is one for the preservation of animal foodstuffs by coating the same with a solution of sodium benzoate, hydrofluoric acid (or boroglycerin), tartaric acid, and sodium bisulphate in a mixture of water, glycerin, methyl alcohol, and ethyl alcohol.—W. P. S.

*Preservative Material for Protecting Food Substances, and Process for Preparing the Same; Impts. in.* E. May and E. Kühne, Vienna. Eng. Pat. 7385, March 22, 1897.

Paper, pasteboard, textile fabrics, bladder, &c., are dipped in a solution consisting of half a litre each of methyl alcohol and ethyl alcohol, about a quarter of a litre of glycerin, and 10 grms. of a fluorine compound (ammonium fluoride) or other antiseptic (borate or benzoate). The meat or other article to be preserved is then wrapped in the paper or cloth so impregnated.—W. P. S.

*Diastasic Preparation to be employed in the Manufacture of Bread and other Foods and Beverages, and to be used as an Aid to Digestion; An Improved.* H. J. B. Mills, London. From A. Dalmote, Ypres, Belgium. Eng. Pat. 8080, March 29, 1897.

The preparation is a wort of undried malt obtained by crushing the malt with water, allowing the milky fluid to saccharify, and filter-pressing. The resulting wort is cooled, again filtered, saturated with carbonic acid gas, and stored in hermetically closed bottles or vessels.—W. P. S.

*Sterilising Milk and other Liquids or Substances, Improved Means applicable for Use in.* R. G. Nash, Lucan, Ireland. Eng. Pat. 8977, April 8, 1897.

The liquid to be sterilised is put into bottles, which are closed with corks having a V-shaped notch at their lower ends. The corks are forced a sufficient distance into the neck of the bottle, so that the top of the notch is just above the lip of the bottle, thus forming a narrow vent for the escape of gases. The bottles are then placed in a steam-tight chamber, and an iron plate in the same is screwed down until it rests just above the corks. Steam is admitted, and when the sterilisation is complete, the iron plate is screwed down, and thus forces the corks completely in. Other devices may be used instead of the above-mentioned notch, such as a small tube by the side of the cork, which is removed before the cork is driven home, or a hole in the neck of the bottle, which is closed when the cork is forced in. The patentee also claims a process for heating the milk before sterilisation, to remove bad odours.—W. P. S.

*Kola-Nut in the Preparation of a Mixture suitable for Beverages; Impts. in and relating to the Treatment of.* C. Junge, London. From E. Amos, Rorschach-am-Bodensee, Switzerland. Eng. Pat. 19,516, Aug. 24, 1897.

According to this invention, the disintegrated kola-nut is mixed with one-tenth of its weight of syrup, to coat the particles, and then slightly roasted. Crushed malt is now added. The object of the invention is to render the mixture of malt and kola-nut suitable for transportation. The malt gives colour to the beverage and prevents over-roasting of the nut.—W. P. S.

## (B.)—SANITATION; WATER PURIFICATION.

*Manchester Sewage Disposal.* Chem. Trade J., April 9, 1898, 254.

*The Davyhulme Experiments.*

In accordance with the directions from the Rivers Committee of the Corporation, Mr. G. J. Fowler (the Committee's chemist) has prepared a brief report describing the small experimental filters at Davyhulme, and summarising the results obtained during the last 12 months. Having given details of the structure and mode of working of the different classes of filters, Mr. Fowler says that the two coke and cinder filters, allowing for Sunday's rest, are now dealing with the effluent at the rate of 677,000 galls. per acre a day; the coal filter is dealing with the effluent at the rate of 508,000 galls. per acre a day, and the burnt-clay filter at the rate of 837,000 galls. per acre a day. As exhibiting the work done by the filters in converting the organic matter in the tank effluent into harmless products, he presents the following table showing the average result of daily analyses during the period from January 6th to May 12th, 1897:—

| — | Oxygen Absorbed Four Hours. | Free and Saline Ammonia. | Albuminoid Ammonia. |
|---|---|---|---|
| | Grains per Gallon. | Grains per Gallon. | |
| Tank effluent | 2·24 | 2·06 | 0·29 |
| Coke filter | 0·91 | 1·52 | 0·15 |
| Cinder filter | 0·72 | 1·27 | 0·11 |
| Coal filter (from week ending Feb. 24, 1897). | 0·73 | 1·75 | 0·12 |

The results of the incubator test are also set out in detail. It is concluded on the data supplied that the most suitable filtering material for artificial filters is furnace cinders

*Comparison on Filters.*

Good results, it is added, can also be obtained from coal, and the material might be burned in case of renewal of the filter beds, but "experience has shown that it is rather more difficult to keep the filtrate non-putrefactive and the surface from clogging in the case of a coal-filter of a comparatively small capacity, such as that at Davyhulme. A coal filter of greater openness does not appear to be likely to offer much advantage over burnt clay, the results from which, however, are not altogether satisfactory." With regard to the capacity of filters, Mr. Fowler reports that there is no doubt the insoluble inorganic matter in the tank effluent must gradually accumulate in the filters, and in time cause a loss of capacity, but this loss of capacity does not continue at the same rate after the first year's working. The life of a biological filter would be increased by the use of a preliminary roughing filter. Whether the first cost and subsequent maintenance of this would entail greater expense than the occasional renewing of the material of the final filter is a matter to be determined. The clogging takes place to the greatest extent on the surface, at the edge of the filters. If this becomes excessive, occasional rest and a turning over of the clogged portions of the surface, so as freely to expose them to the air, enables the filter to work satisfactorily again. It is possible that in a larger area the clogging would be relatively less, the greater part still taking place at the sides, and leaving a large free space in the centre. This can only be determined by experiment.

Regarding the chemical activity of filters, it is stated that "the fact that the albuminoid ammonia figure for the cinder filter, which is the most satisfactory in its working, is slightly less now than at a corresponding period of 1896, shows that the chemical efficiency of this filter is unimpaired. Owing to the presence of oxidisable manufacturing refuse in the sewage, the figures for oxygen absorption are somewhat above the Mersey and Irwell Joint Committee's limit, but the incubator test shows that very little sewage impurity is present in the filtrate from the cinder filter." Mr. Fowler, in conclusion, says: "While it cannot be guaranteed that under no circumstances will a filtrate be obtained which is above the Joint Committee's numerical standard, yet, with very few exceptions, it has been shown that a filtrate which is incapable of undergoing putrefactive change can be constantly obtained from the cinder filter. With increased knowledge of the conditions of successful filtration of such a tank effluent as is produced in Manchester, it is likely that even more satisfactory results, both as to quantity filtered and quality of the filtrate obtained, may be looked for in the future."

## PATENTS.

*Sewage, Impts. in and connected with Apparatus for Treating.* P. S. Hyslop, Walmer, Kent. Eng. Pat. 914, Jan. 13, 1897.

The sewage is led into a receiving and settling tank provided with a loose cover sealed by water, and flows thence, through heat-exchanging casings, in which it cools, and is itself heated by liquid, previously operated upon, to sterilisers, described in Eng. Pat. 6182 of 1896, in which it is sterilised by heat. From the sterilisers the hot liquid passes into a central receiving tank, back though the above-mentioned heat-exchanging casings, where it is cooled, then through passages under a sludge-drying platform into a settling tank, and lastly through filters. The foul gases are treated for the recovery of ammonia and other products of value, and are then burnt.—L. A.

*Sewage and Polluted Water, Impts. in the Purification of, and in Apparatus therefor.* F. P. Candy, Bowdon, Cheshire. Eng. Pat. 4040, Feb. 15, 1897.

Upward-flow precipitation tanks are divided into compartments by means of partitions extending above the level of the water, but not to the bottom of the tank. The sewage, flowing into a central compartment, escapes under the partition walls into the other compartments, whence it is drawn off; a more distributed flow is thus secured.

Filtration is conducted through a series of filters at different levels, the flow of sewage from one filter to another being provided for automatically allowing intervals for the aeration of each filter.—L. A.

## (C.)—DISINFECTANTS.

*Phenol Alcohols, Antiseptic Properties of the.* G. Cohn. Zeits. für Hyg. **26**, 377—383; Chem. Centr. 1898, **1**, [6], 397.

The growth of cholera bacteria and of *Staphylococcus pyogenes aur.* in nutritious cultures, was prevented by the addition of 0·1 per cent. and 2 per cent. of *saligenin* respectively. Cholera bacteria were destroyed in, at the most, 3 hours, by a 0·75 per cent. *saligenin* solution, and in 30 minutes by a 2 per cent. solution; for the destruction of the *pyogenes*, a 24 hours' action of 2 per cent. *saligenin* solution was necessary, whilst a 3 hours' treatment had no effect at all. Diphtheria bacilli offer little more resistance towards *saligenin* than cholera bacteria do.

*Eugenoform* is the sodium salt of the eugenol-carbinol, which melts at 39° C. It occurs in colourless laminæ, melting at 160° C., is soluble in water, slightly soluble in alcohol, insoluble in ether, and is decomposed by acids, even by $CO_2$, with separation of the free phenol. The compound has a faintly bitter taste, is not caustic, but exerts a local anæsthetic action. It is superior to *saligenin* as an antiseptic. It arrests the development of cholera bacteria when in a dilution of between 1 in 500 and 1 in 1,000, and effects the destruction of the same in 3 hours with a dilution of 1 in 500. The development of *pyogenes* is arrested with a dilution of 1 in 500, and complete destruction is effected after 24 hours with a dilution of 1 in 50. Towards diphtheria bacilli, eugenoform acts similarly to saligenin. It is probable that the free eugenol alcohol would act still more energetically.—A. S.

*Formaldehyde as a Disinfectant, Examination of.* K. Walter. Zeits. für Hyg. **26**, 454—475; Chem. Centr. 1898, **1**, [6], 396.

In previous experiments (Chem. Centr. 1896, **2**, 119) it was shown that formaldehyde acts efficiently as a disinfectant with regard to germs superficially situated, but that it is not able to penetrate the object to be disinfected. Experiments with Rosenberg's "Holzin" (35 per cent. formaldehyde solution in alcohol, with 5 per cent. of menthol to prevent formation of methylal) gave results no better than those obtained with other preparations; polymerisation of the aldehyde was not prevented, and the method, at present, is very expensive.

Having regard to the fact that steam which is kept in motion is more efficacious in penetrating objects than steam not in motion, the author tried formaldehyde vapour in the former condition (*i.e.*, in motion), with the object of penetrating the articles to be disinfected, instead of effecting a mere superficial disinfection. Formaldehyde vapour, together with steam under pressure, was passed into a cylinder (130 cm. long and 0·5 m. high), containing the articles to be disinfected; it was found that the formaldehyde penetrated to the interior of the articles. The mixture of aldehyde vapour and steam may afterwards be condensed, and the aqueous solution of formaldehyde thus obtained may be used for washing floors, &c. The author states that the chief result of this experiment is to be found in the indication it affords that the disinfection of a room and its entire contents in one action cannot be carried out by means of formaldehyde vapour, no matter by what method the latter is evolved. However, dresses, uniforms, and the like may be disinfected effectively and without injury by means of a current of formaldehyde vapour.—A. S.

## PATENTS.

*Vaporising or Fumigating Purposes, An Improved Apparatus for.* I. S. McDougall, Manchester, and J. T. McDougall, London. Eng. Pat. 6288, March 10, 1897.

There is claimed "an improved apparatus for vaporising or fumigating purposes, having two chambers or compartments joined together, one above the other, by any suitable support." The apparatus is constructed of tinned sheet iron or other suitable material. Into the upper chamber is put the substance to be vaporised, and the lower compartment contains some safely portable fuel, such as stearin, wax, &c., in which a wick is embedded. Each receptacle has a closely fitting lid, to prevent loss in transit. To start the vaporisation, the cap carrying the upper chamber is taken off and the wick lighted; the cap is then replaced. —H. B.

*Formic Aldehyde, Impts. in Vaporising Apparatus suitable for Converting Methyl Alcohol into.* F. Richard, Brussels. Eng. Pat. 8308, March 31, 1897.

The apparatus is in the form of a lamp, consisting of an annular wick holder which is supplied with methyl alcohol from a reservoir at the side. Above the wick holder, and within the chimney, a layer of platinised asbestos is supported between two perforated discs. Air is supplied partly through perforations in the base of the chimney support, and partly through the central tube of the wick holder. When required for use, the methyl alcohol is first kindled at the wick, and when the platinised asbestos has become red hot, the flame is extinguished. The heat of the incandescent platinum then causes the alcohol to evaporate from the wick, and the vapour becoming mixed with air, is converted into formic aldehyde.—L. A.

## XIX.—PAPER, PASTEBOARD, Etc.

*Rosin-Size made under Pressure.* Papier Zeit. **23**, [4], 110.

F. Arledter, of Christiania, obtained a patent (Ger. Pat. 95,016, 1897), for producing rosin soap which contains a large proportion of free rosin. The rosin is boiled with the alkali under a steam pressure of 10 atmospheres at a temperature of 100° to 200° C., while the mixture is being continually agitated. It is claimed that by this method of preparing the size, a much larger percentage of free rosin is held in suspension. It is also claimed that only 7 to 10 per cent. of alkali is required by this method, against 10 to 15 per cent. by the ordinary method, and the amount of free rosin is about 15 per cent. of the total rosin soap.

The results obtained by this method agree, and confirm Wurster's theory that only free rosin acts as a size upon the paper. By this method it is possible to size paper with $1\frac{1}{2}$ to 2 kilos. of size to 100 kilos. of paper.—S. P. E.

### PATENTS.

*Vegetable Parchment Paper; Impts. in or connected with Apparatus or Machinery for the Manufacture of.* J. S. Rigby, Liverpool, H. Sweetapple, Kensington, and J. Clarke, Liverpool. Eng. Pat. 6051, March 8, 1897.

To completely remove all traces of acid slime from the surface of the parchmentised paper, it is passed, after it leaves the water or weak acid tank, around rollers, against which ribs of rubber, wood, or other suitable material bear, attached to a hollow holder or back. The back may be perforated to distribute jets of water over the parchment. The brushes or scrubbers thus formed, are mounted so as to receive a reciprocating motion along the axes of the rollers against which they press.—R. S.

*Coloured, Marbled, Variegated, or like Papers; A New or Improved Process for.* O. H. Stone, Birmingham. Eng. Pat. 8114, March 30, 1897.

*See under* VI., *page* 348.

*Coloured or Decorative Effects on Paper or other Surfaces, A Process or Means for Producing.* J. Imray, London. From The Société Anonyme A. Roudillon et Cie., Paris. Eng. Pat. 10,901, May 1, 1897.

*See under* VI., *page* 348.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Alkyl Bismuth Iodides and Bismuth Iodides of Vegetable Bases. [Dragendorff's and Mayer's Reagents.]* A. B. Prescott. J. Amer. Chem. Soc. 1898, **20**, [2], 96—100.

The common alkyl ammonium iodides, with solutions of bismuth salts, give bright-coloured precipitates. As formed by quaternary methyl or ethyl ammonium iodides, the colour is orange-yellow in most cases, usually lighter when obtained with bismuth chloride, and darker when obtained with bismuth nitrate. When fully formed by excess of the organic iodide, in bismuth solution not strongly acid, the precipitation is amorphous, and so nearly complete that sulphuretted hydrogen fails to blacken the concentrated filtrate. Strong mineral acids slowly decompose these coloured precipitates, liberating iodine.

What has been known as Dragendorff's reagent for alkaloids is a potassium bismuth iodide, prepared by dissolving precipitated bismuth iodide in a concentrated solution of potassium iodide acidulated with hydrochloric acid, and known as giving reddish-coloured precipitates in solution of the salts of the alkaloids. On trial with pyridine salts, a corresponding precipitate was obtained, dark orange-red and voluminous, analogous to the piperidine compound reported by Kraut.

These organic bismuth iodides are not perfectly proof against decomposition by much water; they are sparingly soluble in ethyl or amyl alcohol, insoluble in glacial acetic acid, in ethyl ether, in chloroform, and in benzene. The tetramethyl ammonium bismuth iodide crystallises from hydrochloric acid, that of sp. gr. 1·19 diluted with an equal measure of water, also from potassium iodide solution acidulated with hydrochloric acid. The pyridine and the alkaloid bismuth iodides crystallise from alcohol. In all these cases the crystals are clearly hexagonal and easily obtained. Both the amorphous and crystalline forms are stable in the air. A sample of tetramethyl ammonium bismuth iodide remained constant in weight at 130° C.; atropine bismuth iodide melts, but at 98° C. remains constant in weight. Reducing agents, as potassium thiosulphate, do not alter these bismuth iodides.

The alkaloidal bismuth iodides are not quantitatively uniform enough to be entirely satisfactory for alkaloidal assay, but are more stable and uniform than the alkaloid mercuric iodide formed by Mayer's reagent. On the other hand, they are more bulky, less easy to gather in a compact mass, and less manageable in filtration. On the whole, so far as found, Dragendorff's reagent gives no general advantage over that of Mayer.—L. J. de W.

*Glycerin, Action of Phosphoric Acid on.* Adrian and Trillat. J. Pharm. Chim. 1898, **7**, [5], 226—230.

According to Pelouze, glycerophosphoric acid is obtained by heating glycerin with glacial phosphoric acid to 100° C., diluting, neutralising with barium carbonate, filtering, and decomposing with the calculated amount of sulphuric acid. The authors obtained by this process a white waxy substance, which they find to contain acid barium glycerophosphate. In the same way, when potassium glycerophosphate is decomposed by tartaric acid, the acid salt of potassium is the chief product. When the salts of lead or copper are precipitated by sulphuretted hydrogen, phosphoric acid is formed. According to their analysis, commercial glycerophosphoric acid is the solution of a mixture of phosphoric acid, glycerin and acid glycerophosphate, with variable amounts of glycerophosphoric acid.

Barium glycerophosphate was decomposed by a slight excess of sulphuric acid, the solution boiled, and then made neutral to Helianthin by means of potash. During the ebullition, phosphoric acid is regenerated; boiling for one hour effected the decomposition of 15 per cent. of the glycerophosphoric acid. Similar results were obtained when the solution was concentrated *in vacuo*. When calcium glycerophosphate is made according to the method of Portes and Prunier (J. Pharm. Chim. 1894, 393), the amount of glycerophosphoric acid formed, increases with the period during which heating is continued, and appears to approach a limit of about 21 per cent. (limit of etherification).

Determinations of phosphoric acid in the product of the reaction of the acid on glycerin, by gravimetric and volumetric methods, show a loss by the latter. This is to be explained by the formation of an ester neutral to indicators, $PO(OH)(C_3H_7O_3)_2$. If excess of phosphoric acid and glycerin be removed by suitable solvents, a viscous substance may be isolated, neutral to Helianthin, slowly decomposed by calcium carbonate in the cold with production of glycerophosphoric acid, decomposed on warming with water or alcohol into phosphoric and glycerophosphoric acids. This substance, on analysis, affords figures approaching those required by an ester of the formula given.—A. C. W.

### *Guaiacol, Two New Derivatives of.* S. di Boscogrande. Atti R. Accad. dei Lincei Roma, [5], **6**, (2), 306; Chem. Centr. 1898, **1**, [3], 207.

*Guaiacol picrate*, $C_6H_4(OH)OCH_3.C_6H_2(NO_2)_3OH$, is obtained from guaiacol and picric acid in boiling alcoholic solution. From boiling water, it crystallises in orange-red needles, m. pt. 80° C., which are decomposed in dispersed daylight, with separation of picric acid.

*Benzyl-guaiacol*, $C_6H_3(OH)(OCH_3).(CH_2.C_6H_5)$, is prepared by the action of benzyl chloride on guaiacol in presence of zinc shavings, hydrochloric acid being evolved. It occurs as a yellowish, beautifully fluorescent oil, b. pt. at 436 mm. pressure, 269°—270° C., not volatile with steam; sp. gr. at 13° C., 1·138.—A. S.

### *Piperidine Guaiacolate, Note on the Physiological Action of.* F. W. Tunnicliffe. J. Chem. Soc. 1898, **73** and **74**, [424], 145.

This substance is a relatively soluble salt, and is said to possess none of the irritant properties of guaiacol itself. The author considers this substance to be of importance pharmacologically, as combining the properties of a strong vascular and nervine tonic—piperidine (probably the strongest, with the exception of nicotine), with an antiseptic,—guaiacol.—A. S.

### *Theobromine, Solubility of, in Solutions of Salts with an Alkaline Reaction.* Brisse-Moret. J. Pharm. Chim. 1898, **7**, [4], 176—178.

Sodium carbonate, the bimetallic alkaline phosphates, and borax do not appreciably dissolve theobromine; but in silicate of soda and in the trimetallic phosphates, it is fairly soluble. At 15° C. a solution of 14·8 grms. of trisodium phosphate in 80 c.c. of water dissolves 3·5 grms. of the alkaloid; the same weight of phosphate in 250 c.c. of solution can dissolve 4·7 grms. This solvent action is evidently due to the sodium hydrate liberated by the dissociation of the phosphate in aqueous solution. These theobromine solutions are decomposed by the weakest acids and by salts with an acid reaction, but their stability in presence of the carbonic acid of the air is much greater than that of solutions in caustic soda or lime water; in addition, they do not possess the caustic action of the latter solutions on the mucous membrane.—A. C. W.

### *Strophanthin, The Preparation of, and the Occurrence of Choline and Trigonellin in Strophanthus Seeds.* H. Thoms. Ber. **31**, [3], 271—277.

The strophanthin met with in commerce usually varies considerably both as regards its chemical composition and as to its toxicological effects. In the present paper an endeavour has been successfully made to prepare pure strophanthin, and to isolate the impurities in the commercial article. Strophanthin is obtained from different sources, the chief of which are the seeds of the plants *Strophanthus hispidus D.C.* and *Strophanthus Kombé Oliv.*

To obtain strophanthin from these, the seeds were freed from oil, and extracted with 70 per cent. alcohol; the solution was then precipitated with tannin, avoiding an excess, the precipitate mixed with lead oxide and dried. After extracting the dry residue with alcohol, the strophanthin was precipitated by mixing the alcoholic solution with a considerable quantity of ether.

The strophanthin thus obtained was still contaminated with small quantities of nitrogenous substances, and was purified by treating its aqueous solution with finely powdered ammonium sulphate, when the substance itself was precipitated and the impurities remained in solution.

The author next proceeded to separate and identify these latter bodies. The ammonium sulphate solution, after separating the strophanthin, was strongly acidulated with sulphuric acid and precipitated with potassium-bismuth-iodide solution. The red precipitate obtained was washed with dilute sulphuric acid, then with water, and decomposed with moist silver carbonate. After removing the silver with hydrochloric acid, on evaporating the acid filtrate, a yellow crystalline residue remained, which, by treatment with cold absolute alcohol, was separated into two fractions, one soluble in alcohol and the other almost insoluble. The former was found to be choline, and the latter corresponded to trigonellin.—W. P. S.

### *Choline and Trigonellin in the Seeds of Strophanthus Kombé.* H. Thoms. Ber. 1898, **31**, [4], 404.

The author having found the above bases in the seeds of *Strophanthus hispidus* (see preceding abstract), has now investigated the seeds of *S. kombé*. The same substances are present; they were separated and identified as before described.—A. C. W.

### *Strophanthin and Strophanthidin.* F. Feist. Ber. 1898, **31**, [5], 534—541. (See also this Journal, 1898, 268.)

Products of very different composition, properties, and toxic action are found in commerce and described in the literature under the name of strophanthin. The seeds of *Strophanthus Kombé* and the strophanthin obtained from them give a green coloration with concentrated sulphuric acid. In commerce, different products are found, derived from the so-called *S. hispidus* seeds, the genuine giving the green coloration, others a brown; these contain no true strophanthin. The author used strophanthin from *S. Kombé*. It was free from nitrogen, did not reduce Fehling's solution, and was optically inactive. Strophanthin absorbs moisture from the air, and appears to form several hydrates; dried over sulphuric acid or at 105° C., it has the formula $C_{32}H_{48}O_{16}$ (deduced from ultimate analysis and molecular weight determination). On warming with acids, strophanthidin separates in needles, which may be recrystallised from ethyl or methyl alcohol; the solution, after removal of hydrochloric acid and evaporation, forms a syrup, which solidifies. This mixture of soluble decomposition products when extracted by methyl alcohol, leaves a white crystalline substance melting at 207°, having the composition $C_{13}H_{21}O_{10}$. This substance is readily soluble in water, fairly soluble in hot alcohol and acetone and almost insoluble in ether and ligroin; it reduces Fehling's solution only after long boiling, does not unite with phenylhydrazine, is not fermented by yeast, and is slightly dextro-rotatory. Oxidation produces oxalic acid, but no saccharic or mucic acid. Boiled with hydrochloric acid, no furfurol is formed, but the residue now readily reduces Fehling's solution.

The methyl-alcohol extract yields a strongly reducing syrup containing a very small quantity of dextrose, and principally consisting of a sugar of melting point 95° C., which forms no osazone, soluble with difficulty.

Strophanthidin melts at 169°—170° C. and decomposes at 176°; it has the composition $C_{26}H_{38}O_7 + 1\frac{1}{2}H_2O$, and is without action on Fehling's solution. Alkaline permanganate oxidises it to oxalic and acetic acids; chromic acid produces benzoic acid. Alkalis saponify strophanthidin with production of a substance $C_{24}H_{30}O_5 + 1\frac{1}{2}H_2O$, an acid anhydride or lactone, melting at 294° C. with decomposition; and a compound, $(C_7H_{10}O_2)x$, melting at 198·5° C., soluble in alkalis, acetone, and alcohol, insoluble in soda.

By the action of bromine on strophanthidin, a white bromide, $C_{36}H_{51}Br_5O_{10}$, melting at 126° C., and a light yellow bromide, $C_{33}H_{33}Br_{11}O_4$, melting at 160°, are formed. By the action of bromine and caustic soda, the compound $C_{39}H_{54}Br_2O_{10}$ is obtained.—A. C. W.

*Strophanthin.* L. Kohn and V. Kulisch. Ber. 1898, **31**, [5], 514—516. (See preceding abstracts.)

The seeds of *Strophanthus Kombé* or *hispidus* were carefully freed from the fibrous tufts, finely powdered, extracted with petroleum ether to free from fatty oils, dried, and exhausted with 70 per cent. alcohol. The filtered extract, after treatment with basic lead acetate and lead oxide, and removal of lead by sulphuretted hydrogen, was evaporated *in vacuo*. After several days, impure strophanthin crystallises out: 3—5 grms. were obtained from 1 kilo. of seeds. After repeated recrystallisation from water, a white microcrystalline product was obtained, neutral, and free from nitrogen. Analysis pointed to the formula $C_{13}H_{20}O_5$ or $C_{31}H_{48}O_{12}$; the results of a methoxy group determination agreed with the presence of one $OCH_3$ in the latter formula. The decomposition product of strophanthin formed by the action of mineral acids, is soluble in water, reduces Fehling's solution, but gives no reaction characteristic of a glucose. Doubt is thus thrown on the supposed glucoside nature of strophanthin.—A. C. W.

*Perfumery.* Pharm J. 1898, [1444], 197—199.

It is stated that the fractional distillation of essential oils obtained from plants, has shown that they are composed of a nearly odourless vehicle, consisting of one or more hydrocarbons of the terpene class; a principal odorous constituent which may be an alcohol, phenol, aldehyde, ketone, ether, or ester, and small quantities of various other compounds. These modifying constituents may vary even in different parts of the same plant. In the preparation of perfumes, terpeneless oils—*i.e.*, essential oils which have been deprived of their terpene by distillation *in vacuo* or by other means—are often used in preference to the natural oils, as they are more likely to be free from adulteration. The principal odorous ingredients of volatile oils, obtained in a comparatively pure state by chemical means or by freezing, may also be employed, but these do not wholly represent the desired flavour, owing to the absence of the modifying constituents present in the natural and also in the terpeneless oils.

The vehicle used in the preparation of the finer class of artificial perfumes is generally grape spirit, which is less apt to contain traces of amyl or other offensive alcohols. To imitate the perfume of a flower, the essential oil of the flower is used when possible, but when the volatile oil cannot be obtained by distillation, an artificial product is substituted (see appended list). If the odour itself is faint or easily passes off, a degree of permanence is given by the addition of a very small quantity of a persistent substance. In some cases where the leading perfume is naturally sweet, as in rose, or honeysuckle, an aromatic persistent substance is added. In the former case, the persistent odour is given by one or more of the following: ladanum, cloves, patchouli, vetivert, sandal wood, almonds, vanilla, tonka bean, verbena, ylang-ylang. In the latter case, the following substances are chiefly employed for giving the persistent sweet odour: liquid storax, musk, otto, bergamot, civet, ambergris, and ambrette or musk seed. In using the more powerful odours, such as patchouli, almonds, cloves, verbena, &c., it is best to use a dilute solution of the oil, so as to add a small quantity at a time, until the required modification of the principal odour is effected.

Directions are given, and also the necessary precautions for the examination of perfumes by means of the sense of smell.

### Principal Oils used in Perfumery.

Bergamot.—The chief constituents are linalool and acetate of linalool.

Bay (*Pimenta acris*).—Citral, methyl-chavicol, chavicol, methyl-eugenol, and eugenol.

Cassia.—Cinnamic aldehyde and acetate of cinnamyl.

Cinnamon.—Bark: Cinnamic aldehyde and eugenol. Leaves: In addition, saffrol, a larger proportion of eugenol, and benzoic acid. Root: In addition, camphor.

Citronella.—Citronellal, geraniol, and methyl-heptenone.

Clove.—Eugenol, furfural, amyl-methyl ketone.

Geranium Oil.—Indian or Turkish: Geraniol, geranyl acetate and caproate, methyl-heptenone.

Rose Geranium Oil.—Geraniol, geranyl tiglinate, and citronellol.

Lavender.—Linalool, linalyl acetate, geranyl acetate, and cineol.

Linaloe.—Linalool, geraniol, methyl-heptenone.

Lemon.—Citral and citronellal.

Neroli.—Linalool, linalyl acetate, and geranyl acetate.

Rose.—Geraniol and citronellol.

Rosemary.—Cineol, camphor, and borneol.

Sandalwood.—Santalol and santalal.

Verbena.—Geraniol, citral, and methyl-heptenone.

Ylang-Ylang.—Linalool, geraniol, and their acetic and benzoic esters, and paracresol-methyl ester. It is suggested that the odour of Russia leather, in perfumes, usually given by the empyreumatic oil of *Betula alba* could probably be obtained in a purer form by heating betulin, the crystalline principle contained in the latter.

### Synthetic Perfumes.

Anisic Aldehyde ("Aubepine").—Possesses the odour of hawthorn blossom, but requires mixing with other odours to render it agreeable.

Benzyl Cinnamate has the odour of balsam of Peru.

Bornyl Acetate has the odour of fir-trees.

Carvacrol has a thyme-like odour.

Cinnamyl Alcohol has an odour of hyacinths.

Citral (geranic aldehyde) has the odour of lemon, and is more readily soluble in alcohol than essence of lemon.

Cumarin has the odour of new-mown hay.

Eugenol has the odour of cloves.

Geraniol (rhodinol, linonol) has the odour of rose. Geranyl acetate has a lavender odour; geranyl formate is also made.

Ionone.—A ketone obtained from citral. Possesses a very powerful odour of violets.

Linalool and Linalyl Acetate have an odour between that of bergamot and French lavender. The latter is the "Bergamiol" of commerce. Linalyl formate resembles the odours of petitgrain and bergamot.

Methyl Benzoate.—The "oil of Niobe" of commerce.

Methyl Salicylate.—Artificial oil of wintergreen.

Saffrol has the odour of sassafras.

Salicylic Aldehyde (Salicylal) has the odour of meadowsweet.

Terpineol has the odour of lilac.

Vanillin has the odour of vanilla.

The synthetic oils of jasmine and neroli and artificial musk are also met with in commerce.

### Recently Introduced Natural Perfumes.

Canada Snake Root Oil.—*Asarum canadense.*

Champaca Oil (Schimmel).—*Michelia champaca* and *M. longifolia.* Aromatic odour.

Champaca Oil (Haensel).—Guaiacum wood oil, derived, according to Haensel, from *Bulnesia sarmienti*, Lorenz. Used with otto of rose to give the odour of the tea rose, &c.

Costus Oil.—*Aplotaxis auriculata.* Odour of violets.

Frejar Oil (Haensel).—Botanical source unknown.

Kuromoji Oil.—*Lindera sericea.* Pleasant spicy odour.

Ladanum Oil.—*Cistus creticus.* Very pleasant odour, somewhat like ambergris.

Nagkesar Oil (Haensel).—*Mesua ferrea.* Odour of a violet character.

Nigella Oil.—*Nigella damascena.* Odour like raspberries.

Opoponax Oil.—*Balsamodendron kafal.* Obtained from the gum resin (Bissa-bol). Somewhat aromatic odour.

Peru Balsam has the odour of balsam, but less empyreumatic.

Sandalwood Oil, African, odour of sandalwood.

Spicewood Oil.—*Laurus benzoin.* Spicy odour.

Storax Oil has the odour of liquid storax.

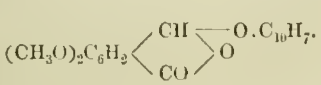
Tolu Balsam Oil, odour of hyacinths.

—A. S.

### *Rhodinol, Esters and a Crystalline Pseudo-Ester of.* E. Erdmann. Ber. 1898, **31**, [4], 356—360. (See also this Journal, 1898, 267.)

In addition to the already known rhodinol acetate, the author has prepared the butyrate, boiling at 142°—143° C. under 13 mm., the isobutyrate, at 135°—137° under 13 mm., the isovalerate, at 135°—138° under 8 mm., and the palmitate of boiling point 260° under 12 mm. The method employed was that of H. Erdmann:—action of the acid chloride upon rhodinol in the presence of anhydrous pyridine, when an addition compound of pyridine and the acid chloride results, which on warming, reacts on the alcohol with production of the ester and pyridine hydrochloride. The esters mentioned are all liquids, and do not solidify on cooling; the odour decreases with the increasing molecular weight of the acid. On warming with pyridine and diphenylurea chloride, they give no crystalline compound; free rhodinol is necessary for the formation of the diphenylurethane.

When rhodinol is heated at 130°—135° with opianic acid, a crystalline compound is formed, which melts at 48°·5, and has the composition and properties to be expected of rhodinol opianate. This ester is decomposed by boiling with water, and is probably to be represented as a pseudo-ester—



The boiling point of pure rhodinol is 110°·5—111° at 10 mm. pressure—not at 16 mm., as appears in a former communication (this Journal, 1897, 823).—A. C. W.

### *Native French Peppermint Oil.* Eugène Charabout. Bull. Soc. Chim. 1898, **20**, [3], 117—120.

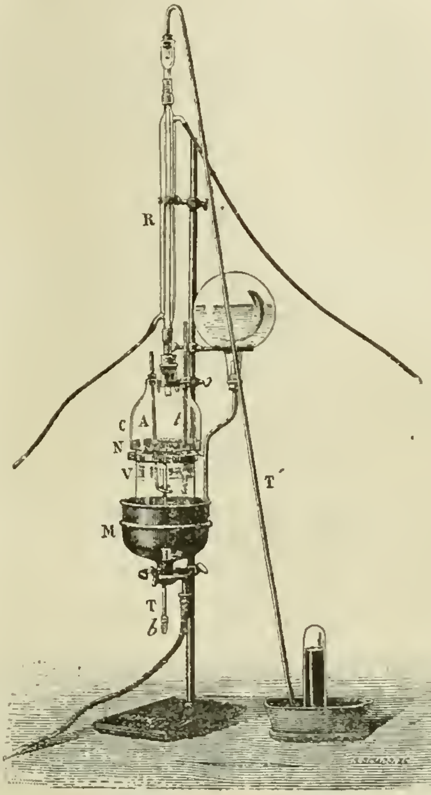
The author finds that French peppermint oil agrees in general characters and constituents with the recorded figures for English and American oil of peppermint, the most notable variation from these forms being the optical rotation. French oil examined by the author is markedly less lævogyrate than either English, American, or Japanese peppermint oils. The figures obtained are from −5° 54′ to −7° 06′ in a 100-mm. tube, the figures obtained by other workers being: for Japanese oil, −26° to −37°; American, −25° to −33°; English, −22° to −31°.

The specific gravity of French oil ranges from 0·918 to 921, the percentage of menthyl esters (acetic and isovalerianic), from 7·1 to 10; of free menthol, from 35·7 to 39·4, equivalent to total menthol from 43·7 to 46 per cent. Menthone was found present to the extent of 8·8 to 9·6 per cent. A curious modification of the inflorescence of the peppermint plant is found in the south-east of France termed by the growers "menthe basiliquée." This presents, instead of the normal inflorescence, an appearance of nodules resembling the ovaries of basilicum directly after the flower has fallen. The author finds that this is due to galls formed by the puncture of an insect. The oil derived from plants thus affected was examined and found to have the following characters, among which it will be noticed that the optical rotation differs markedly from that of normal peppermint oil, being dextrogyrate to the extent of + 7°. The specific gravity was 0·922, the percentage of menthyl esters, 8·2; of total menthol, 41; and of menthone, 3. The oil of the modified plants is also shown, on fractional distillation, to differ materially from that derived from normal healthy plants.—J. O. B.

### *Natural Organic Principles, An Apparatus for the Separation of.* C. Chabrié. Bull. Soc. Chim. 1898, **19—20**, [3], 100.

The apparatus is designed to facilitate the separation of various natural organic products according to their physical properties, between the temperatures of −20° and 100° C., or, if necessary, at other limits of temperature.

The chief advantages claimed for it are: (1) The ready means of decanting various superimposed layers of liquid, by a movable tube T, fitted in the cork B, the upper end of which tube can be adjusted successively to the lower portion of each of these liquid strata, which may be drawn off by removing the small cork *b*, closing the lower end. This method of decantation is specially serviceable in those cases where a solid body occupies the lower layer, since such separation cannot then be effected by an ordinary separating funnel, the solid particles being apt to block the tap. (2) The ready separation of supernatant liquid from a precipitate; and (3) the ease with which the precipitate itself may be collected. If necessary, the last traces of precipitate may be washed out with a jet of water, since the beaker-like vessel V is open at the top, and may be disconnected from the covering vessel C, to which it is attached by a composition or rubber collar N. (4) The advantage of operating in a closed vessel, which allows any gases evolved during the experiment, and which are given off through tube T¹, to be collected for examination. (5) The fact that all volatile matter given off at the temperature of the experiment is condensed by R. (6) The possibility of exact observation of temperature by means of the thermometer *t*, and (7) of thoroughly mixing the various bodies under treatment with the agitator A; also (8) the possibility of working at a wide range of temperature, since the copper vessel M may be used as an oil or water bath, or even as a refrigerator.—J. O. B.



### *Mercury and Bismuth Salts, Separation of.* L. Vanino and F. Treubert. Ber. **31**, [2], 129.

*See under* XXIII., *page* 379.

### *Glycerophosphates, Titration of.* Falières. Repert. Pharm. 1898, **10**, 8.

*See under* XXIII., *page* 386.

### *Spirit from Cellulose and Wood.* E. Simonsen. Zeits. für angew. Chem. 1898, [9], 195.

*See under* XVII., *page* 365.

*Chloroform, The Partial Decomposition of, in the Organism.* Desgrez and Nicloux. Comptes Rend. **126**, [10], 758.

*See under* XXIV., *page* 387.

*The Glycerophosphates, Determination of.* Adrian and Trillat. J. Pharm. Chim. 1898, **7**, [4], 163, and [5], 225.

*See under* XXIII., *page* 386.

*Aconitine, Diacetylaconitine, Benzaconine, and Aconine; Pharmacology of, Considered in relation to their Chemical Constitution.* J. Theodore Cash and Wyndham R. Dunstan. Proc. Royal Soc. 1898, **62**, [385], 338.

*See under* XXIV., *page* 387.

*Rose Oil, Testing of.* P. N. Raikow. Chem. Zeit. 1898, **22**, [17], 149.

*See under* XXIII., *page* 386.

*Lemon Oil, Adulteration of, by Means of Oil of Turpentine; New Methods for Detecting.* G. Mancuso-Lima. Staz. sperim. agric. ital. **30**, 631; Chem. Centr. 1898, **1**, [7], 477.

*See under* XXIII., *page* 382.

*Antipyrine, Action of Iodine upon. Application to the Determination of Antipyrine and of Iodine.* J. Bougalt. J. Pharm. Chim. 1898, **7**, [4], 161.

*See under* XXIII., *page* 386.

*Salol, A Reaction of.* Rev. intern. falsific. **10**, 202; Chem. Centr. 1898, **1**, [3], 222.

*See under* XXIII., *page* 382.

*Cannabinol.* T. B. Wood, W. T. N. Spivey, and T. H. Easterfield. Proc. Chem. Soc. 1898, [191], 66—67.

The authors have continued their examination of cannabinol, the toxic resinous constituent of Indian hemp (this Journal, 1896, 372). The substance boils with slight decomposition at about 400°, its absorption spectrum shows no characteristic bands and its vapour-density at the temperature of boiling sulphur corresponds with the formula $C_{18}H_{24}O_2$, already assigned to the compound.

An account is given of the reaction of cannabinol with acetic anhydride, benzoyl chloride, and phosphoric anhydride; the results indicate that one hydroxyl group is present. In the case of acetic anhydride or acetyl chloride, however, a crystalline compound melting at 75° is one of the products of the reaction. The authors assign the formula $C_{15}H_{18}O_2$ to this compound. The same compound has recently been described by Dunstan and Henry (this Journal, 1898, 269), who ascribe the formula $C_{18}H_{23}O.C_2H_3O$ to it. Reduction with hydriodic acid in sealed tubes produces a hydrocarbon, $C_{10}H_{20}$.

By long-continued boiling with or without dehydrating agents, a hydrocarbon, $C_{10}H_{16}$, is formed.

The action of fuming nitric acid upon cannabinol dissolved in cold glacial acetic acid removes one carbon atom as carbonic anhydride, and produces a red, amorphous substance which gives numbers on analysis agreeing with the formula $C_{17}H_{20}N_2O_6$. This substance, when boiled with nitric acid, yields a light red substance, $C_{17}H_{20}N_2O_8$, which upon further oxidation yields, amongst other substances, a yellow acid crystalline compound, $C_{13}H_{15}N_2O_9$, which is probably a dinitrophenol, and a compound, $C_{11}H_{11}NO_6$, the properties of which agree closely with those of the "oxycannabin" of Bolas and Francis (Chem. News, 1871, **24**, 77). This compound has the properties of a nitro-lactone, as has been already shown by Dunstan and Henry (this Journal, *loc. cit.*). The name cannabinic acid is proposed for the unnitrated parent oxy acid.

*Thorium, Contributions to the Chemistry of.* Bohuslav Brauner. Proc. Chem. Soc. 1898, [191], 67—68.

As there are few direct methods of separating the elements of the rare earths, the author investigated the reaction of Bahr (1864), who showed that thorium oxalate is easily soluble in a solution of ammonium oxalate. Bunsen (1876), though he found that the oxalates of the other rare earths are only slightly soluble in that reagent, had to repeat the process of solution many times to obtain a product which did not show in its spark spectrum the lines of other elements.

The reaction is due to the formation of a double thorium-ammonium oxalate, decomposed by water, and existing in solution only in the presence of free ammonium oxalate. After numerous crystallisations, a salt was obtained having the formula $Th(C_2O_4)_2 + 2(NH_4)C_2O_4 + 4H_2O$, with one-third to one-half of free $(NH_4)_2C_2O_4 + H_2O$. Finding, however, that the solution readily becomes supersaturated, a compound having the formula $Th(C_2O_4)_2 + 2(NH_4)C_2O_4 + 7H_2O$ was obtained in a high state of purity.

This "complex" salt is ammonium thoroxalate. It is decomposed by water, but a definite quantity of the soluble product of decomposition dissolves the insoluble constituent, so that one part of water keeps one part of ammonium thoroxalate in solution if an additional half-molecule of free ammonium oxalate be present. The salt forms two hydrates, in accordance with Potilizin's law that a compound forming supersaturated solutions exists in several hydrate forms, one with $7H_2O$, the other with $4H_2O$. The former loses water in air of average humidity, passing into the latter form, which becomes anhydrous in perfectly dry air, or more readily at 100°, without loss of ammonia. On heating at higher temperatures, large quantities of cyanogen gas are evolved.

The amount of the decomposition of the salt caused by increasing the quantity of water was determined, together with the proportion of ammonium oxalate required to prevent decomposition. Three mols. of ammonium oxalate form, with one mol. of thorium oxalate, a clear solution in 300 mols. of water. This solution is, however, supersaturated with thorium oxalate, the latter separating until their relative proportions become 3·3 : 1. A convenient method for the purification of thorium may be based on these results, as the author has determined quantitatively the great difference in solubility between the oxalate of the feebly basic tetravalent thoria and those of the other trivalent rare earths in ammonium oxalate. The rule was thus established: "the tendency to form complex oxalates is inversely proportional to the basicity of an earth." This, and another property of oxalates, that their stability under the oxidising action of nitric acid decreases considerably with an increasing basicity of the earth, was utilised for the purification of thorium.

On precipitating a solution of ammonium thoroxalate with oxalic acid, an acid thorium oxalate—thoroxalate of thorium and hydrogen—is formed, having the formula $H_2Th_2(C_2O_4)_5 + 9H_2O$. On using mineral acids (Glaser, this Journal, 1897, 468—469, and Zeits. Anal. Chem. 1898, **37**, 25), a different result is observed.

*Hydrogen Cyanide, Anhydrous, and Carbon Monoxide, Preparation of.* John Wade and Laurence C. Panting. Proc. Chem. Soc. 1898, [190], 49—50.

On allowing a cold mixture of equal volumes of sulphuric acid and water to drop on to 98 per cent. "lump" potassium cyanide, hydrogen cyanide, accompanied by traces of water only, is evolved in almost theoretical amount, and with the aid of suitable condensing apparatus is readily collected in quantity. With concentrated sulphuric acid, on the other hand, but still at the ordinary temperature, nearly pure carbon monoxide is evolved, also in quantity approaching the theoretical; provided certain precautions are taken, it is free from dioxide, and is accompanied only by small quantities of hydrogen cyanide.

In discussing the formation of the monoxide, experiments are described showing that part of the water required for the hydrolysis is derived either from the sulphuric acid itself or from the potassium hydrogen sulphate formed in the course of the action, and that consequently the sulphuric acid acts at the same instant both as a hydrolyst and as a dehydrating agent.

## PATENTS.

*Alkylated Dichloroxypurines, Method of Preparing.* C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 8915, April 7, 1897. (See also this Journal, 1898, 180.)

The manufacture of alkylated dichloroxypurines by the action of a suitable ester upon a salt of dichloroxypurine is claimed. Either the acid or normal salt may be used in the dry state or in aqueous or alcoholic solution.—A. C. W.

*1-Phenyl-2-Alkyl-Pyrazolones, Manufacture of.* Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 9268, April 12, 1897.

The manufacture of 1-phenyl-2.3-dimethyl-5-pyrazolone and 1-phenyl-2-ethyl-3-methyl-5-pyrazolone by heating 1-phenyl-3-methyl-5-methoxy-pyrazole or 1-phenyl-3-methyl-5-ethoxy-pyrazole respectively above 200° C., extracting with water, and removing the pyrazolone from the aqueous solution with benzene, is claimed.—A. G. W.

*Formaldehyde and Tannin, Impts. in the Manufacture of Products of Condensation of.* E. H. C. Dürkopf, Darmstadt, Germany. Eng. Pat. 6790, March 15, 1897.

Methylene-ditannic acids have been claimed by the patentee in Eng. Pats., 1896, 816, and 1897, 6686, (this Journal, 1896, 917, and 1898, 178). There are now claimed new products of condensation obtained by the action of excess of paraformaldehyde or solution of aldehyde on tannins. $C_{14}H_{10}O_9 + 2CH_2O = C_{15}H_{12}O_9 + H_2CO_2$.

—A. C. W.

*Preparations for Producing Local Anæsthesia, Impts. in.* [*Cocaine and Ethyl Chloride.*] La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 10,594, April 28, 1897.

The claims are for solutions of local anæsthetics in liquids boiling below 30° C. Solutions of cocaine in such liquids, in ethyl and methyl chloride, or a mixture of these chlorides, are specified.—A. C. W.

*Medicinal Preparations.* [*Glycerin-phenol Esters.*] S. T. K. Endemann, Brooklyn, U.S.A. Eng. Pat. 30,625, Dec. 28, 1897.

The process is claimed for producing esters of glycerin and phenols or their derivatives, which consists in heating the sodium phenolate with monochlorhydrin at 140°—150° C. The substances produced, are claimed as new products. Bodies formed as described, are said to possess a uniform bitter taste, and a specific flavour denoting their origin.

—A. C. W.

## XXI.—PHOTOGRAPHY.

*Silver Bromide - Gelatin, Action of Formaldehyde on.* M. Heilbronner. Phot. Arch. **38**, 166—168; Chem. Centr. 1898, **1**, [4], 289.

It has been frequently represented that the hardening with formaldehyde causes the formation of a coloured film on the plates. The author immersed unexposed and exposed plates, before and after developing, for six minutes in commercial formalin solution, then washed with water and developed in 4 per cent. "Rodinal" solution for 2 minutes, finally leaving for 10 minutes in the fixing-bath. Before development, the developer, as well as the sodium thiosulphate, penetrates into the film with great difficulty, but the impregnation proceeds quite normally, so soon as the negative is first fixed and then hardened. This is the case with both unexposed and exposed plates, as well as when different developers are employed. The plates bathed in formalin before development showed a peculiar net-like structure on the film, that could not be removed by mechanical means, just as reduced silver, that could not be reduced by washing, was noticeable on the gelatin. It is stated that the formation of a coloured film on the plate, through the hardening with formaldehyde, would occur if the latter entered into chemical combination with the developer, but the latter is not the case with "Rodinol" or "Diphenal," since a mixture of formaldehyde with these substances develops the picture, although somewhat slowly. The assumption is confirmed, therefore, that the formation of a coloured film may be traced back to the known reasons of the different degrees of oxidation of the developer during development.—A. S.

*"Ortol": A New Photographic Developer.* C. H. Bothamley. Camera Club J., 1898, **12**, 28.

"Ortol" (Methylorthoamidophenol) (Hauff) makes a very useful photographic developer, yielding negatives much of the same character as those obtained with pyrogallol. Since it only turns brown on exposure to air very slowly, a number of plates can be developed with the same quantity of solution; and neither the negatives nor the fingers are stained, even after prolonged action. A suitable formula is:—(I.) Ortol, 15; potassium "metabisulphite," 7·5; water, 1,000. (II.) Sodium carbonate, 100; sulphite, 100; potassium bromide, 2·25; water, 1,000; equal parts of each to be taken. Ortol can also be employed for intensification, the negative being previously bleached with mercuric chloride in the usual manner; but for this purpose it is better to omit the sulphite. As the intensified image apparently consists of a mixture of metallic mercury and silver, the whole operation may be repeated if necessary.

—F. H. L.

*Photo-Ceramics in Three Colours.* P. C. Duchochois. Anthony's Bull.; through Scient. Amer. Suppl. 1898, **45**, 18,439.

This is a combination of the "three-colour" process used in producing coloured photographic prints with the "dusting-on" process. The three original negatives are prepared on ordinary "ortho-chromatic" plates (not containing any iodide), which are best specially sensitised for red, yellow, and blue respectively by the operator himself, before exposure. The screen for the blue negative is a reddish-orange, absorbing (in the spectroscope) the bands between H and E$\frac{3}{4}$D, and suppressing the ultra-violet rays. For the red negative, the screen is a greenish green-yellow, absorbing the light from A to C$\frac{3}{4}$D. The screen for the yellow negative is a bluish blue-violet, absorbing the light between E and D$\frac{1}{4}$C. From these negatives, which ought to be full of detail but free from fog, diapositives are made in collodion or on transparency gelatin, and they should be clear and transparent with about the density of a lantern slide. In order that they may be printed from in a reversed position so as to give correctly placed enamels, and also that they may be applied to slightly curved surfaces, they must be stripped of their temporary rigid supports and used as films.

The hygroscopic compound consists of glucose, liquid, 50 minims; best gum arabic, 25 grains; saturated solution of ammonium bichromate, 2 drachms; water, 1 oz. After being well filtered, this will keep in good condition in the dark for a week or so; but it is better to use it within 48 hours of preparation. The glass plate, plaque, &c., is thoroughly cleaned, coated (on a slow whirling table for large articles) with the sensitive liquid, allowed to drain for a few seconds, then dried at 43° C. to prevent crystallisation; the whole operation being conducted in a yellow light. Whilst still warm, it is exposed under one of the diapositives for about three or five minutes in bright diffused light; then (in daylight) allowed to absorb moisture for two minutes, and dusted with the corresponding enamel colour by means of a pepper-box. The powder is gently rubbed over the surface, and more is applied at intervals as the film becomes damper. When the details are fully "developed," the plate is heated to remove moisture, the superfluous pigment carefully brushed off, and the image "fixed" (*i.e.*, the excess of bichromate eliminated) by immersion in two successive baths of fused borax, 125 grains; alcohol, 12 oz.; water, 4 oz. The plaque is dried, burnt, coated again, exposed under the next diapositive, &c., again burnt, and the cycle of operations repeated; but the order in which the colours are employed, makes no appreciable difference. It is important, however, that the three exposures shall be equivalent in effect, or the final enamel will not have its tints properly balanced; that is to say, will not be true to the original. It is also necessary that the films shall be most accurately superposed; methods of ensuring this, as well as for manipulating highly convex or concave objects, are fully described in the article.

The translucent enamel colours found in the market are quite suitable, but they often require further grinding. Their fusibility, however, must be considered with reference to the material of which the plaque is constructed; glass needs the most fusible colours; enamel plates the least. After the third proof has been burnt in, it is sometimes advisable to add a fourth in monochrome—bistre, neutral tint, &c.—or the picture may be touched up by hand.
—F. H. L.

*Aldehydes and Ketones, A Photographic Reaction of.* Lumière Bros. and Seyewetz. Bull. Soc. Chim. 1898, **19**, 134.

*See under* XXIII., *page* 380.

### PATENTS.

*Leather for Photographic Purposes, Manufacture of Prepared Sensitised.* O. Imray, London. From Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main. Eng. Pat. 6769, March 15, 1897.

As ordinary leather decomposes gold and silver salts, it is impossible to produce photographic prints directly on it; but if the material be saturated on both sides with a 0·5 or 1·0 per cent. solution of plain collodion, after 24 hours it may be coated with sensitive emulsion and treated in the same way as glass plates.—F. H. L.

*Photographic Developers.* J. Hauff, Feuerbach, Germany. Eng. Pat. 28,596, Dec. 3, 1897.

The inventor claims the dialkylglycines of paraphenylene (tolylene or xylylene)-diamine and of $\alpha$—$\alpha$ and $\alpha$—$\beta$-naphthylenediamine as photographic developers. These bodies may be made into very concentrated solutions which keep better than any other developer, reduce the image rapidly in presence of sulphite, alkali carbonates or hydroxides, do not stain the gelatin, and give a fine grain similar to that of wet collodion plates. A suitable formula is: dimethyl-*p*-amidophenylglycine, 1 grm.; sodium sulphite, 5 grms.; "potash," 5 grms.; water, 100 c.c.
—F. H. L.

*Applying Colours to Photographs, Improved Process of.* V. I. Feeny, London. From F. von Bentivegni, San Francisco, U.S.A. Eng. Pat. 30,115, Dec. 20, 1897.

This is a process for colouring photographic silver prints by fixing them face downwards upon a sheet of glass, sandpapering away the superfluous paper, making the picture translucent by immersion in a bath of wax, and applying some of the colours to the back of the picture and others to a second sheet of glass fastened behind. The novelty appears to consist in the composition of the waxing solution, which is as follows:—Refined castor oil, 1 pint; white vaselin, 1 dessert-spoonful; spermaceti, 1 teaspoonful; rosemary, 20 drops; citronella, 20 drops; heated together to about 60° C., and coloured light red with alkanet root.—F. H. L.

*Photographs by Artificial Light, Flash Powder for the Taking of.* A. Weiss, Strassburg. Eng. Pat. 1126, Jan. 11, 1898.

Powdered aluminium emits a more actinic light than magnesium, but the compositions hitherto employed have proved dangerous to handle. The inventor suggests a mixture of 15 to 60 parts of aluminium with 85 to 40 of potassium perchlorate—the latter proportions being most generally suitable for ordinary photographic purposes.
—F. H. L.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Higher Nitrocelluloses.* C. Hoitsema. Zeits. angew. Chem. 1898, 173—174.

The nitrogen content of the highest nitrocellulose hitherto prepared corresponds nearly with 14·14 per cent., *viz.*, that of the trinitrate, $C_6H_7O_2(NO_3)_3$. The author's attempts to prepare a higher nitrocellulose by the action of nitric acid on gun-cotton in presence of phosphorus pentoxide, sulphur trioxide, or sulphuric acid, failed. A product which contained 14 per cent. of nitrogen was prepared by treating gun-cotton with the mother-liquor left after separating crystals of nitrogen pentoxide from the distillate obtained by heating a mixture of nitric acid and phosphorus pentoxide. By the action of the crystals themselves and of the residue left in the retort, products containing 13·9 and 13·8 per cent. of nitrogen respectively, were obtained.
—E. W. W.

*Nitro-Explosives, The Manufacture of.* A. Prister. Proc. Chem. and Metall. Soc., S. Africa, 1898, 2—12.

This paper gives a detailed account of the ordinary methods of manufacturing dynamite and blasting gelatin, and refers more especially to their manufacture and use on the Rand. The admitted annual consumption of dynamite in the South African Republic is 6,250 tons, which, according to figures published by the Chamber of Mines in 1896, may be classified into blasting gelatin, 4,500 tons; dynamite No. 1, 1,250 tons; gelignite gelatin 1 A. and dynamite No. 2, 500 tons. This would correspond to 5,327·5 tons of nitro-glycerin, for which 2,660 tons of glycerin or 28,535 tons of fat would be necessary—an amount of raw material which it would be difficult to obtain in South Africa. The glycerin is therefore chiefly imported from Europe. For the indicated amount of dynamite No. 1, 313 tons of kieselguhr would be required. This is found in the district of Ermelo, Transvaal. Owing to the hardness of the ores, the most powerful explosives must be used, and blasting gelatin is much more used than dynamite No. 1.

The nitric acid used in the manufacture is prepared from sodium nitrate, all of which is imported from Chili, no such mineral having been discovered in South Africa. The deposits of potassium nitrate at Prieska, Cape Colony, and in the Marico district, are stated to be of no commercial importance. The company floated to work the former is now in liquidation, whilst the Marico deposit was found to contain so little nitre (only 2 per cent.) that it did not pay for the working. Sulphur for the sulphuric acid is imported from Sicily and England. Experiments have been made with the object of utilising native pyrites, but owing to a variety of causes, which the author does not specify, these have not been carried out successfully on a manufacturing scale. The sulphuric acid factory is said to work as well on the Rand as in Europe, but owing to the greater altitude (5,735 ft. above sea level), the chambers have to be made 25 per cent. larger in order to obtain the same results.

Referring to the dangers accompanying the use of dynamite, the author states that freezing rarely occurs on the Rand, though it may occasionally happen in winter. The recent mining disaster at Langlaate is attributed to incomplete explosion of dynamite, the workmen being suffocated by fumes from the burning residue. Nitrogen peroxide is one of the products of burning dynamite, and this has recently been found by German chemists to be as deadly as hydrocyanic acid.—C. A. M.

*Perchromic Acid, Alkali Salts of.* O. F. Wiede. Ber. **31**, [5]. 516.

*See under* VII., *page* 318.

### PATENTS.

*Explosives, Impts. in or relating to.* W. Greaves and E. M. Hann, Aberaman, Glamorgan. Eng. Pat. 9111, April 9, 1897.

The object is to produce a blasting powder safe for use in dusty and fiery mines. The invention consists in substituting for the whole or part of the sulphur, oxalic acid or the oxalates, acid or normal, of sodium, potassium or ammonium. "If desired, for the sulphur may be substituted borax or boric acid alone, or mixed with one or more of the salts mentioned above."—W. M.

*Explosives, Impts. in.* T. Trevlev, Kisslovodsk, Terek, Northern Caucasus. Eng. Pat. 9535, April 14, 1897.

This invention relates to an explosive consisting of two distinct portions—a solid powder consisting of potassium chlorate, ferric oxide and manganese oxide, and a liquid consisting of petroleum and oil of turpentine, almond oil being added in some cases. The solid mixture is filled into cartridges of convenient size made of paper easily permeable by the liquid, and 10 or 15 minutes previously to charging the bore holes, the cartridges are impregnated with the liquid; the mass is then charged into the holes in a similar manner to dynamite.—W. M.

*Blasting Compound, Improved, and Process for its Manufacture.* F. Müller, S. Oberländer, V. H. Fuchs, and S. Gomperz, all of Vienna. Eng. Pat. 6772, March 15, 1897.

"This invention relates to a blasting compound consisting mainly of nitrated tar products, in particular nitrated carbolic acid, with addition of sulphur, potassium nitrate, and cellulose." The compound is prepared by first incorporating with 10 parts of powdered sulphur, 12 parts of crude carbolic acid, so that a semi-pasty mass is prepared. Separately, 18 parts of nitric acid, 40° B., are mixed with 40 parts of $KNO_3$. Both preparations are then intimately mixed, and after completion of the reaction the compound is neutralised by means of 3 parts of sodium carbonate, 7 parts of wood powder being also added. The finished product is made into cartridges, which are dried at 35° C. —W. M.

*Explosive Propelling Compounds, Impts. in, and in Processes and Apparatus for their Manufacture.* Hudson Maxim, London. Eng. Pat. 7178, March 19, 1897.

The object of this invention is to produce an explosive in form of a multi-perforated body, the perforations being so shaped and arranged as to present the minimum of burning surface to the flame of ignition when fired in a gun, and be consumed with a constant increase of burning area. To carry out the invention, a colloid of guncotton or compound of trinitrocellulose, gelatin-pyroxilin, and nitroglycerin (Eng. Pat. 16,311, 1895) is perforated with angular pins or mandrils, preferably either hexagonal or three-sided. Six systems of perforation are pointed out, whereby, when a powder is consumed by combustion within the perforations, the total consumption will be completed at the instant of interjunction of the perforations, and when the perforations have attained their largest size. The author finds that an effective method of removing solvent from a compound consisting mainly or wholly of guncotton, is to place the material in a suitable vessel, from which the air is preferably exhausted, charged with the vapour of a volatile solvent, such as acetone. The explosive material is then heated above the boiling-point of the solvent, which evaporates from the interior more readily, since the formation of a horny exterior layer is prevented by the vapour. —W. M.

*Explosives, Impts. in or relating to.* E. A. G. Street, Paris. Eng. Pat. 9970, March 29, 1897.

This invention relates to an explosive produced by dissolving a suitable nitro derivative or combination of nitro compounds in animal or vegetable oils or fats at a temperature of from 40° to 60° C. It is found that by rapidly cooling and stirring, a thick pasty mass is obtained, varying in consistency according to the proportion of oil and nitro-compound, which can be mixed with chlorate of potash alone, or together with "hydrocarbonated or carbonated substances, such as starch." The mixture gives a plastic explosive easily moulded.—W. M.

*Guncotton, Treating; Impts. in and relating to a Process of, and Apparatus for.* F. G. Du Pont, Wilmington, Delaware, U.S.A. Eng. Pat. 15,693, July 1, 1897.

The object of this invention is to obviate the necessity for drying guncotton previously to treating it with the solvents, which turn it into forms convenient for the arts. The author takes advantage of "percolation" to replace the water in guncotton by any chemical, not a solvent of guncotton, which can be used in the process to which the guncotton is to be subjected. In the manufacture of smokeless powder, where alcohol and ether are to be mixed with the guncotton, the water is displaced by alcohol without at any stage in the displacement drying the guncotton, the ether being afterwards mixed into the alcoholised guncotton, thereby gaining an advantage by reason of the affinity which ether has for alcohol, for the ether penetrates the mass of alcoholised guncotton much more readily than alcohol and ether do when mixed together with dry guncotton. For a description of the special press to be used, the specification must be consulted.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

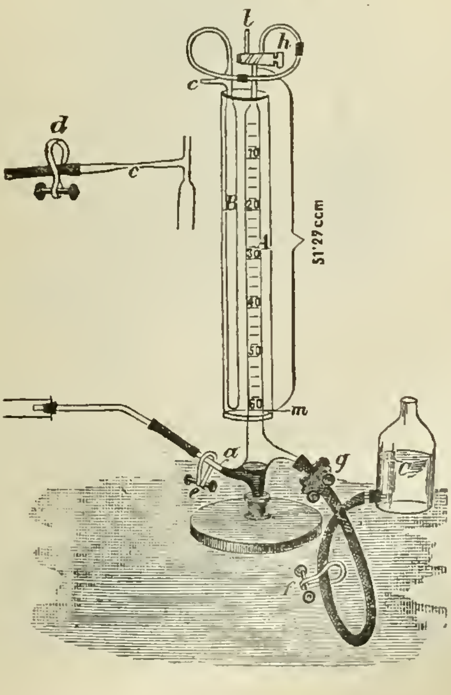
### *APPARATUS, ETC.*

*Absolute Gas Measurement, New Method of.* [*Direct Measurement of Reduced Gas Volumes.*] O. Bleier. Ber. 1897, **30**, [19], 3123—3131. (Compare this Journal, 1896, 294, 616, 672; 1897, 456, 634; 1898, 272.)

Fig. 1 shows the apparatus, which is specially designed for the estimation of nitrogen in ultimate organic analysis. The burette A, which, along with the compensator B, is enclosed in a water-jacket, is graduated to contain 51·37 c.c. between the three-way tap *h* and the mark *m*. 51·57 c.c. represents the volume of 60 mgrms. of nitrogen at 18° C., and under a pressure of 760 mm. This volume is therefore divided into 60 equal parts, which are further subdivided into fifths. If the apparatus is to be used for purposes other than the estimation of nitrogen, the burette must also be graduated in cubic centimetres.

The compensating tube B contains a few drops of 40 per cent. caustic potash, and is filled with dry air at 18° C. and 760 mm. The filling of the tube is accomplished in the following way:—Before the lateral tube *c* is finally sealed up, the burette is nearly filled with air by raising or lowering the reservoir C, containing 40 per cent. KOH. The *exact* volume of enclosed air at the prevailing temperature and pressure is then ascertained. Next, the volume which this

Fig. 1.

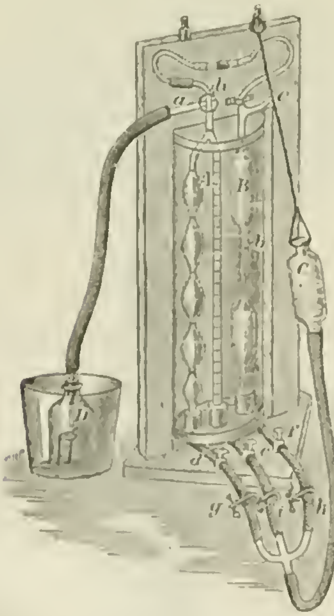


quantity of air, saturated with the aqueous vapour from 40 per cent. KOH, would occupy, if its temperature were 18° and its pressure 760 mm., is calculated in the ordinary way. By raising or lowering the reservoir C, the enclosed air is then compressed or expanded, as the case may be, exactly to the calculated volume. The capillary tube, which forms the communication between the burette and the compensating tube, contains a drop of KOH solution, to indicate whether the pressures in both tubes are the same. (Other liquids, coloured if necessary, may be employed.) By blowing or sucking at *d*, the pressure in B is adjusted, with the help of the differential manometer, until it is equal to that in A, when *c* is sealed off. The position of rest of the index is finally determined by introducing a new volume of air into the burette, and again bringing it to the calculated volume. When this is done, the apparatus is ready for use. Nitrogen from the combustion tube is admitted in the usual way at *a*. When the evolution of gas has ceased, *a* is closed by its nipper tap *e*, and, after waiting 20—25 minutes for the temperature to become uniform, the reservoir C is raised until, on opening the tap *h*, the index remains approximately at zero. *f* is then closed, and the fine adjustment made with *g*. The observed reading of the burette now represents the weight of the nitrogen in mgrms. and tenths of mgrms.

For general gas analysis the author recommends another apparatus, shown in Fig. 2. The measuring vessel A and the compensator B are similar to those already described (this Journal, 1898, 272—273), B being filled to the mark *b*, with moist air at 18° and 760 mm.

(1.) *Manipulation of the Apparatus in Absolute Gas Measurements.*—As an example the estimation of hydrogen peroxide is given. As usual, D contains the measured quantity of the peroxide solution, whilst the inner vessel contains the acidified permanganate solution. Before beginning the evolution of the oxygen, the pressure in B is first made and kept equal to that prevailing in D. The oxygen is then liberated and collected in A, when the pressure common to A and D is, by raising or lowering the mercury reservoir C, adjusted until it is equal to that in B. The gas contained in the measuring vessel A now represents the quantity of oxygen liberated, and in order to read it off, A and B only are placed in communication with each other. The level of the mercury meniscus in B is brought to the mark *b*, and the pressure in A equalised to that in B. The reading is now taken, and each c.c. of oxygen liberated (18° and 760 mm.) represents 0·00142 grm. of hydrogen peroxide.

Fig. 2.



(2.) *Manipulation of the Apparatus in relative Gas Measurements (ordinary Gas Analysis).*—100 c.c. (or 100 parts) of the gas to be analysed are introduced and the analysis conducted in the ordinary way. The introduction of 100 volumes of the gas may be accomplished in three different ways.

(*a*) The pressure in the compensator is made equal to that of the external atmosphere. A slight excess of gas is then sucked into the measurer, the meniscus adjusted exactly at 100, and the tap *h* opened for an instant to allow the excess to escape.

(*b*) About 100 c.c. of the gas may be sucked into A, which is then placed in communication with B. The meniscus in A is now adjusted to 100, whilst that in B is varied until the pressures in A and B are equalised.

(*c*) The level of the mercury in B may be adjusted to the mark *b*, whilst a slight excess of gas is introduced into A. After setting the meniscus in A exactly at 100, the excess of gas is carefully allowed to escape until the pressure in A is identical with that in B.

In each case the compensator must not be altered after the first measurement. The results are obtained directly in percentage volumes. In the third instance the measurements are absolute, but this, in general, is of no consequence in gas analysis.—J. S.

*Gas Analysis Apparatus.* O. Bleier. Ber. **31**, [3], 236—239. (See also this Journal, 1896, 294, 615, and 672; also 1897, 466 and 631, and 1898, 272.)

The principle of the author's compensation method is to measure the volume of a quantity of air which, under the same conditions of temperature and humidity, exerts the same pressure as the gas in question. After every absorption, the gas is brought back to its original volume, or to a volume which bears a definite ratio to the original volume. A two-limbed pipette A—B (Fig. 1) which holds the gas, communicates by means of a differential manometer with the compensator C. Each of the five small bulbs of A occupies 10 per cent., each of the nine bulbs of B, 1 per cent., and the big bulb of air with the capillaries above, 41 per cent. of the total volume A + B (= 100 parts). The upper part of C occupies 100 parts, and the graduated scale 2·5 parts. At the beginning of the determination, the gas in A + B, and the air in C, are at atmospheric pressure. After some of the gas has been absorbed, and the remainder brought back to the original volume, the pressure in A + B is reduced, and, in order to restore equilibrium in the manometer, the air in C is allowed to expand, the amount of expansion being read off on the scale.

Fig. 1.

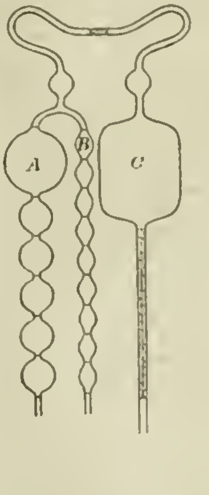
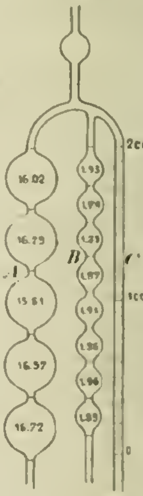


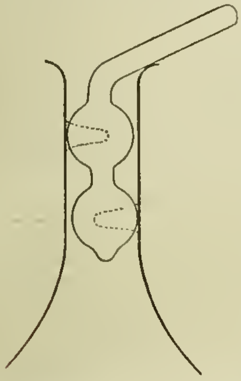
Fig. 2.



For the accurate determination of small volumes, a three-limbed apparatus (Fig. 2) is recommended. In this case the gas itself is measured. The volumes of the bulbs are marked upon them; they do not form any aliquot part of the volume of the whole. Each bulb of B occupies less than the graduated scale C, and the volume of each bulb of A is less than that of the two limbs B and C together. The volume A + B + C = 98·48 c.c., so that a space = 1·52 c.c. must be left between the highest marks on the limbs and the stop-cock above, in order that the whole may contain 100 c.c.

The measurement is effected by bringing the confining liquid up to the appropriate marks on A and B, maintaining it there by means of spring-clips fixed on the tubing which connects the apparatus with the level-bottle, and noting the height of the liquid in C. The volume of the gas is the sum of the volumes observed in the three limbs.
—E. H. T.

*Spurting, Device to Prevent Loss from.* A. H. Low. J. Amer. Chem. Soc. **20**, (3), 233—234.



This is effected by means of the arrangement shown in the accompanying figure. It consists of a piece of glass tubing, on which are blown two bulbs, fitting quite loosely in the neck of the flask. The escaping vapours condense on these, forming liquid joints on opposite sides, whilst no obstruction is offered to the escape of gases. The exterior portion of the tube projects from the neck of the flask at such an angle that any vapours condensed upon it fall back into the flask. It is stated that this device is specially useful in preventing bumping of sulphuric acid in copper assays.—C. A. M.

*Chimney Smoke, Colorimetric Determination of Density of.* P. Fritzsche. Zeits. Anal. Chem. **37**, [2], 92—94.

Into a glass tube, of about 10 mm. internal diameter and 150 mm. length, insert a plug of 2 grms. of loose cellulose (nitrocellulose), and connect the tube by means of a short piece of rubber tubing with another tube of similar diameter, and of such length that it projects a short way into the chimney when introduced through a hole in the wall. Connect the other end of the tube with an aspirator, and suck a measured quantity of 10—20 litres of the chimney gases through the cellulose. Disconnect the tubes, transfer the upper blackened layer of the cellulose by means of forceps to a 300-c.c. stoppered bottle, with a wide mouth, and rub out the tubes well with the remaining cellulose. Put the latter also into the bottle, which now contains all the soot, add 200 c.c. of water, and shake energetically till a uniformly grey pulp is obtained. To estimate the amount of soot present, pour the pulp into a round-bottomed test tube of 40—50 mm. diameter, and compare the colour with the colours of a scale prepared as follows:—Into several flasks, each containing 2 grms. of the nitrated cellulose, put 5, 10, 15, 20, 25, and 30 mgrms. of soot respectively, add 200 c.c. of water, mix thoroughly, and compare the shades with discs of paper which have previously been tinted to different degrees with Indian ink, and allowed to dry completely. These discs are pasted on cardboard, and the amounts of soot to which they correspond are recorded.—H. B.

*Glassware for Chemical Purposes, Judging.* F. Förster. Zeits. für Instrumentenkunde, **13**, 457; Dingler's Polyt. J. **307**, [7], 165.

*See under* VIII., *page* 351.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Carbon Monoxide. Determination of Traces of, in Gases.* M. Nicloux. Comptes rend. **126**, [10], 746—749.

The gas (1 litre; 2 or 3 litres as a maximum) is aspirated through a series of U-tubes, containing respectively solid caustic potash, sulphuric acid, pumice, and iodic acid. The last tube is immersed in an oil-bath kept at 150° C., and communicates in turn with an absorption tube containing 5 c.c. of caustic soda solution of density 1·3 mixed with 5 c.c. of water. The first two tubes serve to remove any carbon dioxide, sulphur dioxide, or hydrogen sulphide, and to dry the gas. Any carbon monoxide is oxidised by the hot iodic acid, according to the equation—

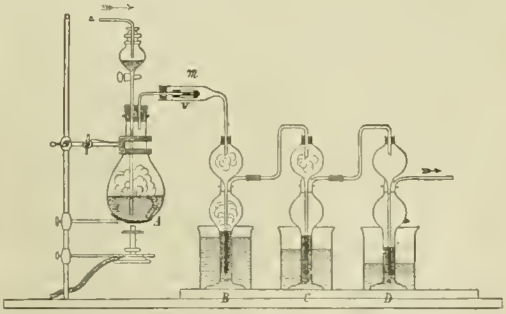
$$5CO + 2HIO_3 = 5CO_2 + H_2O + I_2$$

and the iodine vapours are retained by the caustic soda, the last traces being carried through the apparatus by the aspiration of 1 or 2 litres of air. The soda solution is acidified by sulphuric acid, nitrous acid added to liberate the iodine, which is then dissolved by vigorous shaking with a known amount of carbon bisulphide, and estimated by comparing the tint of the solution with those of corresponding solutions made from known amounts of potassium iodide. The iodic acid tube must be sealed after filling, and blank experiments must be made with the apparatus beforehand, to ensure the absence of organic matters which might liberate iodine. Carbon monoxide forming from 1/1000 to 1/50000 of the volume of the gas to be analysed can be thus determined, with a maximum error of 10 per cent.—J. T. D.

*Phosphorus, Estimation of, in Phosphides.* L. Frank. Zeits. anal. Chem. **37**, [3], 173—176.

The method is based on the decomposition of the phosphide (*e.g.*, aluminium phosphide), and the oxidation of the evolved hydrogen phosphide to phosphoric acid by bromine.

The apparatus consists of a generating flask A, and three absorption vessels B, C, D, filled with 20, 20, and 15 c.c. of a saturated solution of bromine in hydrochloric acid. A Bunsen valve V is introduced between A and B to prevent the absorbent liquid being drawn back into the generator, and provision is made for the passage of a current of carbon dioxide through the apparatus during the operation, the gas feed-pipe being connected with the drop funnel *a*. Dilute sulphuric acid (1 : 2) is employed as solvent, and a blank experiment must be made to test the purity of the reagents.



The freshly-prepared phosphide (about 0·37 grm.) is weighed in a dry, stoppered bottle, which, after the air has been expelled from the apparatus by carbon dioxide, is lowered into the flask, and the current resumed for some time, whereupon 50 c.c. of water, and 50 c.c. of the dilute sulphuric acid, are run in—the latter gradually—through the drop funnel. After half an hour's repose, and the evolution of gas has ceased, the flask A is heated to boiling for half an hour, and the current of carbon dioxide subsequently resumed for an equal period. The contents of B, C, and D are evaporated to dryness, the residue taken up with water, and the phosphoric acid determined by the molybdic-magnesia method.—C. S.

*Phosphorus, Rapid Estimation of.* O. Herting. Stahl u. Eisen, 1897, 1005; Chem. Centr. 1898, **1**, [3], 219.

The author refers to the method given by Widowiszewski (this Journal, 1898, 184), and states that he prefers the process accurately described by himself in a previous article (Chem. Zeit. 1897, [16], 138). This process depends on the titration of the molybdic acid in Sonnenschein's precipitate by means of a standardised permanganate solution. The method is accurate, and the time required for its execution is 65 minutes for pig iron, and 45 minutes for steel and "puddled iron." The following facts are given as the result of the author's personal experience:—(1) For dissolving the iron or steel, nitric acid of 1·135 sp. gr. is most suitable. (2) A saving of time can be effected by vigorous shaking of the iron and molybdenum solution. (3) The factor for the permanganate solution is 0·0164.

It is shown by 10 selected results that the volumetric method is more accurate than the gravimetric process, since the direct weighing of the yellow precipitate in the filter gives inaccurate results.—A. S.

### *Incandescence Mantles of Commerce, Analysis of.* E. Hintz. Zeits. Anal. Chem. 37, [2], 94—111.

Almost without exception commercial mantles consist of $ThO_2$ with 0·5 to 2 per cent. of $CeO_2$; and, apart from a small quantity of CaO, they contain only minute amounts of impurities derived from the thorium nitrate, namely $ZrO_2$, $Nd_2O_3$ (Neodym oxide), $La_2O_3$, and $Yt_2O_3$. The estimation of all these is a laborious operation. To make sure that they have no appreciable influence on the light-emission from the mantle, and that the use of a shortened analytical process is justifiable, the author had photometric experiments made, of which the results are given in the paper, and from which he draws the following conclusions:—(1) $ZrO_2$, $La_2O_3$, or $Yt_2O_3$, up to 1 per cent., does not increase the lighting power of pure $ThO_2$ mantles; and $Nd_2O_3$ increases it a little, the admixture of 1 per cent. giving 13 Hefner candles with an hourly consumption of 111 litres of gas (=11·4 English candles with 3·9 cb. ft.). (2) Admixtures of up to 1 per cent. of $Nd_2O_3$, $La_2O_3$, or $Yt_2O_3$, or from 0·2 to 1 per cent. of $ZrO_2$, have no influence on the light-emissivity of thoria-ceria mantles: the influence of 0·1 per cent. of $ZrO_2$ is doubtful. (3) The addition of up to 1 per cent. of $ZrO_2$, $Nd_2O_3$, $La_2O_3$, or $Yt_2O_3$, has no influence on thoria-ceria mantles containing 0·5 per cent. of CaO. The CaO in mantles is probably derived from the ash of the fabric: it causes serious shrinkage and should be avoided.

In conjunction with H. Weber, the author has examined Knorre's process for estimating ceria (this Journal, 1898, 72), in which the solution of cerous salt is acidified with $H_2SO_4$, and oxidised to the ceric state by boiling with ammonium persulphate, the yellow ceric compounds being reduced by dilute peroxide of hydrogen to the colourless cerous state, and the excess of $H_2O_2$ titrated with permanganate. [10 atoms Fe = 2 mols. $KMnO_4$ = 5 mols. $H_2O_2$ = 10 mols. $CeO_2$; therefore 56 parts Fe = 140 parts Ce = 172 parts $CeO_2$.] 3 grms. of ammonium persulphate are sufficient to oxidise from 0·2 to 0·3 grm. Ce. The process gives correct results with thoria-ceria mantles, when applied carefully, as follows:—Acidify the solution (see below) containing about 1 grm. of $ThO_2$ and 0·01 grm. of $Ce_2O_3$ with 5 to 7·5 c.c. of dilute sulphuric acid (1 vol. $H_2SO_4$ + 6 vols. of water), dilute to 200 c.c., and add part of the ammonium persulphate. Heat to boiling for 1 or 2 minutes, cool to 40—60° C.; add a second amount of persulphate, again boil and cool; add a third portion and boil for 10—15 minutes. Towards the end of the boiling, add 2 c.c. more of dilute $H_2SO_4$. Cool completely, add dilute $H_2O_2$ from a burette till the yellow colour has just gone, and titrate back at once with permanganate solution. Small quantities of nitrates do not affect the results, but acidification with $HNO_3$ instead of $H_2SO_4$ leads to low results.

The simplified process for analysing incandescence mantles is as follows:—Take at least 12 mantles, in the unburnt condition, and cut off the upper and lower ends, retaining only the middle parts, as the upper ends are sometimes treated with a strengthening fluid, and the lower ends may contain an excessive proportion of cerium nitrate. Weigh the quantity taken and extract completely with water containing a few drops of nitric acid. Ignite the remaining fabric, fuse with $KHSO_4$, dissolve in water and HCl, precipitate with ammonia, add the nitric acid solution of the precipitate to the main solution, and dilute to a convenient bulk. 1. Neodymia (1 part of $Nd_2O_3$ to 100 parts of $ThO_2$) may first be detected in some of the concentrated solution by its characteristic colour and absorption bands. 2. Precipitate part of the solution with oxalic acid, after removal of free acid by evaporation, collect the precipitate (*a*) on a hardened filter-paper, wash into a beaker, heat with strong ammonium oxalate solution, dilute, allow to cool, filter after standing a considerable time, ignite, and weigh. A little thorium oxalate will remain insoluble (this Journal, 1898, 66), so that, even in absence of $Nd_2O_3$, $La_2O_3$, and $Yt_2O_3$, the weight of the precipitate will exceed that of the $CeO_2$ present; at the same time the CaO affects the estimation. If the weight, however, exceeds that of the real $CeO_2$ present by not more than 1 per cent., it may be assumed that $Nd_2O_3$, $La_2O_3$, and $Yt_2O_3$ are not present in appreciable quantity. If the excess be greater than 1 per cent., the precipitate must be dissolved with sulphuric acid and examined further for these earths. To test the filtrate from (*a*) for $ZrO_2$, evaporate to dryness, ignite gently, dissolve in HCl, evaporate off the excess of acid, and transfer to an object-glass with a few drops of water. Evaporate to dryness, add a drop of water and a drop of potassium binoxalate solution, again evaporate, and examine under the microscope for the characteristic crystals of zirconium-potassium oxalate. The presence of 0·1 per cent. of $ZrO_2$ may be thus detected in the solution from a single mantle. 3. Precipitate a part of the solution, after evaporating off the free acid, with oxalic acid, ignite, and weigh the precipitate, consisting of the total rare earths other than $ZrO_2$. The result is influenced by CaO, but for practical purposes, in a mantle consisting of, say, 1·2 per cent. of $CeO_2$, 98·4 per cent. of $ThO_2$, and 0·4 per cent. of CaO, it does not matter if the CaO be reckoned as $ThO_2$. 4. Estimate ceria in an aliquot part of the solution volumetrically, noting that any organic matter extracted from the mantle necessitates the use of more persulphate than is mentioned above. On deducting this result from that at "3," the difference, in the presence of only negligeable proportions of $Nd_2O_3$, $La_2O_3$, and $Yt_2O_3$, gives the $ThO_2$.—H. B.

### *Sulphuric Acid, Volumetric Process for the Determination of Combined [in Waters and Wines].* F. Telle. J. Pharm. Chim. 1898, 7, [4], 165—170.

The sulphuric acid is precipitated by excess of barium chloride in acid solution, the excess of barium by potassium bichromate in alkaline solution, and after filtering, the excess of chromate is determined according to the amount of iodine it liberates from potassium iodide. The solutions required are:—Decinormal barium chloride, potassium bichromate equivalent to the barium chloride solution (7·358 grms. per litre), sodium thiosulphate (38 grms. per litre) standardised by means of the bichromate solution, and 10 per cent. potassium iodide solution.

The solution of the ash of a wine extract in hydrochloric acid, is evaporated on the water-bath to remove excess of acid and is then taken up with water; or in the case of a water, this is evaporated to one-fiftieth of its volume and acidified. In either case, the resulting solution is then transferred to a 110-c.c. flask, 10 c.c. of barium chloride solution added, the liquid boiled, then made alkaline with ammonia free from carbonic acid; next, 10 c.c. of bichromate solution are added, the liquid again boiled, and filtered after cooling and filling up to the mark. To 100 c.c. of the filtrate, 5 c.c. of potassium iodide are added, the liquid acidified, and titrated by thiosulphate. In the case of wines, a small error arises owing to the presence of phosphoric acid; if necessary, this may be previously removed by precipitation with ammoniacal calcium chloride.—A. C. W.

### *Bauxite, The Commercial Analysis of.* W. B. Phillips and D. Hancock. J. Amer. Chem. Soc. 1898, 20, [3], 209—225.

The authors state that the formula given by Roscoe and Schorlemmer for bauxite, *viz.*, $(AlFe)_2O(OH)_4$, does not correspond with the composition of the American mineral, which appears to be a mixture of aluminium trihydrate, clay, and possibly a lower hydrate of aluminium. In valuing the mineral for alum makers, it is usual to determine the amount of available alumina by evaporating with sulphuric acid until acid fumes appear.

But the authors argue that a discrimination ought to be made between the trihydrate, which is readily soluble and of greater value to the alum maker, and the other aluminium compounds, which are only soluble with difficulty. They propose to term the alumina soluble in sulphuric acid of 50° B., (sp. gr. = 1·53) after 1 hour's treatment at 100° C. as "free alumina," that dissolved on evaporation of the acid to fumes as "available alumina," and the difference between them as "combined alumina"; and they describe experiments to prove that practically the whole of the aluminium trihydrate is extracted by the former treatment.

The following method of analysis is recommended. The sample is prepared by being passed through a sieve of 100 meshes to the inch :—

*Moisture.*—Two grms. are heated to constant weight at 100° C.

*Available Alumina.*—Two grms. are mixed with 10 c.c. of cold sulphuric acid (50° B.), and gradually heated until fumes appear, when the basin is covered with a watch-glass and the heating continued for 10 minutes The liquid is then filtered, the residue washed, and the filtrate made up to 200 c.c. 50 c.c. are taken for the determination of alumina, ferric oxide, and titanium dioxide, and a second 50 c.c. for the determination of the two latter.

The first portion is diluted to 300 c.c., acidified with hydrochloric acid, and precipitated boiling with ammonia. The precipitate, dried and weighed, gives the alumina, ferric oxide, and titanium dioxide.

The second portion of the original solution is neutralised with ammonia, the addition of the latter being continued until a precipitate forms. This is just re-dissolved in sulphuric acid, and the liquid is made up to 300 to 400 c.c. and boiled for an hour. Any iron present is reduced by means of sulphur dioxide, care being taken that the liquid smells of the gas throughout the boiling process. In this way a precipitate is obtained practically free from iron. The liquid is filtered whilst hot, and the precipitate dried, ignited, and weighed as titanium dioxide.

*Ferric Oxide.*—This is determined in the filtrate from the titanium dioxide by reduction with zinc and titration with permanganate.

*Free Alumina.*—Two grms. of the sample are placed in an Erlenmeyer flask, with 10 c.c. of sulphuric acid (50° B.). The flask, which is fitted with a perforated stopper, is kept in the hot-water bath for an hour, with constant shaking. The liquid is filtered and the alumina precipitated as before.

A little iron is also precipitated, but this is separately determined and deducted. The titanium dioxide does not pass into solution.

*Combined Alumina.*—The difference between the available and free alumina.

It is not advisable to determine silica in the insoluble residue by means of hydrofluoric acid, since some of the titanium dioxide is also volatilised.

In the authors' experience, titanium dioxide is always present in bauxite, and may amount to 4 per cent.

—C. A. M.

### *Gold Bullion, Assaying of.* C. Whitehead and T. Ulke. Eng. and Mining J. 1898, **65**, [7], 189—190.

*Melting and Sampling.*—The gold, after melting, is carefully stirred with a heated blacklead (graphite) stirrer, and poured into an iron mould, and after cooling, the metal is stamped with the number of the deposit, and sampled by taking cuttings from the top and bottom.

*Gold Assay.*—The standard method used in the United States mints and assay offices for assaying "unparted bars" is described :—Two lots of 500 mgrms. each (equal to 1,000 gold weight) are accurately weighed out on a balance sensitive to at least 1 to 20 mgrms. Enough pure silver is added to make the resulting button one part of gold to two parts of silver. The gold and silver are wrapped in a sheet of pure lead weighing 5 grms., together with about 50 mgrms. of pure copper, if there is none in the sample. A check assay or proof is used with each set of assays. This proof is made of gold of the greatest obtainable purity, taken as the standard of fineness. It is weighed out, wrapped in a sheet of lead, together with a small piece of copper and the necessary quantity of silver (2 to 1 of gold), and the assays are then ready for the furnace. The cupellation should occupy about 12 minutes. This stage of the process should be carefully watched, as the proof correction or "surcharge," which is the difference between the loss of gold and the amount of "unremoved" silver, is chiefly governed by the temperature of the furnace. The buttons are flattened, annealed by heating to redness, rolled out into thin strips, numbered, again annealed, and rolled, somewhat loosely, into coils, termed "cornets." The latter are boiled for 10 minutes in platinum boilers containing nitric acid of 32° B., then removed to a second bath of hot acid of the same strength, and again boiled for 10 minutes. The assays are then removed, thoroughly washed, dried, and annealed by heating to redness for a few minutes. After cooling, they are ready for the final weighing. The proofs are accurately weighed, and the correction determined as follows :—Suppose 900 to be the original weight of the proof, then, if the subsequent process of treatment were absolutely perfect, the resulting "cornet" would again weigh 900, and no correction would be necessary. However, in good work, a 900 proof will generally gain; if the final weight be 900·5, then 0·5 would be deducted from the weight of the cornets as the fineness of the bullion. Proof weighed at 900 should not gain less than 0·1, nor more than 1. Duplicate assays should not differ more than 0·2 to 0·3, except in the case of a very base gold bullion. With bullion containing a very large amount of base metal, and also when over 3 per cent. of platinum is present, it is recommended to take only one half, or, in extreme cases, only one quarter of the usual weight for assay, enough pure gold being added to make a "cornet" weighing approximately 900, and silver also being added in the proper proportion. The gold added is, of course, deducted from the final result. In the case of silver bullion containing gold, it is best to add enough proof gold to make the button 900 fine, and then, of course, to add the necessary amount of silver.

Gold ingots are sampled by taking cuttings from the first and last ingots of a lot, and are assayed by the above method, except that :—Three parts of silver to one part of gold are used, and the "cornets" are boiled for 10 minutes in three different acid baths, the first of 22° B., and the second and third of 32° B. This method is said to give more uniform results than the first one, and is to be preferred for extremely accurate work.

*Determination of Base Metals.*—0·5 grm. of the bullion is cupelled, at a low temperature, with 5 grms. of lead, and the resulting button weighed. The loss in weight, corrected for proof, represents the base alloy.

*Silver Determination.*—The silver in unparted bars is generally determined by difference. When platinum is present in the bullion, the silver is determined by one of the wet or volumetric methods. In the determination of silver in gold deposits, 0·5 grm. of the alloy is placed in a porcelain crucible with 5 or 6 grms. of potassium cyanide, and the whole melted. When in perfect fusion, 2 or 3 grms. of cadmium (free from silver) are added, and allowed to perfectly alloy with the bullion, after which the crucible is cooled, and the cyanide dissolved in water. The clear button is dissolved in 25 c.c. of nitric acid of 32° B., and the silver determined either with sulphocyanide or by the usual common salt solution, after adding enough pure silver to make the total present about 1,004 mgrms.—A. S.

### *Mercury and Bismuth Salts, Separation of.* L. Vanino and F. Treubert. Ber. **31**, [2], 129—130; this Journal, 1897, 1043, and 1898, 72.

The method of separation is based on the complete precipitation of mercuric chloride, as mercurous chloride, from a dilute hydrochloric acid solution, with a mixture of hydrogen peroxide and hypophosphorous acid, whilst the bismuth remains in solution, and may subsequently be thrown down as metal by a fresh addition of hypophosphorous acid.

To the dilute hydrochloric acid solution of the mixed mercuric and bismuth salts, excess of a mixture of hydrogen peroxide and hypophosphorous acid (1 drop of commercial hypophosphorous acid to 1 c.c. of hydrogen peroxide) is added. After standing about an hour, the mercurous chloride is filtered off and quickly washed with dilute hydrochloric acid, then with water, and dried at 105° C.

The filtrate is warmed and made slightly alkaline with caustic soda solution. Hypophosphorous acid is added, and the solution stirred whilst heated, over a flame until a black precipitate and a clear liquid are obtained. It is advisable to compress the spongy precipitate to a compact mass with a glass rod, as otherwise a partial oxidation of the bismuth is apt to take place on drying. The bismuth is collected on a weighed filter or in a Gooch crucible, and, after washing with water and alcohol, is dried at 105° C.

—J. McC.

### *Phosphates, Estimation of the "Citrate-soluble" Phosphoric Acid.* O. Böttcher. Chem. Zeit. 1898, **22**, [21], 201—202.

THE author has compared the results obtained by Wagner's method (this Journal, 1897, 1044) with that of Petermann, and finds that, whilst corresponding results are obtained by both methods in the case of bone superphosphate, Wagner's method gave higher results than Petermann's in the case of precipitated calcium phosphate.

The author considers Wagner's method the most reliable which has yet been proposed, but recommends that the phosphate should not be digested in the acid ammonium citrate solution for so long a time as 18 hours. He considers it better to agitate the mixture by means of a mechanical stirrer for about half an hour at the ordinary temperature (about 17·5° C.). 5 grms. of the phosphate are thus digested in ½ litre of the acid ammonium citrate solution, and the phosphoric acid precipitated according to Wagner's directions in 100 c.c. (or 50 c.c. in the case of high-grade phosphates) of the filtrate.—J. W. L.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

### *Aldehydes and Ketones, A Photographic Reaction of.* Lumière Bros. and Seyewetz. Bull. Soc. Chim. 1898, **19**, 134.

THIS test is an adaptation of the process whereby a phenolic developer can be made to act upon the latent photographic image without the addition of alkali, but by using sodium sulphite and an aldehyde or a ketone instead (this Journal, 1896, 920; 1897, 259).

A solution is prepared containing pyrogallol, 1·5 grm.; anhydrous sodium sulphite, 7 grms.; water, 100 c.c.; or, if preferred (the results being practically identical), quinol (hydroquinone), 2 grms.; sulphite, 20 grms.; and water, 100 c.c. Two photographic dishes are filled with 50 c.c. of the liquid; to one is added 25 c.c. of a 1 per cent. aqueous or alcoholic solution of the body to be tested; to the other, 25 c.c. of plain water or spirit to match the first. A normally exposed plate is cut in half, and each section dropped simultaneously into one of the baths. If the image on the film placed in the former dish appears sensibly quicker than that in the latter, an aldehyde or a ketone is certainly present

The reaction, however, is not absolutely general: in the aromatic series especially, there are many exceptions; and these are in all probability the bodies which either do not combine readily with the bisulphite formed during the process, or such as are but slightly soluble in weak alcohol (water is of course necessary for development), while it is possible that some have a specifically inhibitive action of their own.

In the aliphatic series, the simple aldehydes and ketones of saturated or unsaturated radicles, determine, even in small proportions, a more or less rapid development. On the contrary, the following substances do *not* act on the image: paraldehyde, chlorine substitution products of aldehydes, such as chloral; bodies containing one aldehyde or ketone group, and one or more alcohol groups, such as aldol, glucose, and lævulose; bodies containing two ketone groups, such as acetylacetone; acid ketones, such as pyruvic acid, unless the acid group is neutralised; bodies containing two acid and several ketone groups, such as dihydroxytartaric acid.

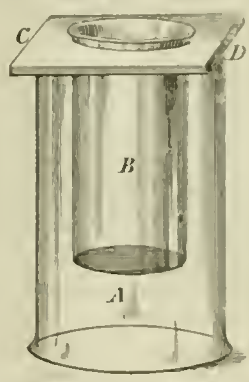
In the aromatic series, an energetic action is obtained only with the simple aldehydes, such as benzoic or cinnamic. Mixed aldehydes only develop if the second group is neutral. 1:4-hydroxybenzaldehyde and vanillin, therefore, are inert; but anisaldehyde and piperonal react, although they are less efficacious than the unsubstituted aldehydes. The aromatic mono- or poly-ketones, either simple or mixed, such as acetophenone, benzophenone, benzil, benzoin, the quinones, &c., do not affect alkaline developers.

The test is sufficiently delicate to detect 1 part of formaldehyde in 25,000 parts of liquid; 1 of acetaldehyde in 15,000; and 1 of propanone in 2,500: in fact, its sensitiveness ranks with that of Schiff's reagent for magenta decolorised by sulphurous acid.—F. H. L.

### *Paraffin, Commercial; Testing the Setting Point of.* R. Kissling. Chem. Zeit. **22**, [22], 209—210.

THE author proposes the following method as being simpler and less liable to error than those in use at present:—

A is a glass cylinder about 8 cm. wide and 12 cm. high, supporting a wooden or mill-board plate, C D, pierced at the centre, with a hole for the insertion of the smaller beaker B—about 8 cm. high and 5 cm. wide—which is thus supported by the rim. About 50 grms. of paraffin—previously clarified (if necessary) by filtration—are placed in B and heated to about 10° C. above the setting point, by immersing the beaker in a water bath, whereupon the beaker is placed in position as shown in the figure, and the contents stirred with a thermometer—the bulb of which is small, and the stem graduated from 20° to 80° C.—until a film of solid paraffin forms on the bottom of the beaker. This film is detected by the marks made in it by the bulb of the thermometer in stirring, and can be best observed through the sides of the apparatus. A skin then immediately forms on the surface of the paraffin, and the previously clear liquid becomes flocculent. The same sample can be used for repeating the test, the beaker being kept covered until stirring is commenced.



The operation lasts only 10—12 minutes, and the results are concordant to within $\frac{1}{10}$° C. of the mean. The apparatus may also be employed for testing the setting point of any solid fat.—C. S.

### *Edible Fats, A Delicate Test for the Detection of a Yellow Azo Dyestuff used for the Artificial Colouring of.* J. F. Geisler. J. Amer. Chem. Soc. 1898, **20**, [2], 110—113.

THOSE engaged in clarifying and decolorising oils and fats are no doubt familiar with the use of fuller's earth as a precipitant of colouring matter, the latter being usually precipitated without any pronounced colour reaction. The author calls attention to the red to pink colour which fuller's earth produces with some of the yellow azo dyes, and to the practicability of using this earth as a most convenient and delicate test for the detection of at least one, if not more, of the yellow azo dyes. The latter are now largely used as constituents of butter-colours, and replace annato to a considerable extent.

A reddish-coloured butter intended for export developed an intense violet-red tint when the attempt was made to remove the colouring matter from the clarified fat by means of fuller's earth. The tint developed depended much upon the relative amount of fuller's earth added, ranging from a deep violet-red to pink. All the colouring matter giving this particular reaction was readily precipitated. The precipitate, after having been thoroughly washed with naphtha to remove the fat, presented, upon drying, a violet-red powder. Contact with alcohol immediately decolorised it, the colour reappearing upon the evaporation of the alcohol. Boiling alcohol readily extracted all the colouring matter from the precipitate, producing a yellow solution. The colouring matter obtained from the latter is quite insoluble in water, sparingly soluble in hot water, but re-precipitated on cooling. It dissolves with a yellow colour in concentrated sulphuric acid, which, upon dilution with water, develops a bright pink to red colour. Strong mineral acids also produce a violet to pink colour, similar to that of Methyl Orange, which does not, however, give the fuller's earth reaction, nor is it as soluble in fats as this particular dyestuff.

Applying the fuller's earth test to samples of butter and oleomargarine, a large number gave the reaction even where the samples were but lightly coloured.

To test the sensitiveness of the reaction, mixtures were made with varying amounts of the yellow dyestuff and pure white lard, and the test applied. With 14 grains to the ton, or one part per million, a pink tint was obtained with half a gramme of the sample spread on white porcelain slab. The reaction is therefore a very delicate one.

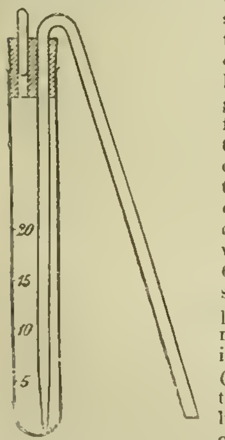
Commercially, the yellow azo dyestuff is generally used in conjunction with an orange variety. The latter does not give the fuller's earth test.

For practical purposes, the test is readily applied by spreading some of the clarified fat to be tested upon a white porcelain surface, and stirring into the fat a pinch of fuller's earth, and observing the change in colour. A pink to violet-red colour will appear within a few moments if any considerable proportion of this colouring matter is present.

It is generally asserted that these azo dyestuffs are not detrimental to health.—L. J. de W.

*Sesamé Oil in Butter and Margarine, Detection of, in the presence of Artificial Colouring Matters.* C. A. Neufeld. Zeits. f. Untersuchung d. Nahr.- u. Genussmittel. 1893, **3**, 156—158.

When butter contains colouring matter which gives a red solution with hydrochloric acid, the colour must be removed before the fat can be tested for sesamé oil. The author recommends the use of the following apparatus for this purpose. A stout test-tube, graduated into 5, 10, 15, and 20 c.c., is fitted with an india-rubber stopper through which two holes are bored. A small siphon-tube passes through one of the holes and reaches to the bottom of the test-tube, whilst the other hole is stopped up with a piece of glass rod. 10 c.c. of the melted and filtered fat are placed in the test-tube together with 10 c.c. of hydrochloric acid (sp. gr. 1·25). The tube is then closed with an ordinary cork and shaken. If the acid be coloured red, the tube is placed in a water-bath having a temperature of 60° C., and the liquids allowed to separate. The india-rubber stopper provided with the siphon is now fitted into the test-tube, which is again placed in the water-bath. (To prevent fat entering the siphon the outlet end of the same should be closed with the finger while the other end is placed in the liquid.) As the test-tube becomes warm, the acid rises in the limb of the siphon, and is allowed to flow over by removing the glass rod from the stopper as soon as the siphon is full of acid. The washing with acid is repeated until all the colour is removed, when the ordinary test for sesamé oil may be applied.—W. P. S.



*Coffee, Artificial Colouring of, and Means for its Detection.* G. Morpurgo. L'Orosi, **20**, 397—403; Chem. Centr. 1898, **1**, [7], 472.

Coffee is coloured artificially to "improve" its appearance. The coffee is moistened with water containing an antiseptic, then dried by admixture with sawdust, and the dry material mixed with the ground colouring material. The following substances are used, it is said for colouring coffee:—Graphite, charcoal from the wood of the lime tree, animal charcoal, soot, ultramarine, mixtures of blue and green, Malachite Green lake, Prussian blue, tannate of iron, aniline colours, lakes, lead chromate, yellow ochre, calcium carbonate, and talc. A gloss is finally imparted to the coloured coffee by means of resin or wax. The following method is recommended for the detection of artificial colouring:—50 grms. of the unground sample are boiled with alcohol for ½ hour under a reflux condenser, the hot alcohol poured off, the residue boiled up with alcohol which has been rendered faintly alkaline, and the latter again poured off. The first extract contains the wax and the easily soluble colours, and the second extract the less soluble colours. Both extracts are allowed to settle, and the sediments examined microscopically. In the alcohol poured off from the sediment of the first extract, the wax may be precipitated by water, and is unsaponifiable. The aniline colour lakes may be recognised under the microscope on slices of the epidermis of beans, as they are dissolved by the alcohol, and are thus distributed over the surface of the pieces. When the sediments of the alcoholic extracts contain fragments of plants, they should be treated, dry, with chloroform, whereby the heavier colouring matters are mechanically separated. In the sediments under the microscope there remain the black particles that consist of graphite and potash, the white particles of talc, indifferent towards alkalis and acids, as also the blue smalts. Calcium carbonate is recognised by the effervescence on treatment with acids, Prussian blue by its reaction with potash, basic copper carbonate by the colour of its solution in potash and acids, and iron oxide by its solution in acids and precipitation by alkalis. Ochre is recognised under the microscope by the yellow colour of its solution in hydrochloric acid, which is not altered by sulphuretted hydrogen; and lead chromate, on the other hand, by its greenish solution, which, with sulphuretted hydrogen, gives a brown colour, and, later, a brown precipitate.—A. S.

*Starch, &c. in Chocolate, Detection of.* G. Possetto. Giorn. Farm. Chim. **48**, 5—8; Chem. Centr. 1898, **1**, [6], 418.

The author tested several kinds of pure cocoas and also cocoas adulterated with starch, potato starch, dextrin, maize flour, and chestnut meal. 2 grms. of the roasted sample were boiled for two minutes with 20 c.c. of water, 20 c.c. more of water added without shaking, and finally 1—2 c.c. of a solution of 5 grms. of iodine in 100 c.c. of a 10 per cent. potassium iodide solution. With adulterated cocoas, an intense colour reaction was obtained, which remained permanent for one hour; but, with pure cocoas, the blue colour either did not appear at all, or it disappeared again after, at the most, 25 minutes. The test is carried out in a similar manner for chocolate, and if, after the expiration of one hour, the blue colour has not disappeared, it may be stated that the chocolate has been adulterated with meal containing starch.—A. S.

*Chloroform, Bromoform, and Chloral; A Reaction for.* A. Desgrez. Acad. d. Science, 1897, 780; Analyst, 1898, **23**, [264], 76.

The reaction depends on the decomposition of chloroform by aqueous potash, in accordance with the following equations:—$CHCl_3 + 2KOH = 2KCl + HCl + H_2O + CO$ and $CHCl_3 + KOH = KCl + 2HCl + CO$. The carbon monoxide may be recognised by its blue flame, its absorption in a solution of cuprous chloride in hydrochloric acid by Berthelot's test (ammoniacal silver nitrate), &c. The reaction is accelerated by a gentle heat and by exposure to light. Bromoform is decomposed in a similar manner, though more slowly; but iodoform, carbon tetrachloride, and methylene chloride do not give the reaction. In the case of chloral, the reaction proceeds more rapidly than with chloroform, on account of the heat generated by its primary decomposition into the latter substance and formate. The reaction cannot be effected with alkaline carbonates and bicarbonates, nor with ammonia in the cold.
—A. S.

*Phenylhydrazine, New Colour Reaction of.* L. Simon. Comptes Rend. 1898, **126**, [6], 483—485.

Sodium nitroprusside can be used to detect phenylhydrazine, as follows:—The solution supposed to contain

phenylhydrazine is heated for a few seconds with a few drops of aqueous trimethylamine ; a few drops of an aqueous solution of sodium nitroprusside are then added, followed by some concentrated caustic potash solution. On the addition of the nitroprusside, a bright blue colour is struck, which is deepened by the caustic potash. A little acetic acid, added either before or after the potash, inclines the tint to sky blue. A solution of phenylhydrazine of 1 in 1,000 gives a strong coloration ; one of 1 in 50,000 can be detected. The colour vanishes after a quarter of an hour or so. Alcohol and ether do not affect the reaction, but chloroform and benzene prevent it. Acetone, if present, gives its own colour (Legal's reaction). Acids retard the appearance of the colour till neutralised by the potash. Acetic acid in large excess, or hydrochloric acid, added to the coloured liquid, causes, first, a reddening, and then the disappearance of the colour. The colour is produced but slowly in the cold, and, if the reagents are added first and then the mixture is heated, the blue colour is very fugitive. The reaction as described, is shown to occur among the various substances which the author has examined, only with phenylhydrazine and such of its derivatives as are formed by substitution in the benzene ring.
—J. T. D.

### *Salol, A Reaction of.* Rev. intern. falsific. **10**, 202; Chem. Centr. 1898, **1**, [3], 222.

A small quantity of salol is decomposed in a porcelain crucible with a few drops of concentrated sulphuric acid to which a nitrite has been added. A yellow coloration, changing to brown and finally to green, is produced. The colour changes to red on addition of water, and greenish on addition of ammonia. Resorcinol, treated in a similar manner, gives a deep blue coloration, turning red on addition of water, and blue on adding ammonia.—A. S.

### *Creosotes and Guaiacols, Reactions of.* H. Fonzes-Diacon. Bull. Soc. Chim. 1898, **19**, 191—192.

10 drops of the material to be tested are dissolved in a litre of water, and 10 c.c. of the solution treated with 2 c.c. of a solution of copper sulphate containing 0·5 grm. per litre, and 1 c.c. of a 0·4 per cent. solution of potassium cyanide. Colorations varying from yellowish-orange to yellowish-green are produced, which are compared with the colorations produced in standard solutions. Precipitates are formed on standing. A 12 to 25 per cent. creosote, a 65 to 70 per cent. and an 85 to 90 per cent. guaiacol may be distinguished without any comparison, by dissolving a trace in water and adding 2—3 c.c. of a 1 per cent. solution of copper sulphate and 1 to 2 c.c. of a 0·4 per cent. solution of potassium cyanide. Striæ of a precipitate are formed, which, seen by transmitted light, are emerald green with the creosote, reddish-grey with the poor guaiacol, and maroon-purple with the rich guaiacol.—T. E.

### *Lemon Oil, Adulteration of, by Means of Oil of Turpentine; New Method for Detecting.* G. Mancuso-Lima. Staz. sperim. agric. ital. **30**, 631—643; Chem. Centr. 1898, **1**, [7], 477.

Olivieri's method has not been found successful, and the author shows that the method of Soldaini and Bertè—in which the rotation values of the oil before distillation, and of the distillate and residue, are compared—sometimes gives uncertain results. Good results were, however, obtained with the following method :—30—50 c.c. of the oil to be examined are shaken with an equal volume of sodium bisulphite solution (saturated at 15° C.) till all the citral is combined. The product is poured into a tubulated retort of 1½ litres capacity, and distilled over the oil-bath in a vacuum till a white solid residue is left behind. The distillate is composed of an acid aqueous layer, and an oily layer containing the terpene. The terpene is run off, dried with caustic potash, and its rotation number determined. If the lemon oil was pure, the rotation of the terpene is 8—11° more than that of the original oil. If about 5 per cent. of turpentine oil has been added to the lemon oil, the rotation of the distillate is the same as, or 2—3° higher than, that of the original oil, but never 8—11°. The lowering of the rotation number varies according to the increase in the amount of turpentine added, each per cent. of turpentine causing a lowering of 1·41°.—A. S.

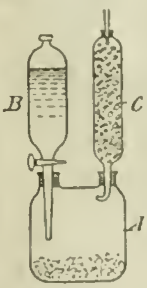
### *Alkyl Bismuth Iodides and Bismuth Iodides of Vegetable Bases. [Dragendorff's and Mayer's Reagents.]* A. B. Prescott. J. Amer. Chem. Soc. 1898, **20**, [2], 96.

*See under* XX., *page* 368.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Calcium Carbide, Analysis of.* H. Bamberger. Zeits. f. angew. Chem. 1898, [9], 196—198.

In the usual methods of analysing calcium carbide, the gas evolved from a given weight of the sample on treatment with water is collected and measured, corrections being made for temperature and pressure and for the amount of gas dissolving in the liquids with which it comes in contact. In order to avoid these calculations and the chance of error in the solubility allowance, the author has devised the gravimetric method described below, which is based on the same principle as the differential determination of carbon dioxide in carbonates, and which, he claims, gives exact results in as short a time and with less trouble than the volumetric methods.



The apparatus consists of a flask A, holding about 400 c.c., and fitted with a dropping funnel B, of about 200 c.c. capacity, and a drying tube, C, filled with calcium chloride. From 50 to 60 grms. of the carbide in small pieces are placed in the flask, and the funnel filled with a 15 to 20 per cent. solution of sodium chloride. The apparatus (about 500 grms. in all) is weighed on a balance sensitive to 1 mgrm., and the salt solution is then allowed to fall drop by drop upon the carbide. After three or four hours, when about half the solution has entered the flask, and the whole of the carbide has been decomposed, the remainder of the liquid is rapidly added, the flask slightly warmed, and a current of air drawn through the apparatus. When cold, it is again weighed, and the loss in weight gives the amount of acetylene evolved. From this the proportion of calcium carbide in the sample and its yield of acetylene per kilo. is calculated, with the data that 1 kilo. of pure carbide yields 40·625 per cent., or 348·9 litres of acetylene.—C. A. M.

### *Prussian Blue in Spent Gas-purifying Material [Oxide], Determination of.* Lubberger. J. für Gasbeleucht. 1898, **41**, [8], 124—126.

Drehschmidt estimates the Prussian blue in spent oxide by boiling the spent oxide in water along with mercuric oxide, then agitating with a little mercurous nitrate and ammonia—whereby sulphocyanide and chloride are precipitated—filtering, converting the cyanogen in the filtrate into ammonium cyanide by addition of ammonia and zinc dust; again filtering; adding to the filtrate slight excess of decinormal solution of silver, and titrating back with determination of the excess, with standard solution of ammonium sulphocyanide. The method is described in detail in Muspratt's Chem. **5**, 505 (1893 edit.). Since it appeared doubtful if all the sulphocyanide and chloride were precipitated according to this method, Burschell modified it by first extracting the blue from the oxide by means of alkali and iron salts, and then making the estimation according to Drehschmidt's method on the blue thus purified. The author found, however, that the final silver precipitate contained no chloride, and only traces of sulphocyanide in some cases. He added excess of silver nitrate to the solution after it had been treated with zinc dust, dried the precipitate formed at 105° C., and weighed it; then heated it to incandescence in both air and a stream of hydrogen, and weighed the silver obtained. He proved that the precipitate contained only traces of sulphocyanide, as the weight of Prussian blue corresponding to the weight of the precipitate (when the latter was regarded as wholly consisting of silver cyanide) was less than the weight found by the volumetric method. If sulphocyanide were present in the silver precipitate, the weight of Prussian blue deduced therefrom would have been greater than that found volumetrically. The following are the percentages of Prussian blue found in four samples of oxide :—

| | | | | |
|---|---|---|---|---|
| Deduced from the silver precipitate, such being regarded as silver cyanide. | 10·3 | 9·34 | 8·58 | 8·74 |
| By Drehschmidt's volumetric method. | 10·5 | 9·39 | 8·60 | 8·80 |
| By the Drehschmidt-Burschell method of extracting the blue. | 7·9 | 6·58 | 6·91 | 7·13 |

The author concludes that spent oxide contains cyanogen compounds, which do not form blue, and are not precipitated by iron salts, and that these compounds are converted into mercuric cyanide when boiled with mercuric oxide. Consequently they are estimated along with the ferrocyanide when Drehschmidt's method is used, though only the ferrocyanide is valuable to chemical works. Unless other cyanogen compounds are known to be absent, he recommends the preliminary extraction of the blue (Burschell's method), which renders the method of estimation reliable and exact for all cases.—J. A. B.

*Spermaceti Examination of.* L. F. Kebler. Rev. intern. falsific. **10**, 208; Chem. Centr. 1898, **1**, [3], 223.

In testing the purity of spermaceti, it is necessary to estimate the specific gravity, melting-point, acid and saponification numbers. The specific gravity is usually determined by suspension in a liquid of known density (dilute alcohol), but the results obtained by this method are too high, on account of the crystalline nature of the spermaceti. To overcome this defect, melted spermaceti is dropped on to a wet plate at 20° C. Solidification then ensues so rapidly that the drops solidify without becoming crystalline. It is better, however, to estimate the specific gravity at the temperature of boiling water. An examination of 12 samples of spermaceti yielded the following results :—

| | Minimum. | Maximum. |
|---|---|---|
| Melting point ........................ | 43° C. | 45° C. |
| Solidifying point........................ | 42° C. | 44·5° C. |
| Acid number ........................ | 0·09 | 0·47 |
| Saponification number.................. | 124·8 | 125·31 |
| Specific gravity at 98°—99° C. ........ | 0·8082 | 0·8160 |
| „ „ 15° C. (in alcohol). | 0·8960 | 0·9103 |
| „ „ 15° C. (by suspension). | 0·9381 | 0·9510 |

—A. S.

*Bone-Fat, Technical Analysis of.* A. A. Shukoff and P. J. Schestakoff. Chem. Rev. Fett- u. Harz-Ind. **5**, [1], 5—8; [2], 21—23.

I. *Moisture.*—The drying temperature varies, in different current methods, from 100° to 130° C., but when the higher limit is approached, the results may come out in excess of the truth, owing to the presence of volatile bodies (*e.g.*, benzene from extracted fat), so that a temperature of 100°—110° C. is most suitable. The time occupied in drying varies considerably: if a loss of 1—2 mgrm. per hour is regarded as sufficient constancy of weight, 3—4 hours suffice for good tallow (0·5 grm of substance), whilst bone-fat containing lime will not be properly constant in 24 hours, owing to its affinity for water. The values afforded by the Tate method come out too low for bone-fat. The usual method may be shortened by the aid of a current of carbon dioxide, except when lime is present, the frothing then produced retarding evaporation.

II. *Extraneous matters* include (organic) dirt, sand, calcium phosphate, and lime soaps, the latter being more or less soluble in the usual solvents, though the extent of solubility is irregular, and depends on the reagent, the temperature and time of the operation, and the water-content of the fat. Solution is complete at first, but re-precipitation occurs on standing, and is more thorough as the liquid cools and when the fat has not been previously freed from water. By using petroleum spirit (b. pt. 85° C.) as solvent, and working with dried fat, almost the whole of the lime-soap is retained in solution. Whether this substance should be considered as extraneous or not depends on its amount and the purpose for which the fat is intended, the presence of a large proportion being prejudicial in soap-making (paste soaps by the direct method).

III. *Ash* must be determined by the combustion of the entire fat, the examination of extract residues giving imperfect results. Calcium carbonate and oxide are the chief constituents, and as the former is derived almost entirely from the lime-soaps, the latter may be estimated by titrating this salt and employing an average molecular weight of 260, for the fatty acids, in calculating. The estimation of sand and calcium phosphate, which necessitates a quantitative analysis of the ash, is seldom required.

IV. *Direct Estimation of Actual Fat.*—The ordinary method of estimating this important constituent, *viz.*, by difference, is manifestly open to serious error. The authors warm 10 grms. of sample for an hour with 3-5 drops of strong hydrochloric acid, shaking the mixture frequently, (thus all lime-soaps are decomposed), and they then dissolve the fat by agitation with 40 c.c. of light petroleum spirit. The ethereal solution is decanted and filtered, and the dirt and acid liquor washed with the solvent, the bulk of the spirit being then removed by distillation, and the fat dried at 100°—110° C. by the aid of a current of carbon dioxide until constant. The extraneous matters are determined by rinsing the dirt and acid liquor on to the same filter and weighing. In this case, moisture can be estimated by difference with tolerable precision, except when lime-soaps are present, the result then coming out too high.

V. *Unsaponifiable Matter.*—The results of the Allen and Thomson, and Morawsky and Demsky methods disagree, owing to the less complete extraction of cholesterin by the latter process. In both cases the unsaponifiable matter has an acid reaction from the presence of acid soaps derived from particles of the soap solution carried over in the extracts, and as these acid soaps are soluble in benzene and petroleum ether, the unsaponifiable matter should be neutralised with caustic soda before treatment with these solvents. The following modification has been devised with a view to overcoming the defects of the aforesaid methods :—Five grms. of the fat are evaporated to dryness with 25 c.c. of 8 per cent. alcoholic caustic soda, then taken up with 80 c.c. of water, and extracted (three times) with 80 c.c. of ether in a separating funnel, a little alcohol being added, if necessary, to cause the layers to separate. The extract is concentrated, the residue rendered alkaline with normal caustic soda, and dissolved in petroleum spirit. The filtered solution yields, on evaporation, an unsaponifiable residue perfectly free from ash.

The *setting point of the fatty acids* is determined by the original Dalican method.—C. S.

*Wax, Examination of.* G. Buchner. Zeits. für öffentl. Chemie, **3**, 570—573; Chem. Centr. 1898, **1**, [2], 151.

On examination of several samples of Indian and Chinese wax, the numbers given in the table on next page, differing materially from the normal ones for beeswax, were obtained (A = Indian wax; B = Chinese wax).

The samples of Indian wax were somewhat harder and more glossy than beeswax, and were distinguished by a higher degree of plasticity.

The author, contradicting K. Dieterich's statements, states that the cold saponification process is as reliable as the hot one, provided that the petroleum spirit used in the former method be vigorously boiling. The Hübl process gives somewhat lower acid numbers, but higher ether numbers. The petroleum spirit used in the cold saponification process should boil between 100° and 150° C.—A. S.

*Drying-Oils, Varnishes, and Dégras Oils; Testing.* A. Gawalowski. Chem. Rev. Fett- u. Harz-Ind. **5**, [1], 9. (See also this Journal, 1897, 922 and 1024.)

To determine directly the increase or decrease in weight sustained by oils, &c., when exposed to the action of air, the author distributes about 5—8 grms of the sample evenly over both surfaces of a tared slip (about 15 by 4 cm.) of thin platinum foil, which is then bent into a spiral and placed in a drying tube between two loose plugs of platinised asbestos. After weighing the whole, a current of air—dried and freed from carbon dioxide—is passed through the apparatus for 2—3 weeks, the moisture and carbon dioxide in the effluent being estimated by absorption in the usual manner. When the absorption tubes cease to increase in weight, the spiral and tube are re-weighed, and, if desired, the ash constituents of the varnish can be estimated.

| —— | Acid Number. | Ether Value. | Saponification Number. | Ratio Number, Acid Number, Ether Number. | Melting Point. | Iodine Number. |
|---|---|---|---|---|---|---|
| | | | | | °C. | |
| A 1 | 6·10 | 77·20 | 83·30 | 12·1 | 65 | 10 |
| A 2 | 6·01 | 76·11 | 82·12 | 12·6 | 66 | 10 |
| B 1 | 7·55 | 86·15 | 93·70 | 11·4 | 66 | .. |
| B 2 | 6·28 | 83·82 | 90·20 | 13·9 | .. | .. |
| B 3 | 6·40 | 90·30 | 96·70 | 15·6 | .. | .. |
| B 4 | 5·33 | 90·28 | 95·61 | 17·9 | 62—63 | .. |
| B 5 | 8·72 | 111·45 | 120·17 | 12·78 | .. | .. |
| B 6 | 7·51 | 84·63 | 92·14 | 11·26 | .. | .. |
| B 7 | 9·74 | 107·66 | 117·40 | 11·05 | .. | .. |

A slight error in excess is produced, in the water and $CO_2$ determinations, by the volatile fatty acids present, but the author hopes to eliminate this by a preliminary passage through a solution containing a salt of one of the noble metals.

The results obtained by the method are threefold, *viz.*, an exact carbonic acid number, an accurate oxygen-absorption number, and data for a comparative valuation of drying fats and varnishes.—C. S.

### *Glue, The Testing of Commercial.* R. Kissling. Chem. Zeit. 22, [19], 172.

THE author gives the results of some comparative experiments he has made on processes of C. Stelling (this Journal, 1896, 620), and J. Fels (Chem. Zeit. 1897, 21, 56). In the former, the non gelatin is estimated after precipitation of the gelatin by alcohol; in the latter, the viscosity of a solution of the glue is estimated. The non-gelatin does not consist merely of the decomposition products of the glutin, but of fat and other impurities and adulterants, besides which, glutin is by no means entirely insoluble in 72 per cent. alcohol. Nevertheless the method gives a partial indication of the value of a glue. Fels' method has only a limited application. The following results were obtained:—

| Variety. | No. | Price in Marks per 100 kilos. | Smell of Powdered Glue. | Relative Gelatinising Property (Tenacity). | Viscosity at 25°. | Per Cent. Non-Gelatin, Stelling's Method. |
|---|---|---|---|---|---|---|
| Skin glue | 1 | 86 | Good | Over 1,000 | 15·0 | 7·6 |
| " | 2 | 86 | " | .. | 9·0 | 8·2 |
| " | 3 | 95 | Very good | 130 | 4·0 | 8·3 |
| Bone glue, powdered | 4 | 70 | Pretty good | 5 | 4·2 | 14·8 |
| " " | 5 | 70 | Unpleasant | 110 | 5·5 | .. |
| " " | 6 | 70 | Pretty good | 1 | 3·7 | .. |
| " " | 7 | 70 | " | 234 | 7·4 | 15·0 |
| Mixed glue | 8 | 80 | Bad | 1·5 | 2·8 | 13·6 |
| Bone glue, in cakes | 9 | 54 | " | 2·4 | .. | 15·4 |
| " " | 10 | 50 | Very bad | .. | .. | 23·2 |
| " " | 11 | 50 | Rather bad | Liquid | 2·8 | 15·6 |
| " " | 12 | 50 | Bad | 120 | 4·4 | 8·75 |

The gelatinising property (tenacity) is estimated by the method described by Kissling (this Journal, 1893, 952), by the time of sinking of glass, zinc, and brass rods into the jelly. The viscosity is estimated on a 17 per cent. solution by means of Engler's viscosimeter at 25°. The figures, as might be expected, show a relation between the gelatinising property (tenacity) and the viscosity, but the former evidently gives more information as to the value of the glue (compare Nos. 3, 4, 6, and 12, wherein the viscosity numbers do not much differ, whilst the gelatinising or tenacity numbers show considerable divergencies). The author shows that the fluidity of glue solutions alters considerably on standing. Stelling's process gives unsatisfactory results as to the quality of the glue, although the glue of the highest gelatinising property shows the smallest amount of non-gelatin. Even here Nos. 7, 8, and 11 are exceptions. In adhesive power, cheap bone glues give almost as good results as the high-priced skin glue. The smell is especially important. It indicates the keeping property of the glue. For some purposes acidity is of great importance (*see* this Journal, 1887, 565). If a glue buyer requires good smell, gelatinising property, and freedom from acidity, a best skin glue must be selected. If only the adhesive properties are to be considered, then a cheap bone glue will answer the purpose.—J. T. W.

### *Glucose, Quantitative Analysis of.* E. Pflüger. Pflüger's Arch. 69, 399—471; Chem. Centr. 1898, 1, [6], 420.

ALLIHN has stated (J. Prakt. Chem. 22, 70), that a single boiling up of the dextrose solution with the alkaline copper solution is sufficient, but the author confirms the objection of Rosham (Dingler's Polyt. J. 293, 236) that, on ebullition, the total amount of cuprous oxide corresponding to the glucose is not obtained, and he also shows that if the boiling be prolonged for 2 minutes, a better separation of cuprous oxide is obtained than by a single boil up. The same results were obtained whether the sugar solution was mixed with the cold Fehling's solution and boiled for 2 minutes, or whether it was poured into the already boiling Fehling's solution.

The copper oxide method, in which the precipitated cuprous oxide is converted into cupric oxide and weighed as such, was found to give reliable results.

The error in the copper sulphocyanide method was found to be from − 0·34 to − 0·5 per cent., but this error is diminished if the atomic weight of copper be taken as 63·4 instead of 63·18. With regard to the cuprous oxide and copper method, if the cuprous oxide be weighed as such, the same values would be obtained as by Allihn's method. The author finds higher values than Rusham for the amounts of copper as cuprous oxide corresponding to the glucose. The "copper-value" decreases relatively with the increase in the amount of sugar, corresponding as follows in mgrms. :—

| | | | | | |
|---|---|---|---|---|---|
| Sugar……. | 12 | 26 | 51 | 76 | 101 |
| Copper…… | 32·8 | 62·1 | 112·8 | 163·8 | 214·3 |
| Sugar……. | 126 | 151 | 176 | 201 | 226 |
| Copper…… | 263·7 | 310·7 | 354·6 | 396·1 | 435·3 |

—A. S.

*Methyl Alcohol, Determination of, in Mixtures with Ethyl Alcohol.* A. Lam. Zeits. f. angew. Chem. 1898, 125—130.

The principle of the author's method consists in preparing from the mixed alcohols the iodides, and determining the specific gravity of the mixed iodides, whence is calculated the proportion of each present. First attempts to prepare and purify the iodides in the usual way from mixtures of the 95 per cent. alcohols showed that the proportions of the iodides were very different from those of the alcohols (the methyl iodide being relatively too high), and that during the purifying distillation there was considerable loss, especially of methyl iodide. The pure iodides were now prepared from 50 per cent. alcohols, the inner tube of the condenser being drawn out to dip under water in the receiver, which was tubulated and provided with a stop-cock so that the iodide could be ultimately run off. The distillate was thoroughly washed in the receiver, first with weak soda and then with water; the iodide was then exactly run off, and shaken with calcium chloride. Density determinations, before and after redistillation, gave identical figures, showing that redistillation was not necessary. Mixtures of the two pure iodides in known proportions were now made, and density determinations showed that no contraction takes place on mixture. The tables following show the densities for varying percentages by weight and by volume of methyl iodide. Experiments were now undertaken with spirits containing 50 per cent. of water, the remaining 50 per cent. being made up of methyl and ethyl alcohols in varying proportions, ranging from 1 : 4 up to 4 : 1; the iodides were formed, washed, and dried as already described, and the weight (or volume) and density of the resulting mixed iodides determined. The amount of methyl iodide so obtained, calculated back to its equivalent amount of methyl alcohol, gave results varying from 96·5 to 102 per cent. of the truth, as long as the ratio of methyl alcohol to ethyl alcohol was not greater than 3 : 2, but with more methyl alcohol, this degree of accuracy is no longer attainable. In all cases, redistillation was found to cause a serious loss of methyl iodide. Though the amount of iodide used was always considerably greater than that needed to convert the whole of the alcohols into iodides, the amount of ethyl iodide obtained was never more than 90 per cent., usually only 60 or 70 per cent., of that corresponding with the whole of the ethyl alcohol present. The author concludes that the process is very satisfactory as far as the direct determination of methyl alcohol is concerned, and that it can be applied to mixtures containing larger proportions of methyl to ethyl alcohol than 3 : 2, by first diluting a known amount of the mixture with a known amount of ethyl alcohol. He has also shown that it is applicable to commercial spirits (Hollands gin, for example). He uses in an experiment, 40 c.c. of spirit (containing about 40—50 per cent. alcohol), 64 grms. of iodine, and 8 grms. of red phosphorus.

DENSITIES OF MIXTURES (BY VOLUME) OF ETHYL IODIDE WITH METHYL IODIDE.

| Per Cent. by Volume of $CH_3I$. | Showing Specific Gravities for Increments of 1 per Cent. of Methyl Iodide. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0. | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. | 9. |
| 0 | 1·9444 | 1·9476 | 1·9509 | 1·9541 | 1·9573 | 1·9606 | 1·9638 | 1·9670 | 1·9703 | 1·9735 |
| 10 | 1·9767 | 1·9800 | 1·9832 | 1·9865 | 1·9897 | 1·9929 | 1·9962 | 1·9994 | 2·0027 | 2·0059 |
| 20 | 2·0091 | 2·0123 | 2·0155 | 2·0188 | 2·0220 | 2·0252 | 2·0284 | 2·0317 | 2·0349 | 2·0381 |
| 30 | 2·0414 | 2·0446 | 2·0479 | 2·0511 | 2·0543 | 2·0575 | 2·0608 | 2·0640 | 2·0672 | 2·0705 |
| 40 | 2·0737 | 2·0770 | 2·0802 | 2·0834 | 2·0867 | 2·0899 | 2·0932 | 2·0964 | 2·0996 | 2·1029 |
| 50 | 2·1061 | 2·1093 | 2·1125 | 2·1157 | 2·1190 | 2·1222 | 2·1254 | 2·1287 | 2·1319 | 2·1351 |
| 60 | 2·1384 | 2·1416 | 2·1448 | 2·1481 | 2·1513 | 2·1545 | 2·1578 | 2·1610 | 2·1642 | 2·1675 |
| 70 | 2·1707 | 2·1739 | 2·1772 | 2·1804 | 2·1836 | 2·1869 | 2·1901 | 2·1933 | 2·1966 | 2·1998 |
| 80 | 2·2030 | 2·2062 | 2·2094 | 2·2127 | 2·2159 | 2·2191 | 2·2224 | 2·2256 | 2·2289 | 2·2321 |
| 90 | 2·2354 | 2·2386 | 2·2418 | 2·2451 | 2·2483 | 2·2515 | 2·2548 | 2·2580 | 2·2612 | 2·2644 |
| 100 | 2·2677 | .. | .. | .. | .. | .. | .. | .. | .. | .. |

DENSITIES OF MIXTURES (BY WEIGHT) OF ETHYL IODIDE WITH METHYL IODIDE.

| Per Cent. by Weight of $CH_3I$. | Showing Specific Gravities for Increments of 1 per Cent. of Methyl Iodide. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0. | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. | 9. |
| 0 | 1·9444 | 1·9472 | 1·9499 | 1·9527 | 1·9555 | 1·9583 | 1·9612 | 1·9640 | 1·9668 | 1·9697 |
| 10 | 1·9725 | 1·9754 | 1·9782 | 1·9811 | 1·9840 | 1·9869 | 1·9898 | 1·9927 | 1·9956 | 1·9985 |
| 20 | 2·0015 | 2·0044 | 2·0073 | 2·0103 | 2·0133 | 2·0163 | 2·0192 | 2·0222 | 2·0252 | 2·0282 |
| 30 | 2·0313 | 2·0343 | 2·0373 | 2·0404 | 2·0434 | 2·0465 | 2·0496 | 2·0527 | 2 0558 | 2·0589 |
| 40 | 2·0620 | 2·0651 | 2·0682 | 2·0714 | 2·0745 | 2·0777 | 2·0808 | 2·0840 | 2·0872 | 2·0904 |
| 50 | 2·0936 | 2·0969 | 2·1001 | 2·1033 | 2·1066 | 2·1099 | 2·1131 | 2·1164 | 2·1197 | 2·1230 |
| 60 | 2·1263 | 2·1296 | 2·1329 | 2·1363 | 2·1397 | 2·1431 | 2·1464 | 2·1498 | 2·1531 | 2·1565 |
| 70 | 2·1599 | 2·1633 | 2·1667 | 2·1702 | 2·1737 | 2·1772 | 2·1807 | 2·1842 | 2·1877 | 2·1912 |
| 80 | 2·1947 | 2·1982 | 2·2017 | 2·2053 | 2·2089 | 2·2125 | 2·2161 | 2·2197 | 2·2233 | 2·2270 |
| 90 | 2·2306 | 2·2343 | 2·2379 | 2·2416 | 2·2453 | 2·2490 | 2·2527 | 2 2564 | 2·2601 | 2·2639 |
| 100 | 2·2677 | .. | .. | .. | .. | .. | .. | .. | .. | .. |

—J. T. D.

*Wine, Estimation of Potassium Bitartrate in.* L. Magnier de la Source. Rev. intern. falsific. **10**, 195; Chem. Centr. 1898, **1**, [2], 149.

A SOLUTION (S) is prepared of 1 grm. of potassium bitartrate in 100 c.c. of water and 10—12 c.c. of alcohol, to which is added an excess of pure potassium sulphate, and the whole filtered. 10 c.c. of the wine to be examined are evaporated to dryness over concentrated sulphuric acid, and 10 c.c. of solution (S) are allowed to drop on the residue, thus extracting all the substances soluble in dilute alcohol,

with the exception of potassium bitartrate and potassium sulphate. The mixture is filtered, and the filter washed with the solution (S). The residue is finally dissolved in warm water, and the acidity titrated with baryta water. An alternative method is to add 1·5 grms. of potassium sulphate to 100 c.c. of the wine, and then evaporate till the volume is reduced to 15 c.c. Let stand for 48 hours, decant off through a small filter, and wash out the crystals with the solution (S). Finally titrate with baryta water as before.
—A. S.

### *Glycerophosphates, Titration of.* Falières. Répert. Pharm. 1898, **10**, 8.

The usual method for estimating glycerophosphates consists in determining the phosphoric acid in the ash, but the author proposes to titrate commercial syrupy glycerophosphoric acid, which usually contains about 50 per cent. of acid, with decinormal caustic potash, using phenolphthalein as an indicator. Two equivalents of the base saturate one equivalent of the acid, and hence 1 c.c. of decinormal potash is equal to 0·0086 glycerophosphoric acid. In determining calcium glycerophosphate, the sample must not contain any free acid (phosphoric, sulphuric, hydrochloric, carbonic), must be free from barium and lead, and have a solubility of at least 5 per cent. The determination is carried out by dissolving 0·2 grm. of the salt in a little water, adding 20 c.c. of decinormal oxalic acid, and making up to 100 c.c. 50 c.c. of the filtrate, containing the free glycerophosphoric acid, and an excess of oxalic acid, are titrated with decinormal potash and phenolphthalein, and twice the number of c.c. required = A. The calcium oxalate remaining on the filter after well washing with boiling water, is dissolved in dilute nitric acid (1 : 10), a few drops of sulphuric acid are added, and the solution is titrated with decinormal permanganate = B The quantity of lime is expressed by B × 0·0028, and for 100 grms. of glycerophosphate = B × 0·0028 × 5 × 100. The formula A + B − 20 gives in c.c. of decinormal potash, the weight of the glycerophosphoric acid contained in 0·2 grm. of the substance, whilst (A + B − 20) × 0·0086 × 5 × 100 gives the percentage amount of glycerophosphoric acid in the calcium glycerophosphate.—T. A. L.

### *The Glycerophosphates, Determination of.* Adrian and Trillat. J. Pharm. Chim. 1898, **7**, [4], 163—165, and [5], 225—226. (See also this Journal, 1898, 66, 73, 78.)

When sulphuric acid is added to a cold solution of the neutral glycerophosphate of an alkaline earth in the presence of Helianthin, the coloration appears when half a mol. of acid has been used, *i.e.*, when the normal salt has been changed into the acid salt. A process may be founded upon this for the determination of normal glycerophosphates in the absence of phosphates and acid glycerophosphates.

The authors find that free glycerin does not interfere with the ordinary use of indicators. Free phosphoric acid may be determined with phenolphthalein or Helianthin in the presence of a normal glycerophosphate. A mixture was made containing known amounts of phosphoric acid, an acid glycerophosphate, and a normal glycerophosphate. The alkali required for neutralisation with either of the indicators mentioned, was the sum of the quantities required by the different constituents. Thus, in dilute solution, phosphoric acid may be determined in the presence of acid and neutral glycerophosphates; and, conversely, glycerophosphoric acid may be determined in the presence of free phosphoric acid by subtracting double the acidity towards Helianthin from the acidity towards phenolphthalein.
—A. C. W.

### *Antipyrine, Action of Iodine upon: Application to the Determination of Antipyrine and of Iodine.* J. Bougalt. J. Pharm. Chim. 1898, **7**, [4], 161—163.

In alcoholic solutions containing corrosive sublimate, 1 mol. of antipyrine absorbs exactly 1 mol. of iodine. The proportion of corrosive sublimate may vary within somewhat wide limits without influence on the result. Solutions are prepared of 1·351 grms. of iodine, 2·5 grms. of mercuric chloride, and 1 grm. of antipyrine, each in 100 c.c. of alcohol of 95°. To 20 c.c. of the antipyrine solution, 20 c.c. of sublimate solution are added, and then the iodine solution, until a permanent yellow coloration is produced. The number of c.c. of solution used, multiplied by 5, gives the percentage of antipyrine in the sample.

The author proposes to utilise this process for the determination of iodine. Antipyrine is easily obtained pure, and easily purified; its solutions do not alter: in these respects it is to be preferred to thiosulphate. The order of addition of the solutions is immaterial: the antipyrine solution may be added to the mixture of iodine and sublimate solutions until the colour disappears. (See also this Journal, 1897, 266.)—A. C. W.

### *Rose Oil, Testing of.* P. N. Raikow. Chem. Zeit. 1898, **22**, [17], 149—150.

The statement recently published by F. Dietze (Chem. Zeit. Repert. 1897, **21**, 288, after Sudd. Apoth. Zeit. 1897, [89]), that the determinations of the acid and ester number of rose oil furnish data of analytical value, is refuted by the author. Dietze stated that a pure rose oil should possess the following characters:—

(1.) Specific gravity not above 0·870 at 15° C.

(2.) Congealing point not lower than 15° to 20° C.

(3.) Optical rotation in 100-mm. tube at 20° C. not more than 1·30°.

(4.) The saponification number (total acid number) not higher than 9·5 to 10, and the ratio of the free acid and ester numbers not exceeding 1 : 7.

The author has applied Dietze's test to eight authentic samples of pure rose oil, and has obtained figures entirely at variance with those given by that worker. Only one oil, the same that was used by Dietze in his experiments, gave figures approaching those attributed by him to pure rose oil. The others all gave results much in excess of Dietze's standards. The author therefore considers the figures obtained by saponification to possess but little value. Moreover, although the number obtained with African and Turkish geranium oil is markedly higher than that of rose oil, the ratio between the free acid and ester values of the former falls well within the limit given by Dietze for pure rose oil. Exception is also taken to the determination of the "congealing point" of rose oil; the temperature at which the first crystal of stearoptene becomes apparent being regarded by the author as of more value. This, indicating the temperature at which the eleoptene is saturated with the stearoptene, is termed the "saturation point," and is determined as follows:—10 c.c. of the sample are placed in a tube of 15 mm. diameter, a thermometer is then immersed in the fluid so that it may be freely moved without touching either the bottom or the sides. The tube is then warmed in the hand, the oil well shaken, and allowed to cool slowly. The temperature at which the first crystal appears is regarded as the saturation point. This was found to range from 11·5° C. to 27·2° C. in the genuine samples of otto examined.—J. O. B.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

### *Beryllium, Preparation by Electrolysis.* P. Lebeau. Comptes Rend. **126**, (10), 744—746.

The double fluoride of beryllium and sodium, $BeF_2.NaF$ or $BeF_2.2NaF$, is prepared by dissolving the requisite quantities of beryllium hydroxide and sodium carbonate in hydrofluoric acid. The solution is evaporated to dryness and the salt fused. In the electrolysis, a nickel crucible serves as cathode and a rod of graphite carbon as anode; a current of 6 or 7 ampères at about 40 volts was used. After three-quarters of an hour a metallic deposit is obtained on the bottom of the crucible, which, after cooling and well boiling out with water, falls to a powder of irregular crystals, unmixed with any amorphous substance. This powder is pure beryllium, quite free from nickel and from iron.—J. T. D.

### *Mercury, Action of Sulphuric Acid on.* J. R. Pitman. J. Amer. Chem. Soc. 1898, 20, [2], 100—101.

FROM the author's experiments it is apparent that there is no reaction between mercury and sulphuric acid at the ordinary temperature, and if Baskerville and Miller observed a reaction, as they state (*ibid.*, **19**, 874), it must have been by means of some different method. (See also this Journal, 1897, 1051.)—L. J. de W.

### *Ammonium Peroxide.* P. Melikoff and L. Pissarjewsky. Ber. 1897, 30, [19], 3144—3146.

IN this preliminary communication, the authors describe the preparation of ammonium peroxide by the action of a concentrated ethereal solution of hydrogen peroxide on an ethereal solution of ammonia at −20° C. The ammonium peroxide separates out as a heavy, viscous liquid, a greater yield being obtained, the lower the temperature. The characteristic smell of ammonia disappears, but returns again after careful washing with cooled ether. This is ascribed to the dissociation of the compound.

The new compound produces the same irritation of the skin as hydrogen peroxide does. By the action of dilute sulphuric acid, hydrogen peroxide is formed. An aqueous solution of the substance decomposes with evolution of oxygen and formation of a small quantity of ammonium nitrite.

### *Ammonium Peroxide.* P. Melikoff and L. Pissarjewsky. Ber. 1898, 31, [2], 152—154.

ON the addition of a large excess of ethereal solution of ammonia to a solution of hydrogen peroxide in the same solvent, followed by immersion in a freezing mixture of calcium chloride and snow, a white crystalline mass is obtained. The crystals, after washing with cooled ether and drying on a porous plate at a low temperature, possess the composition represented by the formula—

$$(NH_4)_2O_2 + H_2O_2 + H_2O.$$

Ammonium peroxide is very unstable; it liquefies at the ordinary temperature, dissociating into ammonia and hydrogen peroxide. This dissociation is followed by evolution of oxygen and formation of small quantities of ammonium nitrite. Nitric acid does not appear to be produced in this oxidation of ammonia.

Ammonium peroxide has an alkaline reaction, absorbs carbonic acid from the air, acts on potash with production of potassium peroxide and ammonia, is soluble in alcohol, almost insoluble in cold ether, and less soluble still in petroleum naphtha at −30° C. In contact with manganese peroxide, oxygen is evolved. Ammonium peroxide dissolves uranium tetroxide with formation of the perurananate—

$$(NH_4)_2O_2 . (UO_4)_2.$$

—A. C. W.

### *Chloroform, The Partial Decomposition of, in the Organism.* Desgrez and Nicloux. Comptes Rend. 126, [10], 758—760.

ST. MARTIN has suggested that the increased amount of carbon monoxide in the blood of animals which have been anæstheticised with chloroform, arises from the action of acetic acid on the blood. The authors find that normal blood of dogs contains a small amount (about 1·5 c.c. per litre) of carbon monoxide, but that this increases, in the same animal three or four fold when the animal has been for some time under the influence of chloroform, whilst no such increase takes place when the anæsthesia is produced by ether. They conclude that the increase is directly due to decomposition of the chloroform, with production of carbon monoxide.—J. T. D.

### *Xanthine, Synthesis of, starting from Hydrocyanic Acid.* A. Gautier. Ber. 1898, 31, [4], 449—450.

E. FISCHER (Ber. 1897, 3151), repeating an investigation of the author's, has obtained a substance very similar to xanthine, but which does not give the murexide reaction. When hydrocyanic acid is heated with water and acetic acid, it is mainly converted into azulmin; xanthine and methylxanthine are only formed in small quantity. The xanthine so obtained was identified by analysis and a study of its reactions.—A. C. W.

### *Aconitine, Diacetylaconitine, Benzaconine, and Aconine; Pharmacology of, considered in Relation to their Chemical Constitution.* J. Theodore Cash and Wyndham R. Dunstan. Proc. Royal Soc. 1898, 62, [385], 338—347.

THE action of diacetylaconitine was not found to differ in character from that of aconitine, but was less intense, the addition of the two acetyl groups merely weakening the characteristic action of the parent alkaloid. The removal of the acetyl group from aconitine, as seen in benzaconine, however, was followed by a complete modification of the pharmacological action; the toxic power was so far modified that benzaconine scarcely falls within the category of poisons. Its action on the heart was quite different from that of aconitine, and, to a certain extent, antagonistic to that alkaloid. Thus aconitine caused the ventricles of the heart to beat more rapidly than, and often independently of, the auricles; the opposite was the case with benzaconine. The latter may, to a certain extent, be regarded as an antidote to the former—a point of considerable pharmacological importance, since benzaconine and aconine are present in varying proportions in *Aconitium napellus*, the source of most of the medicinal preparations of aconite. The removal of the acetyl group also eliminated the stimulating effects of aconitine on the respiratory centres. Benzaconine, however, peripherally depressed the activity of motor nerve endings, and in a lesser degree, that of skeletal muscle, whilst aconitine acted chiefly on the sensory nerve terminations.

The removal of the benzoyl group as represented by aconine, resulted in a complete change in the pharmacological action, since aconine was found to be a heart tonic; so much so, that the administration of small doses of aconine averted the lethal effect of toxic doses of aconitine. It had, moreover, a curare-like effect on the motor nerve endings, which was not observed with either aconitine or benzaconine, nor did it disturb the normal sequence of the cardiac pulsations. Aconine cannot be regarded as a poisonous alkaloid, very large doses being required to cause death.

From these data it is evident that the extraordinary toxic effect of aconitine depends in a great measure on the presence of the acetyl radical, while the activity of benzaconine is dependent in a lesser degree on the presence of the benzoyl radical. The inactivity of the base aconine, denuded of both acetyl and benzoyl groups, is a noteworthy point. From a practical point of view, the demonstration of the antagonistic action between aconitine on one hand, and benzaconine and aconine on the other hand, will throw light upon the mode of action of the alkaloids of *aconitum napellus*.—J. O. B.

### *Sulphur, Note on the Volatility of.* T. C. Porter. Proc. Chem. Soc. 1898, [191], 65.

DURING the analysis of some pumice rock from Teneriffe, the author was led to the conclusion that sulphur is volatile at 100°. Several glass tubes were carefully cleaned, into which specimens of sulphur of varying physical condition and purity were placed. Some of the tubes were exposed to the air, others were exhausted. After being exposed to a temperature of 100° for some minutes, it was observed that, as was to be expected, the better the vacuum, the more rapid is the sublimation of the sulphur. The sublimate consists at first of very pale yellow drops, which possess little viscosity, and remain unchanged in some cases even at 10°, for days. Rhombic octahedra and prisms of sulphur are occasionally formed together; the former crystals sometimes grow at the expense of the drops. Sulphur does not yield a perceptible sublimate at ordinary temperatures, in a "good vacuum," even after a year. The two forms $S\alpha$ and $S\beta$ therefore have their transition point between 90° and 100°.

## New Books.

QUANTITATIVE CHEMICAL ANALYSIS BY ELECTROLYSIS. By Dr. ALEXANDER CLASSEN, Privy Councillor, Professor of Electro-Chemistry and Inorganic Chemistry in the Royal School of Technology at Aachen, in co-operation with Dr. WALTER LÖB, Lecturer on Electro-Chemistry in the Royal School of Technology at Aachen. Authorised translation, Third English from the revised and greatly enlarged Fourth German edition. By WILLIAM HALE HERRICK, A.M., and BERTRAM B. BOLTWOOD, Ph.D. John Wiley and Sons, New York. Chapman and Hall, Limited, London. 1898. Price 3.00 dols.

8vo volume, containing 286 pages of subject-matter, illustrated with 86 wood engravings. The text is classified as follows:—I. Determination of Current Magnitudes. 1. Measurement of the Current Strength. 2. Measurement of the Tension. Sources of Current. Primary Galvanic Elements. Secondary Galvanic Elements (Accumulators or Storage Batteries). Physical Methods of Producing the Current. Regulation of the Current. Process of Analysis, &c. II. Quantitative Determination of the Metals. Determination of the Halogens. Separation of the Metals. APPENDIX.—Some Applied Examples of Electro-Chemical Analysis. Tables for Calculation of Analysis. Reagents.

SCIENCE ABSTRACTS: PHYSICS AND ELECTRICAL ENGINEERING. *Hon. Editor*, J. SWINBURNE; *Sub-Editor*, W. R. COOPER; with Committee and Staff of Abstractors. Issued under the direction of the Institution of Electrical Engineers and The Physical Society of London. Taylor and Francis, Red Lion Court, Fleet Street, London. 1898. Price to Non-Members, 3*s.* per number, or 36*s.* post free per annum.

THIS is a new journal in which abstracts are properly classified, and given, of the various branches of Physics and Electrical Engineering. The classification is as follows:—General Physics. Light. Heat. Sound. Electricity. Electro-Chemistry and Chemical Physics. General Electrical Engineering. Dynamos, Motors, and Transformers. Power Distribution, Traction, and Lighting. Telegraphy and Telephony.

## Trade Report.

### CUSTOMS DECISIONS.

#### UNITED STATES.

Refined carbonate of potash is free as carbonate of potash under paragraph 595 of the Act of 1894.

Antitoxine is dutiable as a medicinal preparation at the rate of 25 per cent. *ad val.* under paragraph 59 of the Act of 1894.

Camphor oil is assessed for duty at the rate of 25 per cent. *ad val.* as an essential or distilled oil under paragraph 60 of the Act of 1894.

### EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

#### U.S. MISCELLANEOUS METAL IMPORTS IN 1896 AND 1897.

*Bd. of Trade J., April* 1898, 416.

| — | 1896. | 1897. |
|---|---|---|
| | Dols. | Dols. |
| Manganese ore | 31,523 | 340,245 |
| Bronze manufactures | 168,884 | 526,741 |
| All other metals and metal compositions | 3,092,937 | 3,543,827 |
| Plumbago | 437,189 | 270,952 |
| Tin | 5,848,263 | 7,415,133 |
| Spelter | 25,904 | 95,883 |
| Antimony ore | 170,032 | 167,373 |
| Antimony regulus | 5,294 | 46,955 |
| Brass and manufactures | 157,798 | 85,341 |
| Copper ore and regulus | 348,629 | 816,540 |
| Pigs and bars | 981,920 | 1,451,016 |
| Manufactures of copper | 85,123 | 78,897 |

#### U.S. EXPORTS OF MISCELLANEOUS METAL ARTICLES.

*Bd. of Trade J., April* 1898, 418.

| — | Values. | |
|---|---|---|
| | 1896. | 1897. |
| | Dols. | Dols. |
| Agricultural implements | 4,643,729 | 5,302,807 |
| Brass and manufactures | 1,026,191 | 1,346,802 |
| Cars for steam roads | 834,288 | 514,528 |
| Cars for other roads | | 763,075 |
| Carriages and vehicles | 1,912,482 | 1,850,779 |
| Cycles and parts of | 3,796,022 | 6,902,735 |
| Copper ore and matte | 2,393,914 | 1,190,029 |
| Copper | 27,822,280 | 30,597,645 |
| Copper, manufactures of | 819,017 | 958,379 |
| Nickel, nickel oxide, and matte | 606,833 | 997,391 |
| Plated ware | 385,824 | 471,528 |
| Quicksilver | 618,437 | 394,549 |
| Tin, manufactures of | 268,581 | 284,020 |
| Zinc ore | 47,108 | 211,350 |
| Spelter | 1,013,620 | 1,356,538 |

#### GREAT BRITAIN AND GERMANY.—EXPORTS OF CHEMICALS, MEDICAL PREPARATIONS, &c., IN 1,000*l.*

*Foreign Office Annual Series, No.* 2040, *March* 1898.

| Year. | Great Britain. | German Empire. | Increase or Decrease for England. | Increase or Decrease for Germany. |
|---|---|---|---|---|
| | £ | £ | £ | £ |
| 1895 | 8,288 | 14,940 | .. | .. |
| 1896 | 8,242 | 15,839 | − 46 | + 899 |
| 1897 | 8,674 | 16,336 | + 432 | + 497 |

#### ESPARTO EXPORTS FROM TRIPOLI.

*Foreign Office Annual Series, No.* 2048, *April* 1898.

In 1897 the export of esparto was 37,200 tons from the port of Tripoli, Khoms, and Zeliten, all of which went to England, showing an increase of 4,610 tons over the previous year. Values, however, remained the same, owing to lower prices in England, the result of wood-pulp entering so largely in the making of paper. Owing to the impending good harvest, the quantity for export in 1898 is likely to be small, but the quality good. Freights fluctuated during the year from 11*s.* to 15*s.* per ton for hydraulic-pressed bales.

#### DRUGS AT LAS PALMAS.

*Foreign Office Annual Series, No.* 2036, *p.* 19.

*Drugs.*—Duty free. Octroi free. Free port, 1 per mil. Discharging, 10 per cent. on freight. Cartage, 1*s.* 6*d.* for 10 cwt. or less.

Special permission and membership of the Spanish Pharmaceutical Society is required for the sale of certain drugs and for medicines. The trade is generally worked through agents in Barcelona, but German houses send out price lists in Spanish weight and metric currency, one of which has been sent to the Association of Chambers of Commerce for reference. The result is an increase of business.

#### FERTILISERS AT LAS PALMAS.

*Foreign Office Annual Series,* 1898, *No.* 451, *p.* 7.

Manure, both natural and artificial, is very extensively employed all over the island, the artificial being sold at about 10*s.* per 100 lb. laid down here, and the import tables show that the money spent abroad in the purchase of ingredients, including freight, amounts to over 27,000*l.* in two years. These ingredients are prepared here by merchants, who compound the different mixtures for the various soils, and sell them on credit, to be paid for when the crop is gathered. These merchants are generally the contractors for the crop. The artificial manures are most largely used in farms devoted to the cultivation of sugar cane and bananas, the nature of this cultivation not permitting of a green crop being planted during the year to provide fodder for the animals sufficient to produce natural dressing.

### IMPORTS OF CERTAIN ARTICLES OF BRITISH MANUFACTURE, &C. DURING THE YEARS 1895–97, INTO HAVRE.

*Foreign Office Annual Series*, 1897, *No.* 2034, *p.* 16.

| Articles. | Quantity. | | |
|---|---|---|---|
| | 1895. | 1896. | 1897. |
| | Tons. | Tons. | Tons. |
| Artificial manures | 20 | 61 | 449 |
| Coals | 581,061 | 592,457 | 654,853 |
| Coal-tar | 6,002 | 7,497 | 10,827 |
| Boracic acid | 66 | 8 | 1 |
| Caustic soda | 64 | 47 | 14 |
| Bicarbonate of soda | 36 | 10 | 54 |
| Crude salts of ammonia | 28 | 384 | 50 |
| Chloride of lime | 11 | 16 | 35 |
| Superphosphate of lime | 1,637 | 1,659 | 540 |
| Sulphate of copper | 55 | 10 | 50 |
| Oil paints | 53 | 54 | 46 |

### BUTTER ADULTERATION AND PRESERVATION IN ITALY.

*Foreign Office Annual Series*, 1898, *No.* 450, *p.* 7.

There is no special law for the protection of the dairy industry, except a law dated July 19, 1894, which regulates the sale of the substitutes of butter, and prohibits and punishes the adulteration of butter with margarine. Besides that law, there is a special service of surveillance in in the principal cities, and public officers for the chemical analysis of all alimentary products.

Generally, no preservative is employed but common salt, which is added to butter when exported. Some large exporters of Venetia and Lombardy, however, employ a special salt obtained from the mines of Lungro (Calabria Citeriore), which, although more adapted than common salt, is said to be still inferior to the salts of Egestorff, Lüneburg, Stade, and Linden, employed abroad for foreign cheeses of a superior quality. Its chemical composition is the following :—

LUNGRO SALT.*

| | Per Cent. |
|---|---|
| Chloride of sodium | 97·69 |
| Sulphate of calcium | 0·66 |
| Chloride of magnesia | 0·05 |
| „ calcium | 0·15 |
| Sulphate of sodium | 1·40 |
| Water | 0·21 |
| Insoluble matter | 0·44 |

* This salt is found in blocks and ground for use.

### PHOSPHATES AND BORACIC ACID AT VENICE.

*Foreign Office Annual Series*, *No.* 2037, *March* 1898, *p.* 14.

So far, preference has been given by agriculturists in these provinces to British phosphates, because they contain phosphoric acid soluble in water. Those imported from Belgium and France contain phosphoric acid only soluble in citrate. English produce would appear to have in future to compete with a composition manufactured by Signor Angelo Scaini, of Udine, who claims to have succeeded in manufacturing superphosphates soluble in water similar to those of English origin, which also contain a certain quantity of nitrogen.

A project has lately been presented to the Italian Parliament for the abolition of the export duty on the boracic acid which is found here. It is alleged that in consequence of the export duty, the boracic products manufactured in Italy have suffered considerably, and the imposition of a duty on the importation of foreign borax and refined boracic acid, which, so far, are admitted free of duty, was urged. These products are chiefly imported from England.

### ARTIFICIAL MANURES AT CHERBOURG.

*Foreign Office Annual Series*, 1897, *No.* 2035, *p.* 6.

There is a considerable trade at Cherbourg in artificial manures. During 1897, British imported manures held their own and maintained their prices by their superior quality, notwithstanding the attempts of the Franco-Belgian syndicate to close French markets to them. The Franco-Belgian firms are selling at a loss, some firms having already broken down under the strain. Norman farmers are very conservative and keep to a good thing until they find a better. A year's trial of continental superphosphates in many cases has proved unsatisfactory. The uneven quality of manures from Germany (viâ Antwerp), Belgium, and the north of France alike is complained of. A uniform percentage of phosphoric acid cannot be counted upon.

The Consul looks forward to a very slow but sure advance in British imports under this head. A very recent cargo of special potato manure went from England to Barfleur direct. That district is the centre of the early potato export industry.

The use of artificial manures for pasture, potatoes, and market gardening is gradually extending through this Consular district.

A small reduction in prices would be followed by some increase in the demand for English-made superphosphates.

Freights from London, 5*s.* 6*d.*; from Liverpool, 7*s.*; from Hull, 8*s.* 6*d.*, are now ruling.

### SULPHATE OF COPPER AT PATRAS.

*Foreign Office Annual Series*, *No.* 2055, *April* 1898.

About 4,000 tons of sulphate of copper, principally of British manufacture, were imported during the past year. It is extensively used against the malady of the vines, named "peronosporos, and since grape and currant vineyards in this district suffered so severely last year, all growers are preparing to adopt the same precautions against the malady. Sulphate of copper is being sold retail at 20*l.* a ton.

The Greek Government intends to take off the import duty on copper in order to encourage the manufacture of sulphate of copper in this country.

### TAX ON BORAX, &C.

*Chem. and Druggist*, *April* 23, 1898, 679.

The proposal to remove the tax from Italian boric acid has not been successful, as on March 18th it was finally agreed to tax pure boric acid at the rate of 5 liras per quintal (4*s.* per 2 cwt.), and borax or other sodium borates at the rate of 4 liras per quintal. The French Chamber on April 4th modified the duty on boric acid, so that the natural product from Tuscany, containing 15 per cent. or more of impurity, will be exempt from duty; other boric acid will be subject to a tax of 18 francs per 100 kilos. on the general tariff, or 12 francs per 100 kilos. on the minimum tariff.

### TRADE OF SAMOA FOR 1897.

*Foreign Office Annual Series*, *No.* 2094, *April* 1898.

There is a considerable sale for patent and other medicines such as Epsom salts, Painkiller, and the like.

English or colonial brands of white lead and paint hold their own here, and are much superior to the American and German brands.

British traders can purchase German linseed oil in Samoa cheaper than the British article can be purchased in Australia.

American yellow soap has a large sale. It is put up in cases of 32 bars, containing a nominal hundredweight, and cost 16*s.* a case landed in Samoa, while it is retailed at 1*s.* a bar. It is harder than the bar soap manufactured in Australia.

Australian and New Zealand brands of yellow soap hold their own in the market. The commoner sorts cost about 14*s.* a case of 44 smaller bars, landed in Samoa. The bar, which is only two-thirds the size of the American, is retailed at 6*d.* The Crown brand, a favourite bar soap of high character manufactured in Sydney, costs about 19*s.* per case landed in Samoa.

There is a market for perfumery of a cheaper kind, although Atkinson's, Piesse and Lubin's, Breidenbach's, and good French makers are to be met with.

A great deal of the starch sold is of German manufacture put up with English labels. The natives will not buy starch in bulk; it must be put up in 1 lb. packets or boxes.

German and American starch is far inferior to the English sold here.

British firms nowadays seem to expect to have markets found for them instead of finding them for themselves as in the past, and to have the most elementary information upon trade matters supplied to them.

The British Consular service has proved itself ready and able to meet these requirements. But it is obvious that in pointing out these markets and in giving this information, the markets in question are really spoilt and rendered useless, for whereas one firm finding out the market for itself might make good profits, those profits dwindle to zero if they are to be shared by a hundred other British firms.

The remedy lies in the employment of really efficient commercial travellers, or, better still, travelling partners. One civil, ingratiating, well-informed traveller is able to do more in one day than five years of circulars can ever accomplish.

Every British trader here has the same story of the refusal of British manufacturers and shippers in England to meet local requirements or to depart from old methods.

British trade will suffer far more severely during the next five years from German competition than it has done already, unless British manufacturers attack the colonial and foreign markets in the energetic, well-organised, up-to-date, and conciliatory manner adopted by the Germans.

Foreign markets, such as Samoa, do not require expensive, durable, and heavy articles. Cheap attractive lines of good appearance, with such quality and durability as may be possible for the money, is what are required.

It is simply suicidal for British manufacturers to ignore the warnings repeatedly issued under these heads.

## TRADE OF BORDEAUX IN 1897.

*Foreign Office Annual Series, No.* 2051, *April* 1898.

1,536 tons of iron ore were exported to Great Britain.

Of chemical products, the principal articles exported to the United Kingdom were chestnut and other vegetable tannin extracts, 59,511 cwt.; glycerin, 2,076 cwt.; cream of tartar, 38,648 cwt.; raw tartar, 12,626 cwt.; and oxide of zinc, 1,836 cwt.

While there was a great decrease in the total importation of chemical products, there was a very substantial increase in them from the United Kingdom, the principal items being sulphate of copper, 263,155 cwt.; superphosphate of lime, 39,980 cwt.; chemical manures, 52,990 cwt.; and coal-tar products, 195,779 cwt.

All the ink (57 cwt.) and all the blacking (73 cwt.) imported comes from the United Kingdom. The principal article of pottery imported is common fire earthenware, 17,066 cwt., while there is hardly any demand for fine earthenware or porcelain. The principal item under glass is empty bottles, amounting to 4,548 cwt., followed by full bottles, 1,340 cwt.

The total quantity of sulphate of copper imported during 1897 was about 13,700 tons, and was almost entirely of British manufacture. The season, however, was not quite so good as the previous one, and though prices opened pretty firmly they were not maintained later on. With the month of May a downward tendency set in which lasted till the end of the year. This fall in prices, just at the principal moment of consumption, was brought on by over-speculation, and by comparatively limited sales compared with 1896. The weather was not favourable, and cryptogamic diseases of the vine did much damage; many vineyards treated after the appearance of the disease could not be saved, sulphate of copper being a preventive and not a cure. The consumption in 1897 was thus nearly 2,400 tons less than in 1896; the trade was able nevertheless to realise fair profits. It appears that sulphate of copper has but a very limited effect against the "black rot," and the department of the Gers had a very bad vintage on account of the ravages perpetrated by this fearful disease, against which no remedy has yet been found effective. Many trials, even with mercurial preparations, have been made, and will be continued during 1898, so it may be expected that growers will soon be able to master this disease. Many vine growers, however, are disheartened, even ruined, and the general aspect is far from being satisfactory.

The demand for artificial manures is steadily increasing, and no doubt prices leave a fair margin of profit to the importers.

The situation, as regards dyewood, is somewhat better for 1897, imports having increased over 1896, with better prices all round. Imports of furniture woods were also on the increase.

## REPORT ON ALLEGED ADULTERATION OF BRITISH PRODUCE IN BRAZIL.

*Foreign Office Annuel Series, No.* 2058, *April* 1898.

"Until quite recently, a very unsatisfactory feature of Brazilian trade has been the ease and impunity with which goods could be offered for sale under false marks imitating those of well-known European makers.

"No repressive measures of an effective nature were, however, put in force by the administration, and so this spurious trade continued to increase and prosper, its development being, unfortunately, facilitated by the fall of exchange and heavy import duties, which have put the prices of many of the genuine foreign commodities beyond the purchasing power of any but the affluent classes. The Government has at last, however, decided to place a control upon this kind of trade, and on November 3rd last a decree was promulgated prohibiting, under heavy penalties, the importation or fabrication of labels intended to imitate those of foreign manufacturers, and requiring that all productions made in Brazil should be labelled in a way to show their origin. Prior to this, a legislative measure had been adopted with the ostensible object of securing the purity and genuineness of alimentary productions, wines, spirits, beers, &c., imported into the Republic.

"For this purpose, one of the clauses of the Budget Law for 1897 provides that wines, pigs' lard, and all other alimentary productions condemned by the National Laboratory be destroyed, and the importers fined in the sum of 500 milreis. Under this provision, all wines and alimentary productions are considered injurious to the public health, and therefore condemned, which contain any of the following elements:—

"Boric or salicylic acid, alcohol of bad quality, free mineral acids, such as sulphuric, sulphurous, nitric and hydrochloric, sulphites, alum, alkaline fluorides and fluosilicates, saccharine, salts of strontium, lead, zinc, tin, arsenic, antimony, sulphate of potash in the proportion of more than 2 grms. per litre of wine; in beer, the substitutes of hops, as absinth, quassia amara, colchicum, picrotoxin, colocynth, nux vomica, picric acid, aloes, and also essences prepared with 'ethers of oily classes,' colours made from coal-tar or with a base of lead, mercury, copper, arsenic, antimony, barium, or any other substance which science has recognised, or may recognise, to be injurious to health.

"In consequence of this enactment, the Public Analyst has condemned, as containing ingredients injurious to health, supplies received from firms, of various nationalities, whose productions had hitherto been admitted without question to this market, as well as all others in the world.

"On examining the official analyses of the condemned goods, it is found that only in one case were the proportions of the different elements discovered clearly stated; all the other analyses are extremely vague, merely asserting the presence of certain injurious ingredients 'in a notable proportion.'

"Some of the importers whose shipments have been condemned, have sought the advice of the Consulate as to adopting proceedings to vindicate the genuineness of the articles received, and protect the good name of the British producer.

"The only advice the circumstances appeared to admit was that British exporters should be made fully aware of the provisions of the Brazilian law, and that future shipments should be accompanied by the analysis of a recognised independent authority, this analysis to be attached officially to the bill of lading by the Brazilian Consul at the port of shipment with his certificate that the analysis refers to the goods mentioned in the bill of lading. If on arrival in Brazil such goods should be condemned, a judicial protest could be entered before the proper local authority—the Brazilian Sectional Judge—and a petition presented for a second analysis by independent and properly qualified parties.

Among the products condemned by the official analyst is lime-juice; consequently the Customs inspector has not allowed recent arrivals to be cleared for consumption. Although, therefore, there are considerable quantities of the article in the Custom-house, there is absolutely none to be bought on the market.

### Manganese Ore in Brazil.

*Foreign Office Annual Series, No.* 2058, *April* 1898.

Manganese ore has formed an article of export during the past four years. The quantities shipped by Messrs. Airosa and Co., and Mr. C. Wigg, who exploit the mines, have been as follows:—

| Year. | Quantity. | To |
|---|---|---|
| | Tons. | |
| 1894 | 1,390 | Middlesbrough. |
| 1895 | 5,490 | ,, |
| 1896 | 14,120 | ,, |
| 1897 | 8,000 | Philadelphia. |

This mineral is brought from the district of Miguel Burnier, in the State of Minas Geraes, which is connected by the Central Railway with Rio de Janeiro. The distance is about 310 miles, and the transport occupies from 8 to 10 days. The mines are situated at an altitude of 4,000 ft., in a pleasant and healthy climate, and the labour employed is chiefly Italian and native, with some Spanish and Portuguese.

Owing to insufficiency of rolling stock and other difficulties, the railway can at present transport only 1,800 tons per month. The rate of freight is high, and the conditions generally are not favourable to the development of this industry. It is expected that about 20,000 tons of ore will be shipped this year.

### Cocaine at Callao.

*Foreign Office Annual Series, No.* 2056, *April* 1898.

The manufacture of cocaine at this port is falling off considerably, in consequence of the recent discovery in Berlin of a substitute called "eucoin," which is largely used in Europe.

## GENERAL TRADE NOTES.

### Petroleum Briquettes.

*Board of Trade J., April* 1898, 471.

In the process for making petroleum briquettes, described in *Kuhlow's*, petroleum refuse is worked up in such a manner as to assume the form of a solid substance, easily handled and constituting a cheap and convenient fuel. About 10 per cent. of soda lye, with about 10 per cent. of any fatty matter—tallow, for instance—is heated in a boiler, either by superheated steam or with exclusion of air; and so much petroleum refuse is then added to the heated mass as to make up 100 parts. The whole is heated, with constant stirring, for about an hour, the time varying with the mode and intensity of heating; but the temperature must never be allowed to reach the boiling point of petroleum, because in that case a large quantity of froth would form. In this incipient state of saponification the mass acquires the property of taking up large quantities of very fluid rock oil, and if this incorporation of the mass should proceed too slowly or remain incomplete, it may be hastened by the addition of a little soda lye. In a short time the saponification of the grease with the fixing of the petroleum will be effected, and the mass thus produced may be run into moulds and allowed to cool, when it may be cut into pieces of any desired form. According to the use for which the briquettes are destined, they can during manufacture receive the addition of coaldust, sawdust or other refuse, and, if it be desired to obtain a product of less firm consistency, the grease may be entirely or partially replaced by resin or resin acid, the product in either case containing more than 80 per cent. of petroleum, more than 90 per cent. of combustible substances, and less than 5 per cent. of incombustible residue.

### Zinc in the Caucasus.

*Eng. and Mining J., April* 2, 1898, 405.

A deposit of zinc ore has recently been discovered in the government of Koutaisk, in the Caucasus, which is now being explored. The ore is a blende, and an analysis made in the laboratory of the Technical Society of St. Petersburg showed: ZnO, 57·82; S, 31·90; insoluble residue, 10·28 per cent.

### Manganese in the Caucasus.

*Eng. and Mining J., April* 2, 1898, 399.

A deposit of manganese ore recently discovered and opened near Tchiatouri, in the government of Koutaisk, in the Caucasus, is said to be very rich, analyses made at St. Petersburg showing from 79·8 to 86·2 per cent. of manganese oxide ($MnO_2$).

### Platinum in New Zealand.

*Eng. and Mining J., April* 2, 1898, 399.

There are reports of the finding of platinum ore in the Nelson district, in New Zealand, where a large outcrop has been located in a volcanic hill near Takaka. The surrounding country is limestone. The statements as to the value of the ore are very conflicting.

### The Manufacture of Caffeine.

*Chem. and Druggist, March* 26, 1898, 494.

The Paris Syndical Chamber of Chemical Products has approached the French Minister of Commerce with a view to obtaining the right for chemical manufacturers to import tea and tea-dust into this country free of duty for the purpose of manufacturing caffeine; or, rather, the object is that an arrangement should be made by which the duty should be charged on the caffeine made from the tea, in order to enable French makers to compete with foreign manufacturers. M. Adrian, the President of the Chamber, had hoped that tea-refuse (déchets de thé) might be used. The quotations obtained from Calcutta for "déchets" is 35 frs. per 100 kilos., and for tea-dust 44 frs. to 69 frs. per 100 kilos., say about 2½*d.* per lb., delivered at Marseilles.

### Oil of Lemon-Grass.

*Chem. and Druggist, March* 26, 1898, 517.

Adulteration of this oil continues. Referring to the article by Mr. J. C. Umney in *The Chemist and Druggist* of December 18, 1897, a German firm of distillers confirm the observations therein recorded, and state that three kinds of oil may be met with in the market—(*a*) natural lemon-grass oil, sp. gr. 0·9041, optical rotation −3·18°, and solubility in 70 per cent. alcohol 1 in 3; (*b*) oil deprived of citral, sp. gr. 0·9102, which is insoluble in 70 per cent. alcohol, and being heavier than that solvent, sinks in it; (*c*) lemon-grass oil adulterated with oil of lemon, sp. gr. 0·8886, optical rotation +18·20°, insoluble in and swims upon 70 per cent. alcohol. We also learn that *Andropogon citratus D.C.*, the plant from which lemon-grass oil is distilled, is now grown at St. Thomas, in Portuguese West Africa, and the oil distilled from it agrees in physical characteristics with East Indian oil, but it is optically inactive. In our report of March 5 we mentioned the importation by Mr. Schlesinger of Tonkin verbena oil. This has now been chemically examined, and found to contain 70 per cent. of citral.

### Tincture Exports.

*Chem. and Druggist, March* 26, 1898, 516.

During the quarter ending on December 31, 1897, the Excise Department paid drawback on 19,021 gals. of proof spirits exported in tinctures and medicinal spirits, as compared with 17,673 gals. in the corresponding quarter of the preceding year—an increase of 1,348 gals. For the three quarters of the current financial year the quantity of spirit on which drawback has been paid amounts to 51,015 proof gals., an increase of no less than 7,723 gals. over the same period in the preceding year. There can be no doubt that the figures for the year ending on March 31 will be the largest of any of the ten years since the concession made by the Treasury.

### The Black-Etard Permanganate Process.

*Eng. and Mining J., March* 19, 1898, 315.

A process which has been recently tried in New Zealand, and which is attracting attention there, is a modification of the chlorination process as applied to gold ores. It is known as the permanganate gold recovery process. It was originally the discovery of M. Etard, a French metallurgist, and, briefly described, it consists in roasting the ore and subjecting it to the action of a dilute solution of permanganate of potash in the presence of hydrochloric acid. The process as applied in New Zealand consists in a very simple modification, namely, the use of sulphuric acid and salt instead of hydrochloric acid, on account of the cheapness of the former compared with the latter. This modification, which is the discovery of Dr. Black, of the Otago University, is patented throughout the Australasian colonies, and is known as the Black-Etard process. Some doubt is expressed as to the validity of the patent on the modification, which, as chemists know, is simply the manufacture of hydrochloric acid in the permanganate solution instead of using already manufactured hydrochloric acid; but the patent has been granted. A company has been formed, with a capital stock of 150,000 dols., to work the process, and an experimental plant has been put up at Dunedin, New Zealand. The advantages claimed by this process are its cheapness, simplicity, the absence of obnoxious fumes, and the high extraction obtained.

The *Australian Mining Standard* says that in this plant a lot of ore from the Mount Morgan mine, in Queensland, which contains so much copper as to render the application of cyanide impossible, was recently treated, and an extraction of 92 per cent. gold contents was obtained after 15 hours' percolation, and in 5 hours by agitation. Another trial on concentrates from Barewood, Otago, which contain antimony and manganese, as well as iron and arsenical pyrites, gave an extraction of 95 per cent. The precipitation is effected by ferrous sulphate. The process has been very successful on the refractory ores of the Thames district in New Zealand, and a plant is to be put up at the Monowai mine in that district.

### A Manganate-Cobalt-Nickel Deposit in Germany.

*Eng. and Mining J., March* 19, 1898, 343.

We learn from a correspondent in Cologne, Germany, that about 12 months ago a company, called the Westerwald Cobalt Mining Company, was formed by Cologne and Brussels capitalists with a paid-up capital of 3,000,000 Marks to buy and work a supposed cobalt mine in the Westerwald. At the time that mining venture was looked upon with grave suspicion by mining authorities and experts. But now, after the mine has been systematically explored and further opened up, even disbelievers in the mine agree that a rare and good deposit has been struck. The property extends over 11 square kilometres, or about 80 miles, and lies close to the Cologne-Giessen railroad. Exploration work has so far brought to light a number of veins of various extent and thickness, but in particular one main or mother lode varying from 9 to 18 metres (or 28 to 56 ft.) in thickness, extending for several miles and already proved to a depth of nearly 200 ft. Various samples of the ore from this mother lode assayed on an average 25 to 30 per cent. manganese, somewhat over 1 per cent. protoxide of cobalt, and about 1 per cent. of nickel. The ore looks like black cobalt earth. The origin of these ore deposits, like that of the entire Westerwald district, is volcanic. The exploration work so far done leaves no doubt that this mother lode rather increases than decreases in extent and richness as greater depth is reached. The importance of this new mine for Rhenish-Westphalian metal industries can hardly be calculated. Nothing like it has been struck in Germany for many years.

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 31st March. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 2,060,013 | 2,053,908 |
| Chemicals and dyestuffs | 1,012,437 | 751,784 |
| Oils | 476,778 | 681,652 |
| Raw materials for non-textile industries. | 3,381,380 | 3,635,334 |
| Total value of all imports | 40,655,114 | 43,412,829 |

### Summary of Exports.

| Articles. | Month ending 31st March. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 3,169,955 | 2,921,031 |
| Chemicals and medicines | 930,096 | 855,535 |
| Miscellaneous articles | 3,073,757 | 3,073,276 |
| Total value of all exports | 21,647,269 | 20,833,865 |

### Imports of Metals for Month ending 31st March.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| Copper :— | | | | £ | £ |
| Ore | Tons | 5,611 | 3,968 | 61,601 | 45,094 |
| Regulus | „ | 12,184 | 8,821 | 296,590 | 243,052 |
| Unwrought | „ | 4,830 | 5,926 | 240,580 | 302,813 |
| Iron :— | | | | | |
| Ore | „ | 563,010 | 521,086 | 417,113 | 401,497 |
| Pig | „ | 5,935 | 7,410 | 22,575 | 20,979 |
| Bolt, bar, &c. | „ | 2,670 | 2,500 | 21,411 | 18,909 |
| Steel, unwrought | „ | 2,410 | 3,762 | 14,380 | 21,220 |
| Lead, pig and sheet | „ | 13,800 | 18,591 | 160,519 | 232,373 |
| Pyrites | „ | 63,740 | 51,597 | 108,241 | 87,089 |
| Quicksilver | Lb. | 394,460 | 1,165,310 | 37,850 | 108,834 |
| Silver ore | Value £ | .. | .. | 129,358 | 110,802 |
| Tin | Cwt. | 90,767 | 54,495 | 271,951 | 171,283 |
| Zinc | Tons | 6,177 | 6,557 | 107,456 | 114,526 |
| Other articles | Value £ | .. | .. | 170,378 | 175,437 |
| Total value of metals | | .. | .. | 2,060,013 | 2,053,908 |

### Imports of Oils for Month ending 31st March.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Cocoa-nut | Cwt. | 7,115 | 43,100 | 7,652 | 47,859 |
| Olive | Tuns | 1,105 | 2,437 | 40,084 | 84,318 |
| Palm | Cwt. | 69,597 | 77,348 | 72,209 | 83,280 |
| Petroleum | Gall. | 11,054,362 | 18,785,823 | 210,282 | 285,108 |
| Seed | Tons | 2,669 | 3,157 | 58,625 | 63,739 |
| Train, &c. | Tuns | 890 | 992 | 7,564 | 18,538 |
| Turpentine | Cwt. | 8,306 | 20,523 | 8,309 | 23,508 |
| Other articles | Value £ | .. | .. | 72,053 | 75,302 |
| Total value of oils | | .. | .. | 476,778 | 681,652 |

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST MARCH.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............. Cwt. | 17,514 | 11,323 | 7,504 | 7,242 |
| Bark (tanners', &c.) „ | 15,456 | 27,818 | 5,035 | 9,492 |
| Brimstone ........ „ | 47,320 | 37,330 | 10,673 | 8,843 |
| Borax ............ „ | .. | 7,112 | .. | 18,357 |
| Chemicals...... Value £ | .. | .. | 126,219 | 120,280 |
| Cochineal ......... Cwt. | 78 | 288 | 483 | 1,802 |
| Cutch and gambier Tons | 2,634 | 1,356 | 49,786 | 24,141 |
| Dyes:— | | | | |
| Alizarin ........Value£ | .. | .. | 23,430 | 21,358 |
| Anilin and other „ | .. | .. | 40,022 | 46,574 |
| Indigo ............ Cwt. | 32,616 | 19,972 | 593,852 | 350,926 |
| Nitrate of potash . „ | 45,470 | 20,574 | 34,025 | 16,573 |
| Valonia........... Tons | 1,625 | 2,311 | 16,507 | 24,039 |
| Other articles... Value £ | .. | .. | 104,901 | 117,094 |
| Total value of chemicals | .. | .. | 1,012,437 | 751,784 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST MARCH.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,950 | 6,382 | 3,813 | 16,934 |
| Bristles........... Lb. | 221,189 | 214,598 | 35,735 | 33,165 |
| Caoutchouc ...... Cwt. | 50,023 | 53,918 | 627,198 | 728,431 |
| Gum:— | | | | |
| Arabic......... „ | 8,587 | 4,638 | 16,293 | 10,079 |
| Lac, &c. ....... „ | 20,468 | 14,707 | 100,225 | 52,518 |
| Gutta-percha .... „ | 2,734 | 2,642 | 25,480 | 35,503 |
| Hides, raw:— | | | | |
| Dry............ „ | 49,888 | 79,509 | 127,537 | 208,878 |
| Wet ........... „ | 51,751 | 56,969 | 103,905 | 115,671 |
| Ivory............. „ | 567 | 763 | 24,882 | 33,735 |
| Manure:— | | | | |
| Guano......... Tons | 983 | 3,782 | 5,736 | 19,457 |
| Bones ......... „ | 6,789 | 7,800 | 29,008 | 30,933 |
| Nitrate of soda... „ | 12,817 | 19,530 | 102,136 | 144,712 |
| Phosphate of lime „ | 23,929 | 26,068 | 38,027 | 42,907 |
| Paraffin........... Cwt. | 67,320 | 79,601 | 61,429 | 68,476 |
| Linen rags....... Tons | 2,069 | 1,925 | 21,135 | 18,541 |
| Esparto........... „ | 25,056 | 22,504 | 99,097 | 83,082 |
| Pulp of wood:— | | | | |
| Chemical ...... „ | 25,855 | 13,292 | 130,091 | 93,245 |
| Mechanical .... „ | | 15,890 | | 49,275 |
| Rosin............. Cwt. | 69,387 | 11,051 | 18,239 | 2,678 |
| Tallow and stearin „ | 182,533 | 137,808 | 175,747 | 132,444 |
| Tar ...............Barrels | 752 | 1,879 | 157 | 2,036 |
| Wood:— | | | | |
| Hewn ......... Loads | 136,051 | 176,964 | 262,682 | 308,265 |
| Sawn .......... „ | 114,231 | 128,793 | 294,903 | 333,490 |
| Staves......... „ | 7,816 | 7,887 | 43,818 | 46,620 |
| Mahogany ..... Tons | 6,692 | 10,209 | 60,963 | 88,768 |
| Other articles....Value £ | .. | .. | 973,144 | 935,491 |
| Total value ....... | .. | .. | 3,381,380 | 3,635,334 |

Besides the above, drugs to the value of 70,335*l*. were imported, as against 73,983*l*. in March 1897.

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST MARCH.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............. Cwt. | 474,790 | 345,873 | 117,538 | 91,238 |
| Bleaching materials „ | 87,479 | 89,889 | 28,532 | 26,033 |
| Sulphate of copper Tons | 11,861 | 11,733 | 200,252 | 190,321 |
| Chemical manures „ | 45,561 | 40,681 | 240,321 | 203,216 |
| Medicines....... Value £ | .. | .. | 106,137 | 93,540 |
| Other articles... „ | .. | .. | 237,316 | 251,187 |
| Total value .......... | .. | .. | 930,096 | 855,535 |

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST MARCH.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............. Cwt. | 11,823 | 10,932 | 49,743 | 43,480 |
| Copper:— | | | | |
| Unwrought.... „ | 26,603 | 38,325 | 70,204 | 99,497 |
| Wrought ...... „ | 27,599 | 21,505 | 86,730 | 67,547 |
| Mixed metal... „ | 17,640 | 11,647 | 44,261 | 28,760 |
| Hardware and cutlery. Value £ | .. | .. | 198,265 | 176,992 |
| Implements ..... „ | .. | .. | 119,450 | 118,167 |
| Iron and steel ... Tons | 351,144 | 285,000 | 2,290,506 | 2,046,199 |
| Lead ............ „ | 2,521 | 3,033 | 34,703 | 44,845 |
| Plated wares ... Value £ | .. | .. | 32,926 | 27,594 |
| Telegraph wires . „ | .. | .. | 139,642 | 135,583 |
| Tin ............. Cwt. | 5,536 | 7,780 | 17,859 | 25,999 |
| Zinc ............. „ | 10,247 | 20,620 | 8,290 | 16,462 |
| Other articles .. Value £ | .. | .. | 77,376 | 89,906 |
| Total value ....... | .. | .. | 3,169,955 | 2,921,031 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST MARCH.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 566,700 | 212,000 | 13,589 | 5,662 |
| Military stores.. Value £ | .. | .. | 91,913 | 128,128 |
| Candles.......... Lb. | 2,284,500 | 2,044,600 | 31,548 | 27,643 |
| Caoutchouc..... Value £ | .. | .. | 109,458 | 122,535 |
| Cement.......... Tons | 40,733 | 33,860 | 67,113 | 58,223 |
| Products of coal Value £ | .. | .. | 162,083 | 153,023 |
| Earthenware ... „ | .. | .. | 167,914 | 153,349 |
| Stoneware ...... „ | .. | .. | 17,017 | 16,468 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 82,964 | 161,369 | 5,749 | 8,132 |
| Flint........... Cwt. | 8,383 | 7,680 | 19,965 | 17,232 |
| Bottles......... „ | 72,530 | 69,988 | 33,472 | 32,785 |
| Other kinds.... „ | 18,006 | 19,390 | 14,039 | 16,517 |
| Leather:— | | | | |
| Unwrought .... „ | 13,593 | 14,665 | 128,927 | 135,793 |
| Wrought ..... Value £ | .. | .. | 32,690 | 35,182 |
| Seed oil.......... Tons | 6,192 | 5,195 | 104,162 | 85,247 |
| Floorcloth ..... Sq. Yds. | 2,623,500 | 2,514,700 | 104,869 | 102,026 |
| Painters' materials Val. £ | .. | .. | 150,261 | 145,890 |
| Paper ........... Cwt. | 93,639 | 105,473 | 140,417 | 146,488 |
| Rags............ Tons | 4,697 | 5,678 | 25,961 | 27,535 |
| Soap............. Cwt. | 65,947 | 72,233 | 67,682 | 71,089 |
| Total value .......... | .. | .. | 3,073,757 | 3,073,276 |

## Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

### I.—PLANT, APPARATUS, AND MACHINERY.

#### APPLICATIONS.

6160. J. A. Drake. Improvements in metallurgical and other like furnaces for calcining, reducing, carbonising, and other purposes. March 14.

6372. L. Sterne. Improvements in condensing and water cooling apparatus. March 15.

6431. E. Schliemann and E. von Boyen. Process of and apparatus for continuous extraction. Complete Specification. March 16.

6640. Willans and Robinson, Ltd., P. A. Low, and W. M. Huskisson. Improvements in furnaces. March 18.

6837. B. H. Thwaite and F. L. Gardner. A method and apparatus for separating dust from blast furnace gases. Complete Specification. March 21.

7024. F. Hugershoff. Improvements in centrifugal liquid precipitating or separating apparatus. Complete Specification. March 23.

7178. K. Abraham. Improvements in filters. Complete Specification. March 24.

7373. P. D. Day and The Acetylene Illuminating Company, Ltd. Improvements in apparatus for mixing two or more gases in any desired proportions. March 26.

7590. J. M. Thèvenet - Le - Boul. Improvements in metallic capsules for storing compressed or liquefied gases. Complete Specification. March 29.

8017. P. Pessé. Improvements relating to the purification and decoloration of liquids and the like and to apparatus therefor. Complete Specification. Filed April 4. Date applied for October 1, 1897, being date of application in France.

8069. A. T. Welch. Improvements in drying apparatus. Complete Specification. April 5.

8232. T. Healy and T. H. Kraft. Improvements in apparatus for drying sugar grain cotton-waste and other substances and materials. April 6.

8238. J. Mittwich. New or improved filtering apparatus. Complete Specification. April 6.

8239. G. R. Seton. Improvements in apparatus for cooling liquids. April 6.

8332. G. Hookham, E. Jones, and Kynoch, Ltd. Improvements in and relating to evaporative surface condensers. April 7.

### Complete Specifications Accepted.

1897.

7661. H. Higgins. Drying and sterilising. March 23.

7667. H. Higgins. Sterilising apparatus. March 23.

7668. H. Higgins. Sterilising apparatus. March 23.

15,231. A. Huillard. Mode of and apparatus for desiccating various substances. March 30.

15,740. J. R. Green. Non-conducting coverings for boilers, pipes, and for other purposes. March 30.

15,917. J. R. Green. Non-conducting coverings for boilers, pipes, and for other purposes. March 30.

1898.

3108. I. P. B. Knudsen. Centrifugal separating apparatus. March 30.

3690. H. M'Phail. Method of and means for drying wood and other vegetable substances. April 20.

## II.—FUEL, GAS, AND LIGHT.

### Applications.

6240. G. de Roussy de Sales and A. Charbonnee. Improvements in apparatus for burning liquid fuel, and the application thereof to heating furnaces or for other heating purposes. March 14.

6748. W. B. Rickman. An improvement in enriching combustible gas and apparatus therefor. March 19.

6869. L. Stoecklin. Process for the manufacture of self-igniting mantles for glow-light. Complete Specification. March 21.

6981. M. Ziegler. Improvements in or relating to the production of charcoal or coke from peat lignite and the like, and the recovery of bye-products. Complete Specification. March 22.

7065. H. S. Ryland and A. N. Townley. An improved acetylene gas generator. March 23.

7070. E. Edwards.—From L. Steiner, Roumania. Improvements in acetylene gas apparatus. Complete Specification. March 23.

7145. J. A. Sinclair and E. R. Powell. Improvements in apparatus for generating and cooling acetylene gas. March 24.

7184. E. Gurovits. Improved treatment of acetylene gas for preventing the deposit of solid carbon therefrom when burning. March 24.

7191. The Mineral Oil Corporation, Ltd., and P. Dvorkovitz. Improvements in the condensation of the vapours of mineral oils and in apparatus for the purpose. March 24.

7206. H. W. Regnander. Improvements in the manufacture of firelighters or artificial fuel from wood, sawdust, turf, peat, or similar materials, applicable for stoves, hearths, fireplaces, or the like. March 24.

7396. W. P. Gibbons and G. B. A. Gibbons. Improvements in the construction of gas retorts and in appliances for making the same. March 26.

7412. P. Parsons. Improvements in apparatus for the complete combustion of fuel and the prevention of smoke. March 28.

7427. J. Liddle.—From L. O. Triebel and D. Whiteis, United States. Improvements in apparatuses for making gas. Complete Specification. March 28.

7499. W. Watson and J. B. Gray. Improved method of and apparatus for producing gas for lighting and other purposes. March 29.

7521. V. Daix. Improvements in apparatus for the production of acetylene gas. March 29.

7592. J. Coyne. Improvements in the manufacture of gas. Complete Specification. March 29.

7671. N. A. Alexanderson. Improvements in the manufacture of candles. Complete Specification. March 30.

7675. M. C. A. Fourchotte. Improvements in or relating to acetylene gas generators. March 30.

7685. J. Lépine. An improved apparatus for the production of acetylene gas. March 30.

7770. E. de Strachl, A. Réchard, and A. Devarenne. New or improved apparatus for generating acetylene gas. March 31.

7809. W. Osten. Improvements in or connected with generators for acetylene gas. April 1.

7838. S. de Szepczynski. An improved automatic acetylene gas generator. April 1.

7951. P. Naef. Process and apparatus for making coke and saving by-products. Filed April 2. Date applied for Sept. 7, 1897, being date of application in United States.

7952. P. Naef. Process and apparatus for making coke and saving by-products. Filed April 2. Date applied for Sept. 7, 1897, being date of application in United States.

7953. P. Naef. Process and apparatus for making coke and saving by-products. Filed April 2. Date applied for Sept. 7, 1897, being date of application in United States.

8030. A. E. R. Söhnel and A. R. Zehner. Improvements in acetylene gas apparatus. Complete Specification. April 4.

8147. C. Kelly. Improvements in and relating to generators for acetylene gas. Filed April 5. Date applied for Feb. 15, 1898, being date of application in the United States.

8174. H. E. Temple. An improved acetylene gas generator. April 6.

8251. Sir H. Grubb, knight. Improvements in apparatus for the production of acetylene and other gas. April 6.

8382. H. H. Lake.—From J. Grand, France. Improvements in apparatus for the production of acetylene gas. April 7.

8399. J. Getzl and S. Landau. Improvements in apparatus for producing acetylene gas. April 9.

8439. J. Stott. Improvements in apparatus for generating and storing acetylene gas. April 9.

8462. J. C. Berntrop and M. L. Q. van L. Hulsebosch. An improved process of solidifying crude and refined petroleum and petroleum residues. April 9.

8557. E. Tooth. An improved illuminating and heating gas. Complete Specification. April 12.

8565. P. Lacroix. An improved automatic apparatus for producing acetylene gas. April 12.

8568. D. Bénézet. A new or improved apparatus for the continuous automatic generation of acetylene gas. April 12.

8675. A. Graetz. Improvements in and connected with acetylene generators. April 13.

8683. J. Aykroyd and B. Aykroyd. Improvements in or appertaining to acetylene generators. April 14.

8694. L. M. Miller. Improvements in generators for acetylene gas. April 14.

8701. J. M. Tavernor and E. L. Levetus. Improvements in or relating to the purification and deodorization of acetylene and other gases. April 14.

8702. J. M. Tavernor and E. L. Levetus. Improvements in or relating to lamps for generating and burning acetylene gas. April 14.

8723. J. W. Turner and C. Jousset. Block fuel. Complete Specification. April 14.

8780. C. H. Uttley. Improvements appertaining to apparatus for use in the production of acetylene. April 15.

8811. J. Wood. Improvements in apparatus for generating and burning acetylene gas for illuminating purposes. April 15.

8813. W. L. Wise.—From A. Piatti and Co., Italy. Improvements in the manufacture of gas. April 15.

8825. A. F. Bowers. Improvements in and relating to apparatus for generating acetylene gas. Complete Specification. April 15.

8873. W. Young, W. R. Herring, and A. Bell. Improvements in the carbonising or destructive distillation of coal shale, carbonaceous tars, and similar substances for the production of illuminating and heating gases and water-gas, and in apparatus therefor. April 16.

8875. J. Holland. A new or improved mantle for incandescent lighting. April 16.

### Complete Specifications Accepted.

1897.

294. O. Kern. Incandescent gas burners. April 13.

4125. J. R. Wigham. Apparatus for the production of artificial light, applicable for lighthouse and ordinary illumination. March 23.

7435. C. Billington, jun. Method of and means for removing "scurf" or retort carbon from gas retorts. March 30.

8513. J. A. Bellon-Lencauchez. Production and purification of gas, and apparatus therefor. March 23.

9387. J. A. Sinclair. Means for lighting by electricity and incandescence. March 23.

10,900. H. Hansen. Manufacture of fuel blocks from petroleum. March 30.

11,010. M. Otto. Manufacture or production of ozone, and in apparatus therefor. March 30.

12,110. G. Trouvé. Apparatus for the production and storage of acetylene gas. April 6.

12,275. W. B. Hartridge. Manufacture of artificial fuel blocks and apparatus for that purpose. April 6.

13,081. F. Dresser. Apparatus for generating acetylene gas. April 6.

13,667. D. Whalley, J. Hacking, and The Ideal Gas Co., Ltd. Acetylene gas plant. April 13.

13,899. R. Laughans. Manufacture of incandescing media for lighting purposes. April 20.

14,208. F. H. Haviland and W. H. Murch. Apparatus for automatically generating, storing, and supplying acetylene gas. April 20.

19,951. L. Chambault. Method and apparatus for generating and burning acetylene gas chiefly designed for use on velocipedes, autocars, boats, and the like. March 23.

20,011. E. J. Dolan. Acetylene gas generators. April 6.

23,237. O. Imray.—From E. Pilous and A. M. Michaelis, Austria. Process for enriching gas of low illuminating power. April 6.

28,226. H. L. Hartenstein. *See* Class VII.

28,909. G. A. Bronder. Machinery for discharging gas retorts. March 23.

29,797. M. Bernstein. Process for manufacturing incandescent bodies. March 23.

29,960. M. Wagner. Apparatus for the manufacture of acetylene gas. March 30.

1898.

1310. G. F. Dinsmore. Process of transmitting heat for industrial purposes. March 23.

2309. G. Jacobi. Acetylene gas apparatus. March 23.

2602. H. O. Marcks. Apparatus for generating acetylene. March 30.

2615. G. Himmel. Bunsen burners for incandescent lighting. March 23.

4113. H. Berger. Apparatus for generating acetylene. April 13.

4194. H. Wolpert. Air-carburetting apparatus. April 13.

5594. C. H. P. Schlüter and C. L. F. Lüdemann. Apparatus for generating acetylene. April 20.

5926. O. Lindemann.—From E. Körting, Germany. Apparatus for spraying or atomising combustible liquids and mixing the same with air. April 20.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Application.

8665. W. Irwin. An improved still for the extraction of benzol tolnol and xylol from their admixture with creosote or other heavy oils. April 13.

## IV.—COLOURING MATTERS and DYES.

### Applications.

6211. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. New process for the production of indoxyl compounds and of intermediate products therefor. March 14.

6533. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of mordant dyestuffs from anthraflavine and isoanthraflavine acids. March 17.

6760. A. G. Green, A. R. Wahl, and The Clayton Aniline Co., Ltd. The production and manufacture of azoxy and azoaldehydes and of colouring matters therefrom. March 19.

6765. H. R. Vidal. Improvements in the production of amidophenols and naphthols. March 19.

6766. H. R. Vidal. Improvements in the production of amidophenols and naphthols. March 19.

7139. L. J. E. Hubou. Application of acetylene gas for the production of a new black called "Acetylene black." March 24.

7474. H. Terrisse and G. Darier. Improvements in the preparation of dyestuffs derived from the Acridine group. March 28.

7659. O. Imray.—From The Society of Chemical Industry, Switzerland. Manufacture of new trisazo colouring matters capable of dyeing unmordanted cotton and of yielding blue shades by development on the fibre. Complete Specification. March 30.

8635. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new anthraquinone dyestuffs. April 13.

### Complete Specifications Accepted.

1897.

8991. A. M. Clark.—From Kullmann and Rapp. Process for the production of new tetrazo azo compounds. March 23.

10,605. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Manufacture or production of colouring matters. April 6.

11,997. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis. Substantive colouring matters. April 20.

12,179. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of ethers of *m*-oxy-*p* amidobenzoic acid. April 6.

13,104. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Bruning. Process for the manufacture of brown to black dyestuffs. April 20.

13,709. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. Production of aromatic aldehydes. April 20.

14,376. H. Imray.—From F. Hoffmann and La Roche and Co., Switzerland. Manufacture of sulphonic acids of guaiacol and of creosote, and of metallic salts of these sulphonic acids. April 6.

1898.

75. J. Y. Johnson.—From F. E. Bucher, Jamaica. Extract of logwood and method of obtaining the same. March 23.

2480. G. W. Johnson.—From Chemische Fabrik Griesheim, Germany. Manufacture or production simultaneously of pure ortho-nitro-chloro-benzene and pure para-nitro-chloro-benzene. March 30.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

6181. H. M. Girdwood. A new manufacture of yarn March 14.

6515. G. Kreuter. Process for producing fabrics from silk rags. Complete Specification. March 17.

6828. A. Glatz and The Vereinigte Filzfabriken. Improvements relating to the treatment of vegetable fibre and to apparatus therefor. March 21.

6845. W. Howarth, J. E. Howarth, and T. N. Grant. Improvements in treating cotton and other fabrics and fibrous materials to render them non-flammable, and compound therefor. March 21.

7317. A. Ashworth. Improvements in the method of and means for imparting a silky lustre to fabrics having a vegetable origin. March 26.

7357. J. Smith and Sons, Ltd., and W. Leach. Improvements in the treatment of wool or other animal fibres to remove grease or other soluble material therefrom. March 26.

7687. H. Gassner. Process for giving an increased finish to "mercerized" yarns, twines, and the like, from vegetable fibres. Complete Specification. March 30.

7688. T. E. Schiefner. Improvements in and relating to the process for "mercerising" yarn, twine, or like thread from vegetable fibres. Complete Specification. March 30.

8872. D. Macfarlane and T. S. Macfarlane. Improvements in "mercerising" cotton warps or like yarns, and apparatus therefor. April 16.

### Complete Specifications Accepted.

1897.

9517. R. Thomas and E. Prevost. Process of mercerising vegetable fibres or textile fabrics. April 20.

10781. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Method of mercerising cotton yarns. March 30.

11917. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Process for chlorination of wool. April 13.

30916. W. P. Thompson.—From J. Rudolf, Germany. Process for rendering textile materials, paper, wood substances, and the like, waterproof, and for finishing and protecting them against spotting and moths. April 6.

1898.

3581. O. Imray.—From United States Flax Fibre Company of United States. Process of and apparatus for cleaning flat fibres. March 23.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

6546. A. J. Boult.—From — Elbers, Germany. Improvements in or relating to indigo printing and dyeing. March 17.

7302. P. Bertram. Improvements in the dyeing of hair and fur. March 25.

7715. E. Sykes and D. Redfearn. Improvements in the method of and apparatus for dyeing, scouring, and bleaching yarn in the warp. March 31.

8029. R. C. W. A. Sieverts. Improvements in processes for bleaching or dyeing Spanish cane (balamus rotang), bamboo, and other plants which have a siliceous coating. Complete Specification. April 4.

8078. A. Marr. Improvements in apparatus for dyeing, scouring, bleaching, and otherwise treating yarn in cop or other compact form. April 5.

8108. F. J. Oakes. An improved process of mordanting fibres or fabrics. Complete Specification. April 5.

8153. E. Holken. Improvements in dyeing with vidal black and colours of the same group. April 5.

8381. G. Young. Improvements in apparatus for dyeing, mercerising, or otherwise treating with liquid yarn, thread, or cloth in warp or on beam. April 7.

8589. H. W. Kearns and J. Barnes. An improved method of fixing tannate of titanium on vegetable fibre. April 13.

### Complete Specifications Accepted.

1897.

9392. C. E. Gassmann. Process of fixation of colours basic and phenolic in printing. March 30.

9804. P. Desne. Process for imitating on vegetable fibres fabrics woven in colours. March 30.

10,933. G. Wendler. Product adapted for use in dyeing wool, in bread or pastry, and for other purposes. April 13.

26,667. P. A. Newton. From The Alexander Smith and Sons' Carpet Company, United States. Methods of and apparatus for printing or colouring yarn and similar material. April 13.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

6987. H. E. Sturcke and W. F. Jobbins. Improved processes of and apparatus for obtaining amorphous carbonate of lime from caustic soda or caustic potash residues, for obtaining sulphate of lime from such residues, and screening or filtering apparatus for use in connection with such processes, or for analogous purposes. Complete Specification. March 22.

7002 L. L. Billaudot. *See* Class X.

7086. R. H. Davidson and The United Alkali Company, Ltd. Improvements in the manufacture of flowers of sulphur. March 23.

7269. W. Baranoff, E. Hildt, and C. F. Claus. Improved processes for producing the sulphides of the alkalies and of the alkaline earths from the sulphates thereof, and for obtaining from such sulphides free sulphur, sulphurous acid, sulphide of hydrogen, and carbonates of the alkalis. March 25.

7341. J. W. Hill. Improvements in the manufacture of oxalic acid. March 26.

7509. G. T. Beilby. Concentrating solutions of alkaline cyanides and ferrocyanides. March 29.

7574. W. P. Thompson.—From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuchel, United States. Improved method and apparatus for producing calcium carbide. Complete Specification. March 29.

7726. J. Grossmann. Improved method of converting acid sulphates of soda or potash into caustic alkali. March 31.

7791. E. S. Bond. Improvements in means for preventing or retarding the decomposition of carbide of calcium. April 1.

8010. I. L. Roberts. Improvements in processes of preserving carbides. Filed April 4, 1897. Date applied for Nov. 24, being date of application in United States. Complete Specification.

8026. C. K. Mills.—From P. H. L. Pellet and C. Reynaud, France. A new process of manufacturing oxide of copper and salts of copper and other salts. April 4.

8471. J. Grossmann. Improved method of converting acid sulphates of soda or potash into caustic alkali. April 9.

8554. E. Tooth. Improvements in the manufacture of carbide of calcium. Complete Specification. April 12.

8557. F. Forti.—From E. Fontani, Italy. Manufacture of aluminium oxide or hydrate. April 12.

8570. W. P. Thompson.—From H. Harland and S. D. Crenshaw, United States. Improvements in roasting ovens or furnaces for the desulphurisation of volcanic sulphur, sulphurous ores, and ores containing a greater or less percentage of sulphur. Complete Specification. April 12.

8756. H. Sez. A new or improved process for removing the fœtid odour of carbide of calcium and for rendering the same air proof. April 15.

### Complete Specifications Accepted.

1897.

7715. J. McGougan and A. McGougan. Process for the production of nitrites of the alkali or alkaline earth metals. March 23.

8671. B. C. Hinman. Manufacture of a salt or compound containing bromide. March 30.

10,183. G. B. Ellis.—From La Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier. Manufacture of aldehydo-benzoic acid and intermediate products. March 23.

11,240. W. Shedlock. Apparatus for obtaining salt from brine and similar purposes. April 6.

11,353. J. R. Wylde, J. W. Kynaston, and the United Alkali Company, Ltd. Manufacture of hydrochloric acid free from arsenic. March 23.

12,534. A. R. Davis. Manufacture of sodium chlorate. April 13.

24,077. A. J. Boult.—From R. Memmo, Italy. Manufacture of calcium and other carbides, and by-products obtained thereby. April 6.

28,226. H. L. Hartenstein. Process and apparatus for carburetting calcium. March 30.

1898.

3801. J. Y. Johnson.—From H. Dercum, United States. Manufacture or regeneration of chromates or bichromates from waste battery or other liquors, or from chrome alum or sesqui salts of chromium. April 6.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### Applications.

6203. F. Vitte, sen. Improvements in processes of and apparatus for manufacturing plate or sheet glass. Complete Specification. March 14.

6632. H. Leek. Improved means of supporting pottery ware in kilns or ovens while being baked or fired. Complete Specification. March 18.

6944. F. H. Becker and L. Schreyer. A method of and furnace for the continuous melting of glass by electricity. Complete Specification. March 22.

7395. W. P. Gibbons and G. B. A. Gibbons. Improvements in the construction of tiles for regenerative furnaces. March 26.

8113. J. C. Mewburn. — From W. Hirsch, Germany. Improvements in the manufacture of glass. April 5.

8426. J. P. Holdcroft, W. Jackson, and A. Tomkinson. Improved heat indicating or trial pieces for use in pottery and other kilns. April 9.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

6458 H. M. Clifford and W. F. L. Frith. Improved compound for paving or covering roads, paths, or floors, and for manufacture of paving bricks or blocks. March 16.

7105. G. F. Lebioda. Improvements in or relating to apparatus for impregnating and dyeing wood. Complete Specification. March 23.

7346. A. H. Crawford. An improved fire-proof floor for buildings and other structures. March 26.

7366. W. Joy. Improvements in the manufacture of Portland cement and in apparatus to be used therein. March 26.

7418. H. Miller. An improved method of treating levigated slate. March 28.

7634. A. Brooke, N. Brooke, and W. Brooke. Improvements in artificial stone. March 30.

7677. H. H. Lake.—From F. L. Konrad, Germany. Improvements in and relating to the preservation and fireproofing of wood and the like. March 30.

7945. R. Wolfers and A. Hanuemann. The manufacture of an improved artificial asphalte. April 2.

8111. H. V. Simpson. Improvements in or relating to the impregnation of wood for the purpose of rendering it non-inflammable or for preserving or colouring it. April 5.

8226. L. P. Ford and T. W. Barber. Improvements in the method of manufacturing artificial stone and concrete. April 6.

8816. J. Feketeházy. Improvements in or relating to fireproof floors and ceilings for buildings. Complete Specification. April 15.

### Complete Specifications Accepted.

1897.

18,779. T. Holden and C. Major. Apparatus for the manufacture of cement. March 23.

24,179. C. E. Lee and C. F. Lawton. Manufacture of cement. April 13.

24,609. L. Brockmann. Production of artificial stone. April 6.

29,420. E. Vleck. Method of manufacturing plates, slabs, or the like, for building purposes. April 6.

30,214. G. Vögerl. Manufacture of artificial stone. March 30.

1898.

822. H. H. Lake.—From V. Micheli, Italy. Process for the manufacture of a substitute for wood or stone. April 6.

830. H. H. Lake.—From V. Micheli, Italy. Process for the manufacture of a compound for use in making pottery bricks, and the like. April 6.

1319. P. Kleber. Process for the production of building stones and like articles. March 30.

1378. P. M. Justice.—From The Lithosite Manufacturing Company, United States. Process of manufacturing cements. March 30.

3793. W. S. Wilkinson. Refining of asphalte. April 13.

3900. F. Grumbacher. Process for preserving, colouring, and hardening wood. April 6.

4591. F. J. Bergmann. Manufacture of cement and building blocks. April 6.

## X.—METALLURGY, MINING, Etc.

### Applications.

6460. S. Stepanoff. Improvements in apparatus for the extraction of zinc. March 16.

6593. G. L. Thomson.—From G. Thomson, United States. Improvements in alloys or metallic compounds. March 18.

6636. C. E. Acker. Improvements relating to the reduction of metals from their salts, and to apparatus therefor. Complete Specification. March 18.

6728. R. I. Roman. The manufacture of a new alloy. March 19.

6729. R. I. Roman. The manufacture of a new alloy. March 19.

6794. J. Dick. Improvements in and relating to the obtaining or recovering of gold or gold amalgams. March 21.

6798. C. Bedford. An improved apparatus for charging pig-iron, and scrap, or other material, into open hearth furnaces. March 21.

6861. H. Schmidt. Process for increasing the density of metal alloys in casting. Complete Specification. March 21.

7002. L. L. Billaudot. Improvements in the manufacture of phosphorus and in apparatus therefor. March 22.

7008. O. Imray.—From The Pennsylvania Salt Manufacturing Company, United States. Improvements in smelting plants. Complete Specification. March 22.

7081. C. B. Phillips. Improvements in the manufacture of armour plates and other objects of steel or iron with a hardened or tempered surface. March 23.

7287. A. Sattmann. Improvements in the manufacture of iron and steel, and in apparatus employed therein. March 25.

7350. J. B. de Alzugaray. Improvements in forming alloys and in reducing and refining metals and alloys. March 26.

7376. H. Wethered.—From J. A. Gilmour and H. S. Young, Nicaragua. Improvements in the treatment of ores containing precious metal for the recovery of the precious metal therefrom. Complete Specification. March 26.

7377. J. A. Hunter. Improvements in the method of converting iron into steel. March 26.

7389. J. B. de Alzugaray. Improvements in the manufacture of alloys and in the refining of metals and metallic alloys. March 26.

7126. J. B. de Alzugaray. Improvements in and connected with the manufacture of metallic alloys, and with the refining of metals and alloys. March 28.

7821. J. B. de Alzugaray. Improvements in and connected with the dephosphorisation, desulphuration, and refining of iron, steel, and other metals and alloys. April 1.

7904. A. M. G. Sebillot. Improvements in method and apparatus for dressing zinc ores and other volatile metals in blast furnaces. Complete Specification. April 2.

7930. F. Player. Rustless white metal. April 4.

8245. F. Schadeloock. Improvements in the manufacture of crucible steel and its alloys. Complete Specification. April 6.

8378. W. P. Thompson.—From G. Behr and T. Wallfisch, Germany. An improved process for hardening steel. Complete Specification. April 7.

8436. J. B. de Alzugaray. Improvements in the manufacture of basic material in connection with and for the refining of iron, steel, and other metals and alloys. April 12.

8528. B. J. B. Mills.—From T. A. Edison, United States. Process and apparatus for concentrating magnetic iron ores and bricking the same for market. Complete Specification. April 12.

8557. F. Forti.—From E. Fontani, Italy. *See* Class VII.

8585. J. J. Lish. Improvements in the treatment of and utilisation of blast furnace slag. April 13.

8641. S. O. Cowper-Coles. Improvements in and relating to the treatment of sulphide ores. April 13.

8767. C. A. Fuge. A new or improved method of coating metals with vitreous enamel. April 15.

### Complete Specifications Accepted.

1897.

4358. A. Phillips. A plunger for the purifying or mixing of molten metals or other bodies liquid at high temperatures. March 23.

8990. G. H. Blenkinsop. Smelting of mixed minerals, ores, and the like containing lead and zinc. March 30.

9103. G. W. Gesner. New and useful alloy of iron and hydrogen, termed Gesner metal, and means for producing the same. April 13.

9409. H. R. Angel. Treatment and reduction of sulphide ores containing zinc or otherwise. April 6.

10,459. A. J. Boult.—From A. Lismann. Method of coating copper or copper alloys with patina. April 20.

10,785. B. H. Thwaite and H. V. Holden. Process for decarburating steel or other compounds of iron and carbon and apparatus for that purpose. April 13.

11,695. J. C. Mewburn.—From La Société Anonyme de Commentry-Fourchumbault. Manufacture of alloys of or containing iron and nickel. April 6.

12,691. G. Weil, E. Quintaine, and C. Lepsch. Nickeling, silvering, gilding, &c. of aluminium. March 30.

22,961. G. Weil and A. Lévy. Processes for coating aluminium or other metals with silver or other metals. April 6.

1898.

285. G. de Bechi. Treatment of ores containing intimately mixed copper, zinc, and lead. April 13.

1432. O. S. Garretson. Smelting and converting of metal ores, and apparatus therefor. March 23.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

30,619a. J. W. Bullock. Improvements in gas batteries. Filed 12 April. Date claimed 28 Dec. 1897.

6179. O. Hamilton. Improvements in secondary batteries. March 14.

6525. W. G. Luxton and The United Alkali Company, Ltd. Improvements in apparatus for electrolytic purposes. March 17.

6526. W. G. Luxton and The United Alkali Company, Ltd. Improvements in apparatus for electrolytic purposes. March 17.

6607. E. J. Clark. A new or improved separator for the plates or electrodes of secondary batteries or accumulators. March 18.

6637. C. E. Acker. Improvements relating to the electrolytic production of metallic alloys and to apparatus therefor. Complete Specification. March 18.

6758. A. M. G. Sébillot. Improvements in electric furnaces. March 19.

7170. F. F. Yeatman and W. Donovan. Improvements in or in the construction of electrical storage batteries. March 24.

7193. A. J. Boult.—From J. Cerpaux and A. Wilbaux, Belgium. Improvements in or relating to primary electrical batteries. Complete Specification. March 24.

7305. J. Tabrar and W. Waller. Improvements in and relating to electrical accumulators. March 25.

7333. E. Mérian. Improvements in the construction of plates for electrical accumulators or storage batteries. March 26.

7423. A. Aschermann. Process for the extraction of pure metals or metallic alloys and carbides by electric heat. Complete Specification. March 28.

7455. F. Frentzel. Improved method of making the active mass in accumulators. March 28.

7470. W. L. Wise.—From Solvay and Co., Belgium. Improvements in and apparatus for effecting electrolysis. March 28.

7471. W. L. Wise.—From Solvay and Co., Belgium. Improvements in electrolysis. March 28.

7514. A. Warburton. Improvements in plates for battery and other purposes and method of making the same. March 29.

7575. W. P. Thompson.—From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuchel, United States. Improvements in electric furnaces for manufacturing calcium carbide, &c. Complete Specification. March 29.

7577. N. B. Stubblefield. Improvements in electrical batteries. Complete Specification. March 29.

7764. J. H. Hope. An improved apparatus for the electro deposition of metals. March 31.

7877. E. Bailey and G. R. Cox. Improved means in the production of white lead and oxide of lead by means of volatilising metals or ores by the use of the electric ore. April 2.

8036. A. J. Ward. Improvements in or relating to electric batteries or the like. April 4.

8037. D. G. Fitz-Gerald. Improvements in the manufacture of the peroxide elements of secondary batteries. April 4.

8278. H. Oppenheimer. Improvements in and connected with primary batteries. April 7.

8319. K. Krebs. Improvements in electrodes and in the method employed for producing them. April 7.

8477. B. A. Bouvier and G. L. Augieras. Improved electric accumulators. April 9.

8489. H. W. Wright. Improvements relating to the electro-deposition of metal. April 12.

8638. P. Determes. Improvement in electric furnaces for the manufacture of carbide and calcium or other electro chemical or electro metallurgical products of the like kind. April 13.

### Complete Specifications Accepted.

1897.

2378. W. Rowbotham. Primary batteries. April 6.

2831. A. R. Adams. Electric batteries. March 23.

7314. F. E. Everard. Accumulator plates or electrodes for secondary batteries. March 23.

8231. G. F. Redfern.—From C. Riordon. Storage batteries. March 23.

9276. W. Rowbotham. Primary batteries. April 13.

9456. J. H. McLean and C. D. Burnet. Secondary batteries. March 23.

9631. J. Dunn. New or improved plate for accumulators or electric storage batteries. March 23.

10,822. W. Evans and J. W. Smith. Improvements in apparatus for the electro-deposition of metals. April 6.

11,190. F. M. Lyte. Electrolysis of fused zinc chloride. March 30.

11,402. W. S. Romme. Process of and apparatus for electrically decomposing solid substances. April 13.

11,861. O. Lindner. Electric accumulators or storage batteries. April 13.

21,976. F. Greenfield. Appliances used in connection with the electro-deposition of metals. April 6.

30,686. C. J. Hubbell, H. G. Hubbell, W. De W. Boyer, and E. P. Mucklow. Primary batteries. March 30.

30,838. C. Marshner. Apparatus for use in the manufacture of accumulator-plates. March 30.

1898.

885. G. Langbein. Apparatus for electro-plating articles in bulk and in large quantities. March 30.

1697. M. Haas. Apparatus for the electrolytic treatment of bleaching liquids. April 6.

2024. C. Koenig. Primary batteries. March 23.

## XII.—FATS, OILS, AND SOAP.

### Applications.

6300. T. Coad. Improved apparatus for extracting moisture from granulated fat. March 15.

6334. J. C. W. Stanley and The Fish, Oil, and Guano Syndicate, Ltd. Improvements in or relating to the extraction of oil from material containing it. March 15.

6464. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in the oxidising of oils. March 16.

7242. J. Hertkorn. A process for the rapid oxidation of drying oils and their fatty acids by mixing and airing the same with pulverised substances for the preparation of linoleum and cork sheeting. March 25.

7428. M. E. B. Leeds. Improved washing compound. March 28.

7561. P. Pessé. *See* Class XVI.

7651. W. S. Thomson. An improved soap, and process of manufacture therefor. Complete Specification. March 30.

7808. R. A. L. Hill. Improvements in the manufacture of a material for use in the purification of certain oils, syrups, and other liquids, and for deodorising and decolorising purposes. Complete Specification. April 1.

7932. W. P. Thompson.—From E. Sieglin, Germany. Improvements in a manufacture of soap powder. Complete Specification. April 2.

8017. P. Pessé. *See* Class I.

8176. J. H. Hunter. Improvements in the purification of butter, fats, liquids, and such like matters, and apparatus therefor. April 6.

8244. C. Davies, H. Arrowsmith, and B. Magrini. Improvements in or relating to the manufacture of soap. April 6.

8784. N. R. Jamieson. Improvements in soaps. April 15.

### Complete Specifications Accepted.

1897.

4830. F. L. Bartelt. Washing and cleansing compound. Dec. 29.

9405. A. G. Haebre. Manufacture of plastic materials. April 13.

17,405. A. J. Boult.—From La Société "Meurice-Save," Belgium. Preparations for laundry purposes. April 6.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

6295. W. H. Wales and J. Cunningham. An improved enamel paint. March 15.

6321. J. S. Beeman. Improvements in quick-drying inks, paints, or the like. March 15.

6344. W. Ramsay. Improvements in the treatment of gutta-percha and like substances. Complete Specification. March 15.

6584. H. E. Taylor. Improvements in and relating to the manufacture of white lead. March 18.

6968. P. L. Clark. Improvements in reclaiming rubber. Complete Specification. March 22.

7146. E. Garnier. Improvements in or relating to the manufacture of rubber or other similar gums. March 24.

7228. H. L. Terry and W. Marsh. A substitute for carbon bisulphide in the cold cure of india-rubber goods. March 25.

7636. E. Garnier. Improvements in or relating to the manufacture of rubber or other similar gums. March 30.

7715. J. Deborde. Improved manufacture of artificial rubber or rubber substitute. March 31.

7848. P. Mallison and Co. An improved insulating composition or paint. Complete Specification. April 1.

8045. K. L. V. Zimmer and The Siderosthen Patent Syndicate, Ltd. Improvements relating to paints suitable for coating iron and other surfaces. Complete Specification. April 4.

8106. H. H. Lake.—From C. Curtis, United States. Improvements relating to the manufacture of a substitute for leather rubber and other flexible materials. Complete Specification. April 5.

8159. A. H. Tapp. Improvements in and in connection with the manufacture of litharge or lead oxide. April 5.

8499. A. G. Brookes.—From A. B. Kittson and A. B. Browne, United States. A new or improved pigment or paint. April 12.

### Complete Specification Accepted.

1898.

3160. A. J. Boult.—From R. G. Work and J. F. Palmer, United States. Treatment of rubber. March 30.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

6269. J. T. Wood. Improvements in the means for and in the method of bateing skins. March 15.

6352. A. M. Clark.—From Nahrmittel-Gesellschaft mit beschränkter Haftung, Germany. Improvements in the production of albumen from blood and animal organs. March 15.

6187. J. Nussbaumer. Improvements in process of tanning. Complete Specification. March 17.

6638. U. de Günzburg. An improved method of preserving and tawing skins. Complete Specification. March 18.

6689. J. K. Tullis. Improvements in treating hides and skins. March 19.

6979. J. Hutchings. Improvements in machinery or apparatus for treating hides, kips, or skins for the manufacture of leather. March 22.

7010. J. Imray.—From E. G. P. Thomas and J. Bonnvita, France. Manufacture of new or improved sizing material. March 22.

7151. A. McQuade. An improved process of treating leather, scrap paper or wood-pulp, sawdust, and the like substances for the production of imitations of various other substances. March 24.

7152. A. McQuade. An improved composition to be used as a substitute for leather and the like. March 24.

7171. E. White. Improvements in apparatus for tanning hides and skins. March 24.

8099. D. Young.—From The Vaughan Machine Company, United States. Improvements in machines for the treatment of hides or skins. Complete Specification. April 5.

8106. H. H. Lake. *See* Class XIII.

8131. E. G. Scott. Improvements in tanning. April 5.

8218. V. Quaedolieg. Process and apparatus for the production of belting leather butts. Complete Specification. April 6.

### Complete Specifications Accepted.

1897.

30,698. J. A. Bragg. Process for removing lime from skins and hides preparatory to tanning. March 23.

1898.

2400. R. Brauer. Manufacture of glue. March 23.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

7561. P. Pessé. Improvements relating to the decoloration and purification of liquids. Complete Specification. March 29.

7808. R. A. L. Hill. *See* Class XII.

8017. P. Pessé. *See* Class XII.

8204. E. H. Kelly. Milk-sugar. April 6.

8232. T. Healey and T. H. Kraft. *See* Class I.

8372. J. M. E. Fontenille and H. G. J. R. B. Desormeaux. Improved process of manufacturing sugar. April 7.

8561. R. A. Chesebrough. Improvements in the process of and means for refining sugar. Complete Specification. April 12.

8592. C. J. Crosfield and S. Stein. Improvements in or connected with the manufacture and refining of beet, cane, and other sugars. April 13.

8593. C. J. Crosfield and S. Stein. Improvements in or connected with the manufacture of cube sugar. April 13.

### Complete Specifications Accepted.

1897.

14,647. H. W. Aitken. Sugar-cane mills. April 20.

29,630. E. W. Gerbracht and F. G. Wiechmann. Process for purifying black-strap or refuse syrups. March 30.

1898.

3659. E. R. Dinsmore. Starch mixture. April 6.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Application.

8656. H. C. Williams and The Dover Electricity Supply Company, Ltd. Process for sterilising, deodorising, and generally purifying beer, cider, and other casks, vats, or containers, with the destruction of microscopic organisms, living or otherwise. April 13.

### Complete Specifications Accepted.

1897.

1902. E. Guillaume. Distilling and rectifying processes, and apparatus therefor. March 30.

9018. O. Trachmann. Process and product for the preparation of certain kinds of beer. April 20.

10,899. R. Willcox.—From The Actien-Maschinenbau-Anstalt vormals Venuleth and Ellenberger. Apparatus for the preparatory treatment of cereals for use in brewing and distilling. April 13.

13,435. A. J. Boult.—From G. R. Besser. Apparatus for treating or maturing fluids containing alcohol. April 13.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

### A.—*Foods.*

7371. T. Eves and G. Cuddon. Improved process and apparatus for preserving milk and other liquids. March 26.

7762. J. W. C. Hamilton. Improvements in the manufacture of food from blood. March 31.

7827. J. Davidson and G. Davidson. Improvements in fish food preparations. April 1.

7854. E. B. McDougall. A new food product and method of preparing the same. April 1.

8525. A. S. B. E. P. Rahgi. Improved condensed tea. April 12.

### B.—*Sanitation.*

7084. C. Zimmermann.—From the Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in disinfecting and in apparatus therefor. March 23.

7094. J. Metzger. An improved process and machine for purifying and sterilising filtering media. Complete Specification. March 23.

8695. J. J. Edmondson. Improved means for the treatment of sewage. April 14.

### C.—*Disinfectants.*

6906. J. H. Williams and W. A. McCubbin. A new and improved phenol antiseptic compound. March 22.

7089. E. T. C. Weihers. Improved mixture for the destruction of microbes of the contagious diseases of animals; also applicable for the treatment of animal skins. March 23.

7169. Sir T. G. F. Hesketh, Bart. Improvements in the manufacture of antiseptic and medicated soaps, ointments, and the like. March 24.

### Complete Specifications Accepted.

### A.—*Foods.*

1897.

11,513. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Albuminous products soluble in water from seeds of plants. March 23.

30,919. J. L. Grant. Article of food. March 23.

### B.—*Sanitation.*

1897.

8281. G. A. de Katow.—From Riensch and Co. Process and apparatus for purifying and utilising town sewerage. March 30.

9558. R. H. Reeves. Treatment of sewage. April 20.

13429. H. McPhail. Purification of liquids. April 6.

### C.—*Disinfectants.*

1898.

1723. E. Fournier. Disinfectant. March 30.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

6651. F. Bühler. Improvements in the preparation of cellulose. Complete Specification. March 18.

6940. The Westfield Paper Company, Ltd., and G. Stewart. Improvements in and relating to the surfacing of paper. March 22.

7746. C. Zimmermann.—From the Chemische Fabrik auf Actien vormals E. Schering, Germany. The manufacture of photo-genic copying paper. March 31.

7906. W. K. Trotman. Improvements in or relating to the manufacture of paper. April 2.

8235. C. W. Kimmins. Improvements in or relating to the manufacture of paper. April 6.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

6238. F. C. Doolittle. An improved tonic and medicinal preparation or composition applicable also for the manufacture of yeast, bitters, beer, &c. Complete Specification. March 14.

7097. A. M. Clark.—From L. W. Gans, trading as the Pharmaceutisches Institut, Germany. Process for the production of albumen substances and their derivatives abundantly substituted with halogen. March 23.

7681. A. Oullié. Apparatus for subliming and refining camphor. March 30.

7740. S. S. Bromhead.—From The Chemische Fabrik, Germany. An improved process for the production of iodized albumen and iodized peptones. Complete Specification, March 31.

8129. E. G. Scott. Improvements in the manufacture of acetone. April 5.

### Complete Specifications Accepted.

1897.

8671. B. C. Hinman. *See* Class VII.

9898. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of a new chemical compound. March 23.

10,790. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. New pharmaceutical compounds. April 13.

11,228. A. Zimmermann.—From the Chemische Fabrik auf Actien vormals E. Schering. Sterile iodoform. April 6.

11,596. J. Wetter.—From J. D. Riedel. Process for the manufacture of salicylo-acetic acid. March 23.

13,993. H. Imray.—From F. Hoffmann, La Roche, and Co. Manufacture of bismuth oxyiodide pyro-gallate, April 6.

1898.

4280. J. C. Newburn.—From Chemische Fabrik von Heyden Gesellschaft mit Beschraenkter Haftung, Germany. Employment and administration of vanillin. April 13.

## XXI.—PHOTOGRAPHY.

### Applications.

6879. J. Paschka. Improvements in the production of photographs with metallic lustre. March 22.

6910. T. R. Watson. New or improved coloured screens for use in photography. March 22.

7971. J. Joly. Improvements in or relating to photography in colours. April 4.

### Complete Specification Accepted.

1897.

21,679. G. A. de Katow.—From A. Schwarz, Germany. Method of and apparatus for toning photographs. March 23.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

6228. P. Cornet. The manufacture of a new explosive. March 14.

6967. W. Salter. Improvements in deflagrating compositions for matches. March 22.

7100. H. P. Hurst. Improvements in explosive shells, and in fuses or fuse fittings for detonating the explosive. Complete Specification. March 23.

7185. P. Marquart. Manufacture of lucifer match composition. March 24.

7562. H. Ritter von Dahmen. Improvements in the manufacture of safety explosives. March 29.

7579. W. Weiffenbach. An improved firework, and process of making the same. Complete Specification. March 29.

8014. L. Muller. An improved process for increasing the resistance of explosives to atmospheric influences, and for increasing their explosive power. Complete Specification. April 4.

8468. A. T. Dawson and J. Karstairs. An improvement in explosives. April 9.

8470. A. T. Dawson and J. Karstairs. Improvements in the manufacture of explosives. April 9.

### Complete Specifications Accepted.

1897.

13,724. E. A. G. Street. Manufacture of explosives and explosive substances. April 6.

13,822. S. Fulop and J. Leckovic. Improved blasting powder. March 23.

## PATENTS UNCLASSIFIABLE.

### Applications.

8582. A. J. Boult.—From L. Hiltner, Germany. Improved process for the treatment of beet-root and other seeds. April 12.

### Complete Specifications Accepted.

1897.

15,170. W. P. Thompson.—From M. Rosenbaum, Germany. Production of porous substances. April 20.

27,121. W. Leuscher. Process for the manufacture of burnt filtering mass. April 13.

* * *

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| H. T. Pentermann | XVI., XVII. |
| J. C. Richardson | XI. |
| F. W. Renaut | Patent List. |
| G. H. Robertson | XI. |
| Chas. Salter | I., II., III., XV., XVI. |
| R. Sandon | II. |
| H. H. B. Shepherd | IX. |
| J. Shields, D.Sc., Ph.D. | Gen. Chem. |
| A. Shonk | Gen. Chem. |
| W. P. Skertchley | XII., XVIII., XX. |
| E. Sonstadt | III., VII., X. |
| A. L. Stern, D.Sc. | XVII. |
| Eustace Thomas, B.Sc. | XI. |
| E. Howard Tripp, Ph.D. | III., VII., XVI. |
| V. H. Veley, M.A., F.R.S. | Gen. Chem. |
| C. Otto Weber, Ph.D. | IV., XIII. |
| L. J. de Whalley, B.Sc. | XVI. |
| E. W. Wheelwright, Ph.D. | XXII. |
| A. Wingham | X. |
| Joseph T. Wood | XIV. |
| A. C. Wright, B.Sc., M.A. | IV., XVIII., XX. |

## Liverpool Section.

*Chairman : Charles A. Kohn.*
*Vice-Chairman : E. K. Muspratt.*

*Committee :*

*P. Bateson.*
J. Campbell Brown.
Eustace Carey.
M. Lloyd Davies.
*W. D. Herman.*
W. Norris Jones.
*A. Smetham.*
T. W. Stuart.
Frank Tate.
*A. Watt.*

*Hon. Treasurer :* W. P. Thompson.

*Hon. Local Secretary :*
T. Lewis Bailey, University College, Liverpool.

Newly elected members taking office in July next :—*Chairman :* A. Smetham. *Vice-Chairman :* Charles A. Kohn. *Committee :* C. L. Higgins, E. K. Muspratt, Max. Muspratt, E. Rhodes.

## London Section.

*Chairman : R. Messel.*
*Vice-Chairman : Boverton Redwood.*

*Committee :*

E. J. Bevan.
B. Blount.
*W. J. Dibdin.*
*R. J. Friswell.*
Otto Hehner.
*D. Howard.*
J. B. Knight.
A. R. Ling.
*H. de Mosenthal.*
B. E. R. Newlands.
F. B. Power.
F. Napier Sutton.
*Wm. Thorp.*
T. Tyrer.

*Hon. Local Secretary :*
*John Heron*, 110, Fenchurch Street, E.C.

Newly elected members taking office in July next :—*Chairman :* Boverton Redwood. *Secretary :* A. R. Ling. *Committee :* E. Grant Hooper, D. L. Howard, W. J. Leonard, Walter Reid, and Frank Wilson.

SESSION 1897-98.

Monday, June 6th.—Prof. V. B. Lewes. "The Conditions Existing in Acetylene Generators."

Monday, June 20th.—Dr. Hans Goldschmidt and Mr. Claude Vautin. "Aluminium as a Heating and Reducing Agent."

## Manchester Section.

*Chairman : George E. Davis.*
*Vice-Chairman : D. Watson.*

*Committee :*

B. W. Gerland.
J. Grossmann.
*J. M. Irving.*
E. Knecht.
*M. J. Langdon.*
*I. Levinstein.*
J. Lewkowitsch.
*A. Liebmann.*
A. Rée.
*E. Schunck.*
C. Truby.
*W. Thomson.*

*Hon. Local Secretary :*
J. Carter Bell, The Cliff, Higher Broughton, Manchester.

Newly elected members taking office in July next :—*Chairman :* D. Watson. *Vice-Chairman :* J. Grossmann. *Committee :* G. H. Bailey, George E. Davis, H. Grimshaw, W. H. Perkin, jun., F. Scudder.

## Newcastle Section.

*Chairman :* F. S. Newall.
*Vice-Chairman :* G. T. France.

*Committee :*

A. Allhusen.
P. P. Bedson.
T. W. Hogg.
H. Louis.
T. W. Lovibond.
N. H. Martin.
*John Morrison.*
John Pattinson.
W. W. Proctor.
W. L. Rennoldson.
G. Sisson.
J. E. Stead.

*Hon. Local Secretary and Treasurer :*
Saville Shaw, Durham College of Science, Newcastle-on-Tyne.

Newly elected member taking office in July next :—*Committee :* J. E. Stock.

## New York Section.

*Chairman : Charles F. Chandler.*
*Vice-Chairman : T. J. Parker.*

*Committee :*

H. Clementson.
*Virgil Coblentz.*
*H. Endemann.*
W. F. Fuerst.
*Jas. Hartford.*
W. D. Horne.
E. J. Lederle.
G. A. Prochazka.
*Clifford Richardson.*
*Wm. Jay Schieffelin.*
*R. C. Schüpphaus.*
G. W. Thompson.
Max. Toch.

*Hon. Treasurer :* R. C. Woodcock.

*Hon. Local Secretary :*
H. Schweitzer, 40, Stone Street, New York, U.S.A.

Newly elected members taking office in July next :—*Chairman :* T. J. Parker. *Vice-Chairman :* Clifford Richardson. *Committee :* C. F. Chandler, B. B. Goldsmith, J. Hasslacher, H. T. Jarrett, C. E. Pellew, and Claude A. O. Rosell.

## Nottingham Section.

*Chairman :* J. O'Sullivan.
*Vice-Chairman :* J. M. C. Paton.

*Committee :*

L. Archbutt.
S. F. Burford.
F. J. R. Carulla.
R. M. Caven.
*F. Clowes.*
H. Forth.
W. G. Johnston.
J. F. Kempson.
F. E. Lott.
J. J. Sudborough.
G. J. Ward.

*Treasurer :* S. J. Pentecost.

*Hon. Local Secretary :*
J. T. Wood, 29, Musters Road, West Bridgeford, Nottingham.

Newly elected member taking office in July next :—*Committee :* F. Stanley Kipping.

## Scottish Section.

*Chairman :* G. T. Beilby.
*Vice-Chairman :* G. G. Henderson.

*Committee :*

A. C. J. Charlier.
John Clark.
*R. Cox.*
C. A. Fawsitt.
J. Gray.
*D. Harris.*
R. A. Inglis.
Robt. Irvine.
A. Macdonald.
R. C. Menzies.
*Sir R. Pullar.*
J. B. Readman.
*H. Rose.*
P. Rottenburg.
E. C. C. Stanford.
R. T. Thomson.

*Hon. Secretary and Treasurer :*
Thomas Gray, Technical College, 204, George Street, Glasgow.

Newly elected members taking office in July next :—*Committee :* A. P. Aitken, J. Christie, W. Foulis, W. Frew.

## Yorkshire Section.

*Chairman : Thos. Fairley.*
*Vice-Chairman : Christopher Rawson.*

*Committee :*

*H. E. Aykroyd.*
*F. W. Branson.*
J. Cohen.
*N. Farrant.*
T. Glendinning.
A. Hess.
J. J. Hummel.
W. McD. Mackey.
H. R. Procter.
F. W. Richardson.
A. Smithells.
*Geo. Ward.*

*Hon. Local Secretary and Treasurer :*
H. R. Procter, The Yorkshire College, Leeds.

Newly elected members taking office in July next :—*Chairman :* Christopher Rawson. *Vice-Chairman :* George Ward. *Committee :* J. E. Bedford, H. K. Burnett, T. Fairley, and G. W. Slatter.

The names in *italics* are those of Members of Committee who retire at the end of the current session.

## NOTICES.

### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Nottingham on the 13th, 14th, and 15th July next. A Programme is issued with this number. Tickets of Membership will be issued in time for the meeting.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. George Beilby has been nominated to the office of President; Prof. F. Clowes, D.Sc., has been nominated Vice-President under Rule 11; and Mr. George E. Davis, Mr. John Heron, and Dr. Chas. A. Kohn have been nominated Vice-Presidents under Rule 8.

Sir Robert Pullar has been nominated an Ordinary Member of Council under Rule 24, and the Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Members are hereby invited to nominate fit and proper persons to fill four vacancies among the Ordinary Members of Council. Forms for this purpose can be obtained from the General Secretary on application.

*Extract from Rule* 18:—"No such nomination shall be valid unless it be signed by at least ten Members of the Society who are not in arrear with their subscriptions, nor unless it be received by the General Secretary, at the Society's Office, at least one month before the date of the Annual General Meeting, to the election to take place at which it refers. Nor shall any such nomination be valid if the person nominated be ineligible for election under Rules 12 or 15. No member shall sign more than one nomination form."

### ALTERATION OF BYELAWS.

Notice is hereby given, in accordance with Rule 54 of the Bye-laws, that the Council will propose to the forthcoming Annual General Meeting the amendment of the Society's Bye-laws as follows:—

1. Rule 7. To add rule 19 to the list of exceptions.
2. Rule 19. To add "and no ballot shall be taken. If four persons only be nominated, no ballot shall be taken."
3. Rule 20. To add at beginning "If the number of persons nominated, as provided by Bye-law 18, be more than four—"
4. Rule 56. To replace the words "be at the discretion" by the words "subject to the sanction."

### COLLECTIVE INDEX.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

| | |
|---|---|
| To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List. | Each copy 10*s*. |
| To Subscribers .................... | „ 12*s*. 6*d*. |
| To others ......................... | „ 15*s*. |

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

### TELEGRAMS.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

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### LIST OF MEMBERS ELECTED 23rd MAY 1898.

Andreoli, Marcel, Rand Central Ore Reduction Co., P.O. Box 1891, Johannesburg, Civil Engineer.

Beck, Herbert H., 20, South Prince Street, Lancaster, Pa., U.S.A., Chemist.

Busch, Dr. Albert, 21, Laurastrasse, Elberfeld, Germany, Chemist.

Crichton, Jas., Raeburn, Stonebridge Park, Willesden, N.W., Merchant.

Dent, Dr. Frankland, 93, Holly Avenue, Newcastle-on-Tyne, Consulting Chemist.

Duguid, Jas., 2, Sotheby Road, Highbury, N., Journalist.

Olden, Chas., The Oaklands, Warley, Langley, Birmingham, Metallurgical Engineer.

Richmond, Jno. R., Woodend Park, Grassendale, Liverpool, Alkali Works Manager.

Sen (Gupta), Nagendra Nath, 18, Lower Chitpur Road, Calcutta, India, Physician and Chemist.

Smith, H. Gibertson, Hollies, Park Hill, Clapham, S.W.

Smith, R. F. Wood, Laboratory, 89, Bartholomew Close, E.C., Consulting Chemist.

Stevens, Arthur F., 12, Versailles Road, Anerley, S.E., Paper Examiner.

Ward, John, Barnstone Blue Lias Lime Co., Ltd., Barnstone, Notts, Manager.

Young, W. Gathorne, G.N.R. Plant, Doncaster, Yorks, Chief Chemist.

### CHANGES OF ADDRESS.

Barrett, A. A., l/o Corso Vittorio Emanuele; 5, Strada del Pozzo Leone, Messina, Sicily.

Beckett, G. H., l/o Sweden; Wolston, near Coventry.

Beufey, Dr. Hans, l/o Plymouth Grove; 128, Upper Brook Street, Manchester.

Boothby, Chas., l/o Brixton; 12, Brookfield Road, South Hackney, N.E.

Brothers, H. E., l/o Tunstall; Highfield House, Goldenhill, Stoke-on-Trent.

Chadwick, W. N., l/o South Shields; 24, West 3rd Street, Bayonne, N.J., U.S.A.

Conroy, Dr. Jas. T., l/o New Brighton; 12, Howard Drive, Cressington, Liverpool.

Cullen, Wm., l/o Ardeer; The Thames, Stanford-le-Hope, Essex.

Edwards, H. W., l/o Denver; Glasdir Copper Mines, near Dolgelly, Wales.

Erskine, J. K., l/o Box 540; c/o New Kleinfontein Co., per private bag, Johannesburg, S.A.R.

Farmer, J. E., l/o Salford; c/o J. J. Cooper, Percy Road, Beddington Corner, near Mitcham.

Gane, Eustace H., l/o Ann Street; 95, Fulton Street New York, U.S.A.

Greenway, T. J., l/o Harrow Road; Marlboro' Street, College Park, Adelaide, S. Australia.

Gref, Anthony, l/o 253; 141, Broadway, New York, U.S.A.

Heriot, T. H. P.; Journals to Pln. Windsor Forest, West Coast, Demerara, until end of year; then to Trinidad as before.

Herriot, W. Scott; l/o Osmaston Road; 11, Rose Hill Street, Derby.

Higgin, W. H., l/o Hopefield; Hollywood, Lostock, near Bolton.

Huntington, Dr. Harwood, l/o Front Street; 260, West Broadway, New York, U.S.A.

Jones, Martin L., l/o Glasgow; 3, Rugby Road, Neath, South Wales.

Labonde, Dr. Leon, l/o Washington Avenue; 174, Lincoln Avenue, Newark, N.J., U.S.A.

Leslie, Hugh M., l/o Glasgow; Marikuppam, Mysore State, South India.

McNaughtan, W., l/o Calder Street; 11, Albert Road (East), Crosshill, Glasgow.

Marshall, Dr. T. Rhymer; Journals to 19, Sandiford Road, Glasgow, Professor of Chemistry.

Pidduck, E. W., l/o Southport; Chemical Laboratory, University College of Wales, Aberystwith.

Pope, Samuel, l/o Handsworth; 35, Victoria Road, Runcorn.

Root, W. Lacy, l/o Boston; 11, Church Street, Pittsfield, Mass., U.S.A.

Shaw, F. W., l/o Chorley; Temple House, Heapey, near Chorley.

Simpson, W. S., l/o Stamford Hill; Halcyon House, Private Road, Enfield, N.

Slatter, G. W., l/o Saltaire; 12, Bromley Road, Shipley, Yorks.

Takamine, J., l/o Chicago; c/o Parke, Davis, and Co., 8, South Howard Street, Baltimore, Md., U.S.A.

Thiel, Dr. C. Walther; Journals to c/o F. Reddaway and Co., Ltd., Pendleton.

Towns, Jas., l/o Perranporth; 27, Sydney Grove, Newcastle-on-Tyne.

Vogel, J. L. F., l/o East Molesey; 91, Blackfriars Road, S.E.

Wallace, Robt., l/o Coates Place; 20, Murrayfield Avenue, Edinburgh.

Watson, Chas., l/o Manchester; The Primitiva Gas Co., Buenos Ayres, Argentina.

Wingate, H. M., l/o Glasgow; P.O. Box 19, Paeroa, Thames, New Zealand.

Witthaus, Dr. R. A., l/o West 77th Street; 414, East 26th Street, New York, U.S.A.

## Death.

Cochrane, Chas., Green Royde, Pedmore, near Stourbridge. May 11th.

## Liverpool Section.

*Meeting held Wednesday, April 27th*, 1898.

DR. C. A. KOHN IN THE CHAIR.

### THE LIFE AND WORK OF FERDINAND HURTER, Ph.D.

CONTRIBUTED BY MEMBERS OF THE LIVERPOOL SECTION.

Few members of the Society of Chemical Industry and certainly no member of this Section of the Society have stood so deservedly prominent throughout the 16 years of the Society's existence as Dr. Ferdinand Hurter.

As one of the original members of the Society, a member of the committee of the Liverpool Section of the Society from its commencement, a member of the publication committee for 15 years, twice a vice-president of the Society, and twice chairman of the Liverpool Section (1889–90, and 1890–91), he was most intimately connected with the progress and the development of the Society, and there is no department of its work that has not been enriched by his wise suggestions and ever ready help.

But whilst as one among many helpers, personal recollections will alone hand down the record of his labours on behalf of the Society in this respect, the pages of the journal will enrich all time with his magnificent contributions.

His wide knowledge and consummate skill in bringing the highest branches of pure science to bear upon the details of practical working were unique, whilst his ardour and capacity for work were only equalled by the masterly grasp and enthusiasm with which he communicated his discoveries.

A paper by Hurter was always looked forward to with a full conviction of its importance, and his hearers were always charmed by the special directness of his methods and the lucidity with which he brought forward his conclusions. Completeness and exceeding care characterised all his work, whether a small communication or an elaborate investigation. He made no distinction in the quality of his researches; if a subject, however trivial, were worth investigating, it was worth his best efforts. Criticism of his work was always received with the greatest cordiality and consideration. No critic left a discussion with Hurter without being markedly a wiser, though often a sadder man, for few drove home their points more effectively than he did. His criticism of the work of others, if severe, carried but the severity of an ardent searcher after truth; theoretical considerations alone never satisfied him; his views were invariably founded on his own direct experiments.

Such work has been an ennobling example to this Section for 16 years.

To-day it is our sad duty to record its end. Although during the last two years Hurter's untiring activity had somewhat taxed his health, his death, on March 12, 1898, was extremely sudden and unexpected.

We already miss him in this Section, but the loss is one extending far beyond the bounds of Liverpool, and the Society of Chemical Industry; it is the loss of one of the most able workers in applied science, during a period of unprecedented activity in the development of manufacturing chemistry. Future generations of chemists will appreciate Hurter's share in this development as fully, perhaps more fully, than we do to-day. To us, whether as a personal friend, a fellow-worker, or a fellow-scientist, he was further endeared by all the charms of greatness, simplicity, sympathy, and sincerity.

Ferdinand Hurter was born at Schaffhausen, on March 15, 1844. He was educated at the State schools and gymnasium of his native town until 16 years old, when he went to Winterthur to be apprenticed to a dyer, with whom he remained for three years. By this time he gave such evidence of capacity and ability that it was deemed desirable that he should pursue his studies further than circumstances had hitherto permitted. With this object he went to the Polytechnic at Zürich, where he studied under Stadeler, and subsequently to Heidelberg, where his studies were completed under Bunsen and Kirchhoff. In 1866 he took his degree of Doctor of Philosophy at Heidelberg.

In 1867 Hurter came to England, where the first position he occupied was that of chemist to Messrs. Gaskell, Deacon, and Co., alkali manufacturers, Widnes. In this office his marked ability was soon appreciated, and he rapidly gained the confidence of his employers, with whom he remained until the amalgamation of the alkali works took place in 1890. He was then appointed chief chemist and scientific adviser to the United Alkali Co., in which capacity he remained until his death.

Hurter's contributions to manufacturing chemistry necessarily formed the essential part of his life's work and his published papers record but a small portion of his activity in this connection.

It is no easy task to record his share in the many advances associated with the development of the alkali industry during his lifetime, but the following statement of his chief lines of work will serve to show the many-sidedness of his originality and power of application.

As chemist to Messrs. Gaskell, Deacon, and Co., his early work was naturally in connection with the *Deacon chlorine process*, to the successful development of which he contributed very largely. Subsequently the *manufacture of sodium bicarbonate* occupied his attention. His patents on this subject taken out in conjunction with members of his firm, resulted in a process which was successfully worked for many years. Closely connected with the foregoing are his investigations on *the removal of impurities from Leblanc vat liquors* for the purpose of obtaining pure alkali direct from black ash. The patents in connection with the *removal of cyanides from vat liquors* and for *the manufacture of crystal carbonate* established a process which only surrendered recently to the powerful rivalry of the ammonia soda process. It was in connection with the former that he worked out his methods for *the analysis of cyanogen compounds*.

In 1885 he obtained a patent in conjunction with his firm for the "*treatment of sulphate of ammonia for obtaining the ammonia in the free state and utilising the sulphuric acid*." The process is an extremely interesting one, as the decomposition of sodium sulphate and ammonium sulphate in presence of steam, into sodium bisulphate and free ammonia had not been previously observed. During the last few years, Hurter devoted considerable time to *methods for the preparation of sulphocyanides and cyanides*; his numerous patents in association with his assistants record his activity in this direction. One who was long associated with Hurter speaks as follows of his technical ability and method :—

"He had the advantage of a sound and wide education during his student years; but to his credit it may be said that by strenuous and unremitting effort he continued his education during his whole life. He was an intellectual athlete, and kept himself in splendid mental training by increasing practice. The faculties of his mind were under complete control, and he possessed a most retentive memory, so that whenever occasion required, he could set himself to work, bring out of his treasury things new and old, and solve technical problems with amazing rapidity and accuracy.

"Amongst the most marked elements of his success as a technical chemist were the ability he possessed in a remarkable degree of devising new apparatus for new problems and his insight into the nature of the technical and commercial problem before him. He was never lured into any side-issue, however interesting from a scientific point of view; he kept but one end in sight, the solution of the problem in hand, and his grasp of the situation, both technically and commercially, was always complete and clear."

The Journal of this Society is honoured by the best of Hurter's published work. These investigations are essentially concerned with problems affecting the industry with which he was connected. The very first paper contributed to the Liverpool Section of the Society was by Hurter : "*The dynamic theory of the manufacture of sulphuric acid*." This paper, whilst able and exhaustive, has been generally considered beyond the domain of practical working conditions. It is replete with characteristic mathematical considerations of a manufacturing process, and gives evidence of a line of thought which in subsequent papers was brought much nearer to the requirements of practical problems.

In 1883 he published a valuable paper entitled "*Prospects of the manufacture of chlorine*." The methods in use for the manufacture of chlorine are reviewed and their relative values compared, especially the rival Deacon and Weldon processes, as well as the "modified Weldon process," which formed the beginning of the Weldon-Pechiney process. The conditions of decomposition of chlorine compounds for the liberation of the contained chlorine is studied on the basis of their thermochemical relationships and with characteristic insight the value of such data as a basis for comparison of the relative efficiency of the methods as manufacturing processes is most exhaustively examined. Reference is also made to the Solvay ammonia soda process and the statement that "the low cost of the carrier of chlorine has enabled the ammonia soda process to take the lead in the manufacture of soda, and the peculiarly loose combination of hydrogen and chlorine has given the Leblanc process the command of the chlorine industry" indicates the line of argument adopted in the paper. Hurter regarded this condition then (1883) as likely to remain, and his view "that the only practical way of decomposing common salt on account of the very great affinity of the two elements, sodium and chlorine, is by a double decomposition" is of interest to look back upon at a time when electrolytic methods have become serious competitors to the unrivalled processes of 15 years ago.

As a side issue, the following list of costs of the various bleaching agents is of interest. It was compiled from a consideration of Mond's suggestion for the use of hydrated calcium peroxide as a bleaching agent. The cost is calculated for such quantities as contain 16 tons of available oxygen.

| | £ |
|---|---|
| Bleaching powder.................. | 1,000 |
| Potassium chlorate ................ | 1,500 |
| „ permanganate........... | 2,550 |
| „ bichromate ............. | 5,495 |
| Barium peroxide .................... | 24,167 |
| Hydrogen peroxide................. | 74,800 |
| Potassium ferricyanide............ | 131,600 |

In introducing a discussion on "*International standard methods of sampling and testing commercial products*" in 1883, Hurter's practical knowledge and scientific attitude of mind are again evident. The difficulty of arriving at standard methods of sampling is pointed out, whilst special stress is laid upon the desirability of expressing results on unalterable units, that of soda ash for instance, on pure sodium carbonate, and not upon $Na_2O$ as in the Lancashire test, because this latter rests upon the atomic weight of sodium, which is always subject to revision and possible alteration.

A paper published in 1884 "*An attempt to utilise waste heat by Perkin's pipes*," deserves passing notice. Hurter shows that although it is not possible to obtain a figure for the value of waste heat of general applicability, its cost can be ascertained for particular applications. In this case the value of waste heat for the evaporation of liquids is studied, and its cost (reckoned simply as the interest on the capital expended on the apparatus employed, the heat itself being taken as of no value) compared with that of heat raised direct from coal. It is clearly proved that the value of this waste product depends upon its temperature, and that in this country economy involves a temperature of at least 220° C. Hurter records the failure of attempts to use the waste heat from a black ash revolver, by means of coils of pipes on Perkin's high-pressure system. Mathematical considerations trace this to the too slow circulation and consequent too great friction in the pipes. It is interesting to note that the extensive and useful applications of mathematics made throughout Hurter's work were almost entirely the outcome of his own teaching.

In the same year (1884) Hurter read the first of a series of papers on "*The comparative efficiency of various modes of treating liquids with gases*," which form his most valuable contribution to applied chemistry.

These papers were a further outcome of his desire (the paper on the "Dynamic Theory of the Manufacture of Sulphuric Acid" being the first) to gather into a concise form, or formulate so that it could be readily transmitted, the experience gained by practical men during many years of work. In the first paper he shows that all apparatus for treating liquids with gases can be divided into three classes: (1.) The gas divided into small bubbles passing through a column of liquid. (2.) The liquid divided into fine drops falling through a column of gas. (3.) Solids with extensive surfaces covered with the liquid, the gas filling up the interstices. In order to compare these three classes of apparatus it was necessary to have a unit of contact, for upon the intimacy of the contact between the gas and the liquid depends, to a great extent, the value of the apparatus. He defines the unit of contact to be that unknown number of molecules of a gas which would impinge in one second upon one square metre area, if the gas were under a pressure of one metre of water. The mechanical efficiency of any piece of apparatus could be measured by the number of such units of contact yielded by a given expenditure of work. Apparatus belonging to class (1) is discussed, and experiments are described made for determining the size and speed of gas bubbles under varying conditions, such as height of liquid, diameter of orifice of distributing pipe, and nature of gas. The velocity was found to be uniform and, approximately, 1 ft. per second. The size was found to vary with the diameter of the orifice from which they issued, and the nature of the liquid, and to be independent of the depth of liquid and the nature of the gas. After indicating how the number of units of contact produced by a bubble in rising would be calculated, he gives formulæ for determining the relative efficiencies of the three modifications of apparatus in which bubbles of gas are made to rise through the liquid. (1) The gas is simply blown through the liquid. (2) The gas is pulled through the liquid. (3) The gas is blown through under pressure. Of these the first is shown to be by far the cheapest, and stress is laid upon the necessity of carefully considering the chemical side of the question, as well as the mechanical, if the efficiency as represented by the cost is to be kept up to the maximum.

In the second paper some of the usual methods of distributing a gas at the bottom of a column of liquid are considered and a number of experiments described, which were made with a view of determining the relation between the total area of the perforations and the amount of gas to be distributed. These experiments show that when the supply of gas gets below a certain minimum fewer and fewer of the perforations deliver gas, but that the amount delivered from each of those that do work is just the same as when they are all working. If the amount of gas is more than sufficient to keep all the perforations supplied, then each one delivers more, but all alike. By further experiment it was found that the discharge of gas from an orifice under water obeys the same law as the discharge from a similar orifice into air with the same hydraulic head. Taking the smallest pressure at which all the perforations of a distributor are supplied as ¾″, the linear velocity of the discharge is 35′ per second, and, dividing the total amount of gas to be distributed by 35, the total area of the perforation is obtained. The paper concludes with a series of calculations showing, from a case in actual practice (carbonation of vat liquors), that when a gas is blown through a liquid there is a certain depth of the liquid which gives the most economical results, depending on the cost of compression, the value of the gas, and the rapidity with which the useful constituent of the gas is taken up by the liquid. The Weldon process is instanced as a case in which it is profitable to use a considerable depth of liquid, in spite of the extra cost of compression, on account of the slowness with which the gas is absorbed.

In March 1893 the third and last of his series of papers dealing with this subject was read. In it Hurter discusses, first, the class of apparatus in which the liquid divided into fine drops falls through a column of gas; second, the coke condenser, as the representative of that class of apparatus in which solids with extensive surfaces and interstices are covered with the liquid, the gas passing through the interstices. After pointing out that in most technical processes the volume of the gas is at least 100, more often 1,000, times the bulk of the liquid, mathematical proof is given of the superiority of the method of subdividing the gas over the method of subdividing the liquid, both as regards the units of contact obtained and the consumption of mechanical energy. The reasons for the great efficiency of the coke tower are next examined. On account of the large rough surface of the coke and the slowness with which a stream of water descends over it, the liquid contact surface is very great, and is active upon the particles of the stream of gas for a period depending upon the rate at which the gas is passed through. By calculation a formula is arrived at for expressing the amount of contact surface available per unit volume of gas per second, in terms of the total surface exposed, and the volume of gas passing per second. Using this formula as a measure of the contact, the three classes of apparatus are compared, and the coke tower is shown to be the most efficient. After pointing out that another great advantage of the coke tower is the large amount of liquid which it holds, and describing an experiment made for the purpose of investigating the distribution of the liquid, the plate tower is compared with the coke tower, and the results of his investigations of the methods of treating liquids with gases are summed up as follows:—The subdivision of the liquid into drops or spray is of little practical use and requires the expenditure of too much mechanical power for a given effect. The subdivision of the gas into bubbles is better, but the cheapest and best method of presenting a liquid to a gas is by means of a coke tower. The comparative value of any apparatus for producing contact is the amount of total surface presented to 1 cb. ft. of the gas per second, the formula referred to being a true measure of the value of such apparatus.

In November 1889 a most important paper was read on "*The condensation of hydrochloric acid.*" Many years of experience in dealing with hydrochloric acid on the large scale had forcibly brought before Hurter's mind the great want of a theory of the condensation of hydrochloric acid which would answer satisfactorily questions continually presenting themselves in practice, such, for instance, as the following: What is the highest strength of liquid hydrochloric acid that can be obtained from a gas of known composition, and what conditions must be satisfied in order to obtain acid of that strength? As such a theory did not exist this paper is an attempt to supply a method of solving problems of the kind arithmetically, based upon the best known experimental researches, especially those of Hammerl, Berthelot, and Roscoe and Dittmar.

From the data given by Hammerl and Berthelot he shows for any percentage of acid, the amount of hydrochloric acid per gramme of water, the units of heat evolved, and the rise of temperature during its formation. A table representing these results is given which shows very plainly the large amount of heat generated by the condensation of hydrochloric acid gas. It is then pointed out that, owing to the low specific heat of hydrochloric acid gas, cooling previous to condensation, is of little practical importance, the great amount of heat to be removed being only generated when and where the gas comes into contact with water.

A second table shows the maximum tension of aqueous vapour in presence of hydrochloric acid, and the minimum tension of hydrochloric acid, at different temperatures. From these figures it is seen that much less steam can exist in hydrochloric acid gas than in other gases at the same temperature. The fact is thus explained, that when hydrochloric acid gas escapes into damp air a large amount of the aqueous vapour is at once thrown down as fog.

By means of these tables the work done by cooling pipes can be calculated, and the influence which the admixture of air and steam has on the condensation can be shown. Instances are given of the agreement of the results so calculated with those obtained in actual practice and the great importance of previous cooling when the gas contains steam is clearly demonstrated. In order to answer the question, What is the highest strength of acid obtainable from a gas of given composition under given conditions? it is necessary to know how the amount of hydrochloric acid absorbed by a given amount of water varies with the temperature and the partial pressure of the gas. A formula is given which will calculate, approximately, this amount, using as a basis the researches of Roscoe and Dittmar, and tables are added to facilitate calculations by means of the formula. These tabulated results render it possible to calculate approximately the alterations which may occur during the cooling of the gas, the evolution of heat, the amount of condensation, and the strength of acid formed. A most carefully carried out experiment to determine the heat dissipated by the sides of a coke condenser is next described, and having shown how, by means of this quantity and the other data given in the paper, the work that stone condensers will do can be calculated and the close agreement of such calculations with actual work, this most interesting and instructive paper is brought to a close by briefly referring to another part of the subject, *viz.*, the amount of hydrochloric acid which an inert gas will carry away if it passes through a liquid acid of given strength.

In a very interesting address, as chairman of the Liverpool section in 1888, Hurter gives a valuable comparison of the "*Relative efficiencies of the various alkali processes.*" The different methods for splitting up sodium chloride and water into caustic soda and chlorine are discussed from a thermochemical point of view. He refers to the usual methods of calculating the theoretical amount of heat necessary to bring about the reactions of the Leblanc and ammonia-soda processes, which show that the quantity actually used in practice is at least 50 times this amount. Such methods of calculation he shows to be erroneous, for the simple reason that when the products of combustion of the fuel have cooled down to that temperature at which only the reaction will proceed, the heat which they contain is no longer available for that reaction, since heat cannot pass from a colder to a hotter substance. The reaction in the Leblanc revolver is criticised from a similar standpoint, and it is clearly shown that far from being the wasteful process generally supposed, it has an efficiency comparable with that of the best caloric engines.

The increasing interest and importance of electrolytic methods of manufacture attracted Hurter's interest at an early stage, and from 1887 onwards he made many investigations on the commercial value of electrolysis in relation to the alkali industry. His published work on this subject commences with a criticism of "*The Hermite process of electrolytic bleaching.*" In 1887, Messrs. Cross and Bevan read a paper on this subject before the London section of the Society, in which they contrasted the efficiency of bleaching powder and of electrolysed magnesium chloride solution. From their results they concluded that "(*a*) the bleaching efficiency of the electrolysed solution is considerably in excess of that of a solution of sodium hypochlorite, measured in terms of the usual standard, an alkaline solution of arsenious acid; (*b*) the oxidising efficiency (*i.e.*, free or active oxygen) thus measured and expressed in terms of the current is in excess of that calculated on the basis of the electrolytic law, as directly interpreted."

These conclusions, especially the latter, appeared to Hurter so much at variance with his own experience that he at once subjected them to the test of independent experiment. These experiments were conducted with apparatus specially designed for obtaining all the data that could possibly throw light on the progress of the decomposition and the efficiency of the process. All possible reactions, both primary and secondary, were carefully considered, and the results were such that Hurter could not accept the view of the formation of bleaching compounds of very "high tension." The production of oxidation products of chlorine he could not detect at all, and he was consequently obliged to ignore their existence, nor did he find any proof that the results obtainable were above what is theoretically required according to Faraday's law.

The possibilities of "*Electrolytic methods of manufacturing caustic soda and bleach from aqueous solutions of salt*" are again the subject of Hurter's thought in the second portion of his address to the Section in 1888. In this paper it is pointed out that the relative superiority of electrolytic methods lies in the fact that the energy can be supplied at ordinary temperatures, and that in transforming heat into mechanical work, greater intervals of temperature can be made use of than in the direct application of heat. Their special disadvantage is that the separation of the products of the reaction, caustic soda, and chlorine presents very great difficulties, whereas in the Leblanc and ammonia soda processes this separation is easy and practically complete. Such general considerations, always based on sound experiment and argument, have been of the utmost service in considering the possibilities of new processes or in pointing to their fundamental fallacies.

One of the most interesting of Hurter's papers on "*Electrolysis*" was given before the Liverpool Physical Society in 1893, a society with which he was intimately connected for many years. This paper was his inaugural address as President of the Society. As it has never appeared in any form in the Journal, a somewhat full account of its contents for which we are indebted to Dr. J. L. Howard, may be of interest.

It deals with the application of the physical laws of electrolysis to the technical problem of the electrical conversion of common salt into caustic soda. Hurter begins by explaining Faraday's law that every monad atom carries an equal charge, every dyad atom double that charge, and so on, the charge being independent of the nature of the atom, but proportional to its valency. It is then indicated that, although this law is accurately true in all cases, it by no means follows that the same quantity of electricity will always liberate the same amount of any given substance; in fact, this condition is only fulfilled when the current passes through a single electrolyte which does not change its constitution during the electrolysis. In ordinary cases of electrolytic decomposition secondary reactions during the process give rise to products which invariably reduce the efficiency of the operation.

In decomposing common salt, using carbon electrodes, several secondary reactions occur, among which are (1) the formation of hypochlorites and chlorates by the action of the liberated chlorine on the sodium hydrate; (2) the production of some oxygen by decomposition of the water present; and (3) the conveyance of a portion of the current by means of the sodium hydrate, such portion consequently producing no decomposition of sodium chloride. To these must be added (4) the phenomena of migration of the ions investigated by Hittorf and others, which cause differences in the percentage composition of the solution round the two electrodes. Of these effects, (3) and (4) are the most important, and are discussed at length. Experiments on the electrical conductivity of mixtures of sodium hydrate and chloride, made by Dr. Hurter and Mr. Auer, are described; these furnish an empirical formula from which the conductivity of any specified mixture of these substances containing 5 grm. molecules of sodium in a litre can be calculated. Assuming the electrolytic cell to contain initially only sodium chloride solution with 5-grm. molecules (292·5 grms.) to the litre, and that the solution is well stirred throughout the whole time of passage of the current, Hurter calculates that the percentage of current utilised in decomposing the chloride (the *efficiency* of the current) would decrease as follows:—

| Per Cent. of NaCl converted into NaHO. | Efficiency at any Moment. | | Mean Efficiency for whole Period. | |
|---|---|---|---|---|
| | I. | II. | I. | II. |
| 0 | 1·00 | 1·00 | 1·00 | 1·00 |
| 20 | 0·718 | 0·80 | 0·847 | 0·898 |
| 40 | 0·489 | 0·60 | 0·698 | 0·784 |
| 60 | 0·298 | 0·40 | 0·546 | 0·655 |
| 80 | 0·137 | 0·20 | 0·387 | 0·497 |
| 100 | 0·00 | 0·00 | 0·00 | 0·00 |

In this table the first column gives the percentage of chloride which has been decomposed, the second and third columns give the current efficiencies at the different stages specified, while the last two columns give the efficiency of the whole operation from the commencement up to the specified stage. The numbers in the columns marked I. are calculated on the assumption that the conductivity of a mixture of chloride and hydrate is the sum of the separate conductivities of its constituents, while those in the columns marked II. are obtained by assuming the chloride and hydrate to be equally good conductors; these two assumptions correspond respectively to the conditions for least and greatest efficiency.

Finally, the effects produced by migration of ions are considered, by supposing the electrolytic cell to be filled with the same sodium chloride solution as before, and to possess a porous membrane separating the two electrodes. During the decomposition sodium travels towards the negative electrode and chlorine in the opposite direction; their velocities are, however, different, and as the operation progresses the concentrations of the solutions on the two sides of the partition will differ more and more. Hurter calculates that by the time all the salt is converted into hydrate, the useful current would only amount to 18 per cent. of the total current, and the efficiency of the whole operation would be only 40 per cent. Experiments by himself and Mr. Auer confirmed this result fairly well.

In 1895 Hurter published a further contribution to the technology of electrolysis, a paper entitled "*Notes on the Efficiency of Electrolytic Apparatus*," which was to some extent preliminary to a more elaborate paper in conjunction with Dr. Zaborsky, "*The Efficiency of an Electrolytic Cell*," the last of Hurter's contributions to the Society.

In the first of these papers Hurter points out that, in order to judge of the value of any electrolytic process, certain electrical factors are essential, *viz.*, the current efficiency, the E.M.F. of polarisation, and the resistance of the cell. The difficulties in the way of precalculating these factors is referred to, and a series of methods are described for the determination of the back E.M.F. in a cell.

In the second and more important paper, precalculation of the necessary factors for understanding the conditions of electrolysis of mixed electrolytes is the object in view, and the experimental methods and mathematical deductions are characteristically direct and exhaustive. The importance of the laws regulating the migration of ions for a proper understanding of the current efficiency in any electrolytic process is handled with great clearness and precision, and brought into special prominence in respect to the electrolysis of solutions in which the constitution of the electrolyte changes during the progress of the decomposition. A very large number of experiments are included showing how a current divides between several electrolytes in one solution, but it is distinctly stated that much more work in this direction is necessary before any exact precalculation of current efficiency can be made. This current efficiency is dependent chiefly upon the phenomena of migration, or, what is practically the same thing, the conductivity of the solutions, and although no accurate calculations can be made at present, a higher limit can always be assigned which in practice is never reached.

Amongst other contributions to technical chemistry may be mentioned a very complete paper on the "*Technology of Chloride of Lime*," published in Dingler's Polytechnisches Journal for 1877, in which the rate of absorption of the chlorine and its relation to the exposed surface of lime are dealt with.

The experiments described lead to the conclusion that the depth of the lime layer has no influence on the absorption, the chlorine in equal times penetrating to equal depths. Working then on theoretical lines, Hurter arrives at a formula in which the depth of a lime layer which is converted into 36 per cent. bleach is expressed in terms of the time and the percentage of chlorine in the gas. Inquiries into the effect of foreign gases, such as $CO_2$ and HCl, on the preparation of bleach are also recorded, and their injurious action clearly proved.

The subject of "*Denitration and loss of nitre in the vitriol manufacture*" occupied Hurter's attention to a considerable extent in 1878 and 1879, when he carried on a controversy with Prof. Lunge in this connection, the details of which are recorded in Dingler's Journal. Hurter also contributed a number of letters to the "Chemical News" in 1879 on the same subject, in which he maintained the loss of nitrous compounds in the Glover tower.

Further papers were contributed by Hurter to the two British Association meetings held in Liverpool in 1870 and 1896 respectively. On the first occasion his paper was on "*The Time needed for the completion of Chemical Change*" when he deduced, mathematically, a means of calculating the rate of reaction between such substances as sulphuric acid and zinc. From the formulæ thus obtained he calculated the time required for the oxidation of manganous oxide to manganese dioxide in the Weldon process and the calculated results were shown to agree perfectly well with those obtained practically on a large scale.

At the 1896 meeting Hurter's subject was "*The Manufacture of Chlorine by means of Nitric Acid*," and the paper, which was very neatly illustrated by experiment, aroused considerable interest. Dry hydrochloric acid, when passed into a mixture of nitric and sulphuric acids, was supposed to yield nitrogen dioxide, chlorine, and water, but Hurter found and showed that the product of the reaction is a colourless gas which decomposes spontaneously or by the action of light and heat into nitrogen dioxide and chlorine. The gas is probably $NO_2Cl$, but the process is of no commercial value on account of the expensive evaporation and concentration of materials necessary. Hurter characteristically remarked "that this latter contingency rendered the nature of the gas of no further interest to him."

Hurter's remarkable activity in all that concerned the industry with which he was connected would have earned him well-deserved leisure, but his leisure hours appear to have been no less busily engaged than those of more responsible work. Had he spared his energies more, this record might have been long delayed.

Amongst the many branches of science that interested Hurter from time to time, *Photography* stands foremost. The leisure of 10 years resulted in a brilliant and exceptionally original contribution to the science of photography, which was followed by a number of allied papers. In the whole of this work Mr. V. C. Driffield was associated with Dr. Hurter, and that their results, despite much adverse criticism in the start, were valued and appreciated is shown by their having been chosen by the Council of the Royal Photographic Society as the joint recipients of the Society's "progress medal" in January of this year.

It was in 1876 that Mr. Driffield interested Dr. Hurter in photography. The lack of scientific method and of quantitative relationships in photography as usually practised was not long in appealing to the exacting acumen of a mind like Hurter's, and the possibility of obtaining such data as should reduce the exposure of a plate to an exact science was the goal he aimed at. To realise this necessitated a sequence of investigations into an almost untrodden field of inquiry, but patient perseverance, combined with his characteristic faculty for originating new methods of inquiry and basing thereon his power of mathematical application, and aided by such a capable and experienced experimentalist as his colleague, he did succeed in his aim and thus added the long-sought factor of photographic reliability.

This work commenced when the collodion plate was the only sensitive medium at the photographer's disposal, and the first necessary step was to find means to determine the correct exposure in the camera. Numerous experiments with a variety of forms of apparatus led finally to the *Actinometer*, described in the Journal in 1890, by means of which the actinic value of diffuse daylight can be measured. Such a measurement was new, and on the results obtained with it was based the *Actinograph*, patented by Hurter and Driffield in 1888. The little instrument is a reliable and most useful aid to every photographer. Meanwhile the gelatin dry plate had come into use, and, accustomed to the uniform speed of the old collodion plates, the enormous variations in the speed of gelatin plates became a serious source of embarrassment. This variation indicated the importance of discovering some method for ascertaining the relative speed of plates.

The first step necessary to meet this need was to carry out a series of investigations in order to ascertain the law which expresses the action of light upon the sensitive plate. The only relationship between the amount of metallic silver and the exposure producing it which had hitherto been advanced was that the silver was directly proportional to the exposure, but it was quite apparent that if this relationship held good, truthful representation, by means of photography, was impossible. On the other hand, it was recognised that photography is capable of truthful representation. The immediate and most important result of these investigations was the discovery of what was termed the "characteristic curve" of the plate. This curve gave evidence of four distinct relationships between the exposure and the silver deposited, and which respectively explained the familiar phenomena of under-, correct-, over-exposure, and of reversal. It was now apparent that a negative can only yield a truthful result when its gradations fall within the correct period of the "characteristic curve" in which the silver deposited is proportional to the logarithm of the exposure.

The knowledge so far acquired proved the stepping-stone to the discovery of a method of speed determination, which not only provides a numerical expression for, and proportional to, the rapidity, but indicates other characteristic qualities of the plate.

These investigations were the subject of Hurter and Driffield's first public communication, and were published in the Journal of the Society in May 1890. Serious exception was taken to the work on two grounds. The authors had been most careful throughout in establishing a system of units and in clearly laying down definitions. Hitherto the terms "density" and "opacity" had been indiscriminately used by photographers to express the same idea, and hence considerable objection was raised when they gave different significations to these terms. Then, again, their conclusions regarding the unalterability of the density ratios were most seriously assailed by photographers who held that errors in exposure can be corrected by appropriate development. These views, however, remained unshaken, and subsequent investigations carried only an increased conviction of the fact that the doctrine of the unalterability of the density ratios is a fundamental truth which it is absolutely necessary to recognise if photography is to be practised as a quantitative science. A correct exposure was always held to be a *sine quâ non*, and the only legitimate control that there is over the gradations of the negative lies in the control which exists over the development factor.

Thus, one of the greatest needs of plate-makers and of photographers in general was supplied by enabling them, for the first time, to ascertain accurately the sensitiveness of different plates, and, by means of the actinograph, to apply this information in practice. It is not too much to say that the practical application of Hurter and Driffield's method of speed determination has been an invaluable aid to makers of dry plates, giving them a most useful means of controlling and adding to the rapidity of their plates.

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A complete list of Hurter's most important papers is appended. It remains but to add that he was joint author with Prof. Lunge of that *vade mecum* for all alkali-makers, "*The Alkali-maker's Pocket-book*," first published in 1884, and re-issued in a modified and enlarged form as the "*Alkali-maker's Handbook*" in 1891.

The object of this notice has been essentially to collate in permanent form Hurter's invaluable contributions to science, which will bring back to many of us happy recollections and associations. But in another direction it may serve a purpose; it will, we hope, keep more vividly before us than might otherwise be the case the example of one member of this Section whom we have to thank for much of our past success and present prosperity. He is no longer here to teach us how to maintain the high level of work he had so well set before us, but this record may perchance stimulate us to our best efforts in memory of a chemist whose association with this Section will always be a source of pride to its members.

### List of Dr. Hurter's Published Papers.

*The Time needed for the Completion of Chemical Change.* British Association Reports, 1870. (See Chem. News, 1870, xxii., p. 193.)

*Interaction of Arsenious Chloride and Dry Sulphuretted Hydrogen.* Chem. News, 1876, 81.

*The Technology of Chloride of Lime.* Dingl. Polytech. J. 1877, Vols. **223** and **224**.

*Denitration and Loss of Nitre in the Vitriol Manufacture.* Dingl. Polytech. J. 1878–79, Vols. **227** and **228**.

*Loss of Nitrous Compounds in the Glover-tower.* Chem. News, 1879, 170, 215, and 237.

*Volumetric Estimation of the Cyanogen Compounds in Soda-Lyes.* Chem. News, 1879, 25.

*Dynamic Theory of the Manufacture of Sulphuric Acid.* J. Soc. Chem. Ind. 1882, 8, 19, and 83.

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*Discussion on International Standard Methods of Sampling and Testing Commercial Products.* J. Soc. Chem. Ind. 1883, 441.

*An Attempt to Utilise Waste Heat by Perkin's Pipes.* J. Soc. Chem. Ind. 1884, 67.

*The Apparent Efficiency of certain Modes of Treating Liquids with Gases.* J. Soc. Chem. Ind. 1884, 639.

*On Hirsch and Folkhard's Pyrometers.* J. Soc. Chem. Ind. 1886, 634.

*On Hermite's Electrolytic Process for the Manufacture of Bleaching Powder.* J. Soc. Chem. Ind. 1887, 337.

*Comparative Efficiency of certain Modes of Treating Liquids with Gases.* J. Soc. Chem. Ind. 1887, 707.

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*Efficiency of an Electrolytic Cell.* (With B. Zahorski.) J. Soc. Chem. Ind. 1897, 97.

#### *Papers on Photography.*

*An Instrument for the Measurement of Diffuse Daylight and the Actinograph.* J. Soc. Chem. Ind. 1890, 370.

*Photochemical Investigations and a New Method of Determination of the Sensitiveness of Photographic Plates.* (With V. C. Driffield.) J. Soc. Chem. Ind. 1890, 455.

*On the Accuracy of the Grease Spot Photometer.* (With V. C. Driffield.) J. Soc. Chem. Ind. 1890, 725.

*The Sector and Grease Spot Photometers and their Results.* (With V. C. Driffield.) J. Soc. Chem. Ind. 1891, 20, 98, 318.

*Relation between Photographic Negatives and their Positives.* (With V. C. Driffield.) J. Soc. Chem. Ind. 1891, 100.

*The Action of Light on the Sensitive-film.* Proc. Liverpool Physical Soc. 1891 [Vol. I.], and Photography, Feb. 19, 1891.

*Latitude in Exposure and Speed of Plates.* (With V. C. Driffield.) Photography, July 13, 1893.

*The Latent Image and its Development.* (With V. C. Driffield.) Photographic Journal, 1898.

# London Section.

*Meeting held on Monday, May 2nd, 1898.*

DR. R. MESSEL IN THE CHAIR.

## SELF-INTENSIVE REFRIGERATION OF GASES: LIQUID AIR AND OXYGEN.

BY W. HAMPSON.

This apparatus depends upon a method by which a moderate amount of refrigeration, produced by the expansion of a gas, may be accumulated and intensified till it reaches the point at which the gas used becomes liquid under atmospheric pressure. The method consists in directing all the gas, immediately after its expansion, over the coils which contain the compressed gas that is on its way to the expansion-point. The cold developed by expansion in the first expanded gas is thus communicated to the on-coming compressed gas, which consequently expands from, and therefore to, a lower temperature than the preceding portion. It communicates in the same way its own intensified cold to the succeeding portion of compressed gas, which, in its turn, is made colder, both before and after expansion, than any that had gone before. This intensification of cooling goes on until the expansion-temperature is far lower than it was at starting; and if the apparatus be well arranged the effect is so powerful that even the small amount of cooling due to the free expansion of gas through a throttle-valve, though pronounced by Siemens and Coleman incapable of being utilised, may be made to liquefy air without using other refrigerants.

The amount of refrigeration due to free expansion was ascertained by Joule and Thomson, and is in the first place proportional to the fall of pressure. Air at 0° C. is cooled ·29 of a degree C. for every atmosphere of pressure-drop. This cooling, however, increases with the descent of the temperature from which expansion takes place, and the law is that it is inversely proportional to the square of the absolute temperature. Thus expansion of air from $4\frac{1}{2}$ atmospheres to 1, and from a temperature of 0° C., *i.e.*, 274° absolute, gives about one degree of cooling in the air itself. But when the air expands from two-thirds of that absolute temperature, *i.e.*, from − 91° C., the cooling for the same pressure-drop is nine-fourths of one degree, *i.e.*, $2\frac{1}{4}$ degrees.

The efficiency of the method depends upon the arrangement and construction of the coils and chamber forming the interchanger in which the expanded gas takes up heat from the compressed gas. To make this action as perfect as possible, the conditions to be aimed at are: having a large surface exposure between the compressed and expanded gases; having little thickness of material between them, and that little of high conductivity; having both compressed and expanded gas in small masses in close contact with the conducting material; having the total volume of the interchanger small so as to expose little surface for the entrance of external heat; and having the total mass of coils and interchanger which have to be cooled down with the gas small and of little specific heat. These are all thermal advantages, and the arrangement of parts shown in the apparatus illustrated is the most efficient hitherto designed for gases.

It has been attempted to obtain thermo-dynamic advantages by combining with a large difference of pressures a small ratio of pressures. This is done by expanding from the higher to an intermediate pressure, so as to have three pressures altogether. The action takes place in an interchanger of concentric spiral tubes, but at the cost of the thermal advantages named above; since the considerable intermediate pressure of gas in a pipe which encloses and is concentric with that for the high-pressure gas, and is itself enclosed in a third pipe for the low-pressure gas, necessitates great thickness and large mass of pipes, and prevents close coiling, thus increasing the size and mass of the interchanger, and diminishing the conductivity of the pipes.

In practice the thermal advantages are found to be the most important, so that the apparatus which embodies them not only liquefies air most quickly, but produces a given quantity of liquid with the smallest expenditure of power.

The present apparatus is capable of working continuously with a compressor able to deliver air at a pressure of 80 atmospheres or over. It will also produce liquid from a single cylinder of air or oxygen. In the latter case the coils are previously cooled by passing over them cold vapour from a cylinder of liquid carbonic acid. The conditions of working in each case are as follows:—

*I. With a compressor:*

Compression, 80 atmospheres or over.
Time for liquefaction of air, with 130 atmospheres, 10 minutes.
Time for liquefaction of air, with 120 atmospheres, 16 minutes.
Horse-power employed, $3\frac{1}{2}$ indicated.
Liquid air produced, 1·2 litres per hour.
No preliminary refrigeration required.

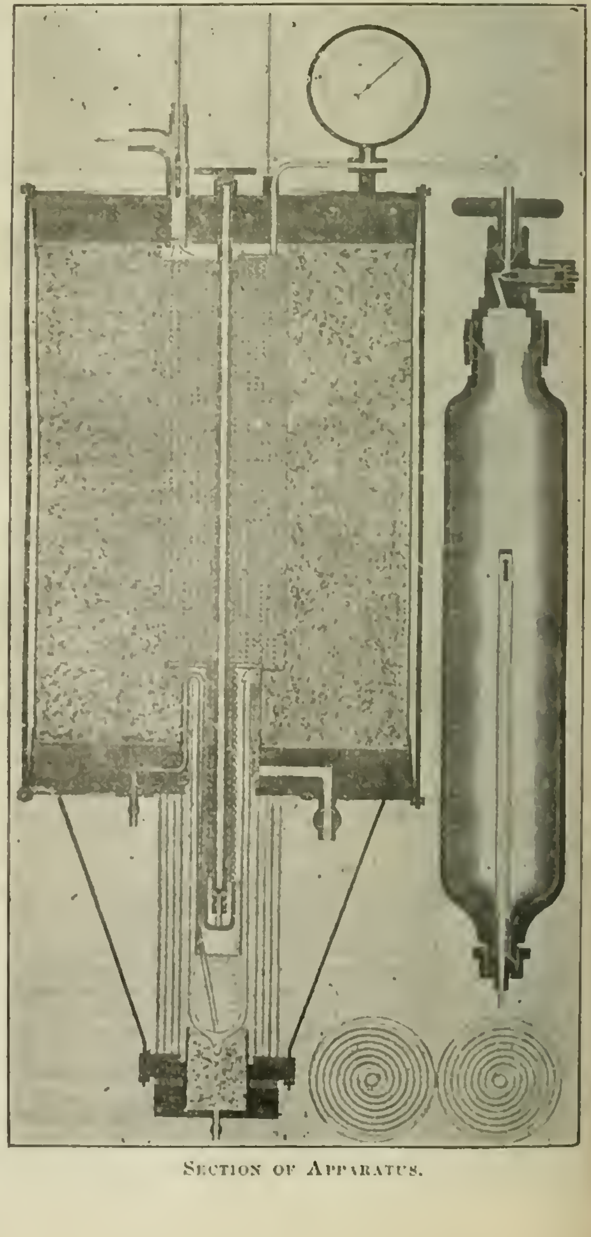
*II. From a cylinder:*

Preliminary cooling, cylinder of liquid carbonic acid.
Cylinder of oxygen, 120 atmospheres.
Liquid produced, up to 125 cubic centimetres.

In both cases only one valve and one gauge, which require very little attention.

The whole apparatus, including purifying cylinder, stand, and connections, easily carried by one man, and covering one square foot of floor.

In the illustration the inlet for the compressed air, and the outlet for the expanded air, are respectively by the small and the large pipe at the top of the interchanger. The expansion-valve is at the lower end of the coils, in the vacuum vessel, which acts as a receiver. The valve is controlled by a hand wheel at the top of the apparatus, acting through a hollow steel spindle. Liquid may be withdrawn by the syphon through the tap seen at the bottom of the interchanger on the left; or the receiver may be removed with its contents by unscrewing the vulcanite cap beneath the concentric glass cylinders, which, by enclosing layers of still air, give additional insulation. In the cylinder at the



SECTION OF APPARATUS.

side of the illustration the compressed air from the pump passes through caustic potash, which removes moisture and carbonic acid, and so enables the apparatus to produce a clear liquid. When the liquefier is worked with a cylinder instead of a compressor, the preliminary cooling by carbonic acid is done by passing in the cold vapour at the tap to the right beneath the interchanger, whence it passes directly to those parts of the coils which have to reach the lowest temperature. The plan of the alternate layers of coil in the interchanger is shown in the lower right hand corner.

The method of self-intensive refrigeration, already described, is most simply and clearly explained by a sketch like Fig. 4 on Sheet I. of diagrams. The compressed gas expanding through the constricted orifice at the left of the smaller tube is thereby cooled, and as it passes back in the larger tube cools the compressed gas which is next moving to the left in the smaller tube, so that the expansion is continually taking place from and to a lower temperature than before. It is all-important that the tubes be long enough and well enough arranged to make the interchange of temperatures as complete as possible; otherwise much of the cold developed will escape from the apparatus in the stream of expanded air. In the present apparatus, with a difference of temperatures between the top and bottom of the interchanger more than double the range between boiling and freezing water, the expanded air leaves less than 1° C. cooler than the compressed air enters. The attainment of this result, in an interchanger of very small bulk, is the explanation of the efficiency of the apparatus.

There are now so many methods of producing refrigeration, and so many varieties of each method, that the subject easily becomes confusing to those who have not made it a special study, and it will not be unprofitable to clear away by a careful examination some of the confusions that abound as to the process of self-intensive refrigeration. Their abundance has been much favoured by a growing abuse of the word "regeneration" and kindred terms.

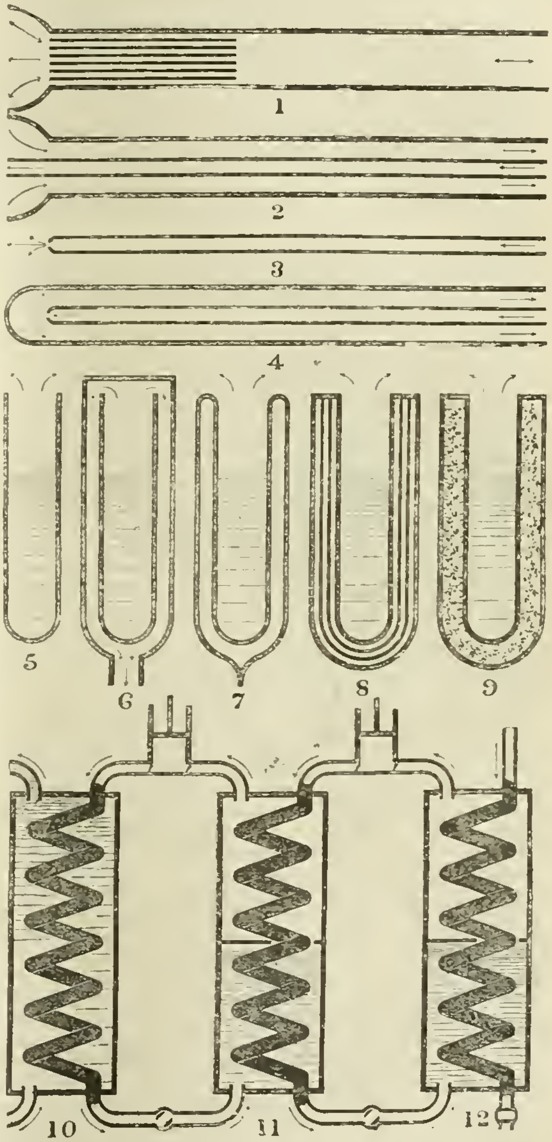
In apparatus containing a hot or cold substance, when it is desired to prevent heat from leaving the hot substance or from entering the cold substance, this may be done to a great extent by any of the familiar methods of insulation.

Insulation merely means making the walls of the enclosing vessel or chamber consist of material which affords small facilities for the passage of heat.

For work on a fairly large scale and at moderate cost there is no better insulator than a layer of wool, as in Fig. 9. Layers of still air, separated by successive laminæ, as in Fig. 8, also make a very good insulator, and have the advantage over wool that they can be rendered transparent by using laminæ of glass. More convenient still, as not only being transparent, but giving better insulation with small thickness, is the now familiar vacuum vessel, seen in Fig. 7. It has, however, the disadvantage that it cannot be employed conveniently in large sizes. Mons. Solvay, the well-known chemist, testifies, in communications to the Scientific Academy of Paris, reported in Comptes Rendus of 30th December 1895 and 13th January 1896, that the vacuum vessel was first applied to refrigerating work by Mons. Cailletet, the famous pioneer in low-temperature research, in whose laboratory he saw it in use in 1883. In this country it has since been improved in various ways, and its use made more widely known, by Professor Dewar, who, moreover, had used vacuum insulation in brass vessels long before, though Cailletet has established priority in the application to refrigeration, and in devising a form suitable for this work at intensely low temperatures.

A method of keeping heat out or keeping air in, quite distinct from insulation, is that of interception. This consists in catching the heat as it passes, and carrying it back in a current of fluid. In Fig. 6 the cold liquid in the central vessel, while boiling, gives off a cold vapour, which is made to pass over the outer surface of the inner vessel, where it intercepts, that is, it catches while in motion, and carries back, the heat which, entering from the outside, would otherwise penetrate to the cold liquid. Heat-interceptors of this kind have been used by Professor Dewar, De Vries, Wroblewski, Olszewski, and Professor Onnes, of Leiden. The latter experimenter so perfected this system that he was able to keep liquid oxygen with a surface still enough for observations of capillarity without the employment of vacuum-vessels. V. Communications from the Leiden University Laboratory of Physics, by Professor Onnes, No. 14, p. 26.

I



Another kind of heat-interceptor is the regenerator, shown in its simplest form in Fig. 1. This, it will be seen, answers to the excellent definition of a regenerator given by Professor Ewing in his book on the steam-engine, p. 59:—"A passage through which the working fluid can travel in either direction, whose walls have a very large capacity for heat, so that the amount alternately given to or taken from them by the working fluid causes no more than an insensible rise or fall in their temperature." Professor Wood, at p. 166 of his book on Thermo-dynamics, gives practically the same definition:—"Regenerators consist of a chamber well filled with thin plates of metal. During the flow from the cylinder the plates act as a refrigerator, by abstracting heat from the gas; but during the return of the gas they act in the opposite sense, and hence become regenerators." Peabody and Rankine agree exactly with these definitions, and both Rankine and Professor Ewing speak of the heat being "stored and restored" by the massive walls of the passages, a phrase which clearly brings out the etymological meaning of the word "regeneration."

This word, however, has of late been wrongly applied to heat-interceptors, of the kind shown in Fig 2; where the currents of fluid, instead of flowing alternately in opposite directions in one set of passages, flow constantly through two different sets of passages with unchanged directions in each; where the walls of the passages, instead of being massive with a large capacity for heat, must be light and of the lowest possible specific heat; where the heat, instead of being stored and restored by the passage walls, passes directly from one fluid current to the other: where the currents instead of passing only in one direction at a time, pass in opposite directions simultaneously; and where the temperature, instead of alternately rising and falling as in the regenerator, remains free from alternating changes at any point. Such interceptors are more correctly called interchangers, exchangers, or economisers.

Regenerators were used in Stirling's hot-air engine, Siemens' blast furnace, Kirk's refrigerator with wire-gauze or sheet metal, and Solvay's refrigerator with long massive piston and cylinder.

Interchangers were employed for liquids in the economiser of an ammonia absorption machine or a plant for producing carbonic acid; and for liquid and vapour in stills or condensers. But for gases in both currents only very rude attempts at interchange had until lately been made or suggested by Siemens, Laidlaw and Robertson, Solvay, and Windhausen, as well as in apparatus for using combustion gases to superheat steam or to heat air for combustion.

The scheme for a practically complete interchange between two constant currents of gas in one and the same cycle, flowing in opposite directions, one over the other, and the combination of this with expansion-cooling between the currents of compressed and expanded gas, constitute the invention of the self-intensive system of refrigeration embodied in the apparatus before you, of which the scheme is shown diagrammetically in Fig. 4, and the actual realisation in the double-size sectional drawing.

The first public discussion, by a learned Society in England, of the self intensive process, illustrated in Fig. 4, was opened in a paper read on the 19th December 1895, before the Chemical Society, by Professor Dewar, the present President of that Society, and reported in the Proceedings of the Society issued on the 14th of January 1896; a paper which, for the sake of brevity, may be referred to in what follows as the paper of December 1895. The process was on that occasion shown at work both in a very elementary and in a fairly complete form, and the author of the paper had previously shown it at the Royal Institution on the 2nd of the same month (*v.* Times, 3rd December 1895). The paper in question, however, does not represent the process by which the author had produced such striking results as one that contained any essential novelty, but on the contrary regards it as a mere improvement in details of methods well known before. The opinions of an authority who holds so distinguished a place in the scientific world must be received and considered with respect, and it will not be thought that the present author can examine with too much care such important opinions on a process of which he considers himself the original inventor, for which he claims essential novelty, and which he patented and communicated to persons holding important scientific positions long before there was any publication of the process, and without the least idea that anyone else was working on similar lines.

On the question of novelty the paper of December 1895 contains the following (p. 222). "In all continuously working circuits of liquid gases used in refrigerating apparatus the regenerative principle applied to cold first introduced by Siemens in 1857, and subsequently employed in the freezing machines of Kirk, Coleman, Solvay, Linde, and others, has been adopted. Quite independently, Professor Kamerlingh Onnes, of Leiden, has used the regenerative principle in the construction of the cooling circuits in his cryogenic laboratory (see paper by Dr. H. Kamerlingh Onnes, on the "Cryogenic laboratory at Leiden, and on the production of very low temperatures," Amsterdam Akademie, 1894). Apart, therefore, from important mechanical details, and the conduct of the general working, nothing new has been added by any investigator to the principles involved in the construction and use of low temperature apparatus since the year 1878."

The statement here quoted, that in all continuously working circuits of liquid gases used in refrigerating apparatus the regenerative principle applied to cold first introduced by Siemens in 1857 has been adopted, includes the apparatus used at the Royal Institution for liquefying air, which will be referred to shortly. It includes also thousands of ether, sulphur dioxide, ammonia, and carbonic acid machines, made on the De La Vergne system, the Linde system, the Hall system, and many others, not one of which uses any approximation to the principle which Siemens suggested. These commercial machines all have a condenser, or water-cooler, with the refrigerant inside the pipes, and water outside. That condenser corresponds to the water-cooler which Siemens proposed to have, with the refrigerant air inside the pipes and water outside. But Siemens proposed to have also an interchanger, with the compressed refrigerant inside the pipes, and the same refrigerant, expanded and partially heated, outside them. This is the feature referred to as the adoption of the regenerative principle, and it is entirely absent in any form from the vast majority of the continuously working refrigerators which, according to the paper of December 1895, all possess it The statement is also incorrect with regard to Kirk, Coleman, Solvay, and others; but it would take too much time to show this in detail; it will suffice for the present to say that none of them obtained a temperature below − 95° C., whereas the self-intensive process, in liquefying air, reaches − 191° C. There remain the methods which did, before December 1895, liquefy air by "continuously working circuits of liquid gases." One of these methods was that used by the author of the paper of December 1895, whose statement quoted above claims for it the possession of the regenerative principle. The other he refers to when he says:—"Quite independently Professor Kamerlingh Onnes, of Leiden, has used the regenerative principle in the construction of the cooling circuits in his cryogenic laboratory."

A careful examination will show how far either of these two methods contained the process of self-intensive refrigeration by expansion with counter-current interchange which was shown in December 1895, and which is described as using the regenerative principle.

First consider the application at the Royal Institution of the vapour-jacket, which has been mentioned in this connection, p. 222 (this Journal), of December 1895. Let Fig. 5 represent a vessel containing liquid ethylene, under atmospheric pressure. It boils at − 100° C., and continues at that temperature till all has boiled away. Vapour is all the time given off at the same constant temperature, the latent heat of vaporisation passing from outside through the walls of the vessel. Let Fig. 3 represent a tube containing air at freezing temperature and 100 atmospheres pressure, which expands through a small orifice at the left to atmospheric pressure. The free expansion causes cooling, and the expanded air issues constantly at − 29° C. as long as the operation continues. Now enclose the apparatus of Figs. 3 and 5 in outer envelopes as in 4 and 6, and compare the results in the two cases. In Fig. 4 the expanded air, as it returns in the outer passage, keeps cooling the compressed air in the inner tube to a lower point, and so the expanded air itself keeps reaching a lower temperature, until, if the interchange and the insultation be good enough, the temperature descends from − 29° to − 191°, and a portion of the air passing remains liquid at atmospheric pressure in the outer chamber. In Fig. 6 the cold ethylene vapour passing in the annular space over the vessel of liquid ethylene, intercepts much of the heat which would otherwise have passed through to the liquid, carrying it away at its escape, and so diminishing the quantity of ethylene vaporised. This is an economical arrangement, but the ethylene, though boiling more slowly, continues to boil at exactly the same temperature, − 100° C., while the pressure remains the same. In Fig. 4 there is self-intensification of cooling through 162° C.; in Fig. 6 there is none whatever. In Fig. 4 there is a pressure-drop of 99 atmospheres between the contents of the inner and outer tubes; in Fig. 6 there is no pressure-drop. In Fig. 4 the compressed gas becomes cooled by expansion; in Fig. 6 the liquid becomes no cooler on conversion into vapour; in one sense it contains actually more heat,—the latent heat of vaporisation. In Fig. 4 a continuous gradation of temperature between the cold end at the left and the warmer end at the right is established by interchange: in Fig. 6 the liquid is all of the same temperature: and so is the gas, as far as regeneration or interchange between it and the liquid is concerned. In Fig. 4 all the refrigerant starts as a gas, and finally part of it becomes liquid: in Fig. 6 all the refrigerant starts as liquid, and it ends by becoming gas. In fact, Fig. 4 represents the system of self-intensive refrigeration by gas-expansion with efficient counter-current interchange: Fig. 6 represents heat-interception by vapour-jacketting. Two processes could not well differ more widely, and though they have both been called regenerative,—a term to which it has been shown that neither of them is properly entitled,—it is hardly likely that the distinguished experimenters who used the vapour jacket at the Royal Institution would, after studying the foregoing analysis, consider it to be substantially the same thing as self-intensive refrigeration by expansion and interchange. The economy of liquid ethylene would be obtained equally well by insulation with vacuum, air layers, or wool, as in Figs. 7, 8, and 9, if these arrangements were in other ways suitable.

Next, let us examine the action of what Professor Onnes calls the regenerator in his air-liquefying apparatus. The system, as he describes it, is, with the exception of details, the same as that described as in use at the Royal Institution. Whether the action claimed as regenerative in the latter case is the same as that described by Professor Onnes, the public have no means of knowing; but in the absence of fuller information it must be taken to be so, since the nature of the cycle in the two systems is in its main features identical, and the paper of December 1895 accepts without criticism the account given by Professor Onnes of what constitutes regeneration.

The two systems may be examined together by means of the diagram in Figs. 10, 11, 12. Water, circulating in 10, cools and condenses carbonic acid, nitrous oxide, or methyl chloride in the coil, under a pressure maintained by the pump and throttle-valve. This liquid refrigerant, passing the valve, is in 11 partly vaporised, and thereby cooled under a lower pressure on its way back to the pump, itself cooling and condensing ethylene vapour under pressure in the coil of 11, which ethylene in its turn, at a lower pressure, and temperature in 12, cools and condenses compressed oxygen or air in the right-hand coil.

This is the familiar cascade method of refrigeration, initiated by Pictet, completed by Olszewski, and applied on a large scale by Professor Dewar. It may be apparently simplified towards the left by using a cylinder of liquid carbonic acid to replace Fig. 10, and the left-hand pump; but as the carbonic acid has to be compressed and cooled at a factory, it is necessary for the completeness of the cycle to represent the operation as in the diagram.

Consider the ethylene which circulates between Figs. 11 and 12. If in Fig. 12 it is kept by the pump and throttle-valve at a pressure equal to about 1 inch of mercury, its temperature is—130° C. The vaporisation of the liquid ethylene at this temperature condenses oxygen at the same temperature, and however long the operation is kept up, the liquid ethylene, at the same pressure, will not become a fraction of a degree cooler. There is clearly no self-intensification here. As the vapour comes off at the low temperature of the liquid, Professor Onnes takes advantage of its cold to give a preliminary cooling in the upper chamber of Fig. 12 to the compressed oxygen coming down. He does the same with the cold methyl-chloride vapour and warmer ethylene vapour in the upper chamber of Fig. 11. It is a good and economical arrangement, and he calls the upper chamber a regenerator. It has been shown above that the process at work here is more properly called interchange than regeneration. But apart from the proper use of words, is the process essentially identical with the self-intensification of Fig. 4? Firstly, then, in the upper chamber, as in the lower, there is no progressive descent of temperature. Whatever temperature is established at any point after the start remains constant, under constant conditions of pressure and circulation, throughout the process. There is no self-intensification at all. Other points of divergence from the process of Fig. 4 are:—the ethylene at lower temperature and pressure, after passing the throttle-valve, is not made to return over the pipe which contains the ethylene coming forward at higher temperature and pressure: the ethylene all passes the throttle-valve as a liquid, and all finally escapes as vapour: the oxygen inside the coil of 12 is a different substance from the ethylene outside, and belongs to a different circulation. In details therefore, as well as in general result, the process of Figs. 10, 11, 12, is quite different from the self-intensification process of Fig. 4; and it is unfortunate that a confusion of names which has been gradually growing should have made it possible to call them both regenerative, while neither of them possesses a regenerator in the correct sense of the word.

It is hoped that a clear line has now been drawn between the method of self-intensive refrigeration used in the apparatus before you, and the methods of low-temperature research known and published before the year 1895. Corroborations of this view may be found in editorials in "The Engineer" for 1895, 6th December, p. 555, and 27th December, p. 644, the paper which on October 4th, of the same year made the first publication in this country of the self-intensive process; and in Wiedemann's "Annalen der Physik und Chemie," Part 2, 1896, p. 332, in a note by Dr. Linde to his article. And Professor Dewar himself has since said at a meeting of the Society of Arts on the 2nd of March 1898, V. Journal, 11th March, p. 382, in referring to the self-intensive process as embodied in Dr. Linde's apparatus, that "he must confess that the practicability of such a mode of working had never struck him," during his "fourteen years' work" at "low temperatures."

An equally clear line could be drawn between the self-intensive method of liquefying air and the methods previously used for the moderate refrigeration required in cold stores and similar commercial work; but time for this examination is not available this evening.

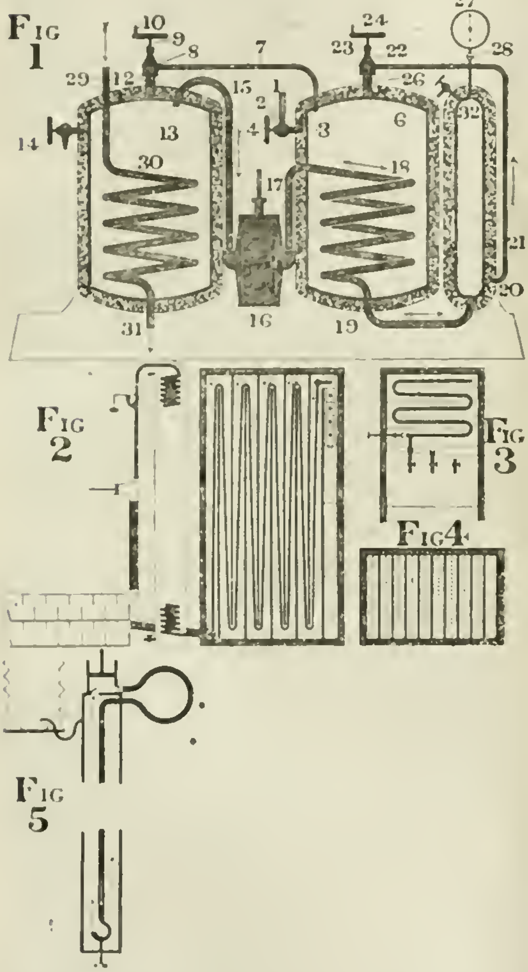
Another experimenter has, however, lately come to the front, whose work it is necessary to consider, since he has recently liquefied air in large quantities. This is Mr. C. E. Tripler, of New York. A letter signed "Arcuel" appeared in "Engineering" of the 22nd of April 1898, in which the writer, referring to the scheme described in Mr. Tripler's British Patent, No. 4210, 1893, says: "This is probably one of the best examples describing absolutely the same process and principle, as that which Dr. Hampson claims as his own, thus anticipating Dr. Hampson by at least 12 months."

Now it will appear from a glance at Fig. 4, and at the double-size sectional drawing, that an essential part of the process and principle of the self-intensive method is to have the compressed-air pipe surrounded right up to the expansion-point by the return stream of expanded air. The drawing with Mr. Tripler's patent, Sheet II., Fig. 1, shows that this is not the case in his scheme, wherein the compressed air pipe, through Nos. 19, 20, 21, 22, just in the coldest part, and where it is most necessary to expose it to the lowest temperature of the newly expanded air, is not exposed at all to the expanded air, which is not made, as in the self-intensive process, to return directly over the pipes of compressed air.

Secondly, Mr. Tripler's scheme has two condensers, but no interchanger. If, however, the condenser, No. 6, be regarded as intended for an interchanger, then the expanded gas enters at the wrong end to allow it to act as such. In an interchanger the compressed warm gas and the expanded cold gas enter at opposite ends, and there is a continuous gradation of temperatures between these opposite ends; while in Mr. Tripler's No. 6 the warmer compressed gas and the colder expanded gas both enter at the top, and the temperatures are approximately the same all through the chamber.

Thirdly, in Mr. Tripler's condenser, No. 6, the source of cold is the vaporisation of a substance at a certain rate from the state of liquid at a certain temperature, and the source of heat is the condensation of the same substance, at the same rate to the state of liquid at the same temperature. It is obvious that the heating and cooling will exactly neutralise each other if the expanded gas leaves at the same temperature as the compressed gas enters. But this cannot be. For, in the case where the apparatus is to condense air in the coil of condenser 13, at atmospheric pressure, and, therefore, at a temperature of − 191° C., the expanded refrigerant, having already taken up its latent heat of vaporisation, can only cool and condense the air in this coil by taking up specific heat; that is to say, it must enter the condenser 13 at some lower temperature, say − 200° C., and must, therefore, leave condenser 6 at the same temperature. Having been raised, while cooling and condensing air in the coil of condenser 13, to − 191° C., if it went straight back to condenser 6, it would thus be returning at a higher temperature than it had left it. But it has to pass on its way through the pump 16. Suppose it is compressed to five atmospheres from one: then its temperature will be raised by compression from − 191° C. to about − 150° C.: that is, it will return to condenser 6 about 50° C. hotter than it had left. The amount of heat represented by this difference of temperature, together with that which passes through the insulation, being in excess of what can be neutralised by the cold developed in condenser 6, the temperature in that condenser will constantly rise; in other words the apparatus is a heating machine, not a refrigerator. Nor can this difficulty be overcome by supposing that a cooler in connection with the pump 16 has been inadvertently omitted from the drawing. For, in the first place, it has been already shown that, even without passing through the pump, the refrigerant would return to condenser 6 warmer than it had left; and in the next place, the scheme of Mr. Tripler's design places the pump where no cooling agent could be used in connection with it. The pump receives the refrigerant at a temperature at least as low as − 191° C., and delivers it perhaps at − 150° C. It is plain that neither water nor any other available cooling agent could be used to bring down the temperature again from − 150° to − 200°; and if the pump produced a higher pressure, and, therefore, a higher temperature, that would mean more heat supplied to condenser 6.

II



Fourthly, Mr. Tripler himself, if we may accept his own account of his apparatus, does not expect it to act as a self-intensive refrigerator. On page 2 of his patent, at line 21, he thus describes the way to begin the operation:—"Liquid is allowed to expand through the valve 2 into the expansion cylinder or chamber 6." This liquid is the refrigerant which performs the cooling cycle, and the patent explains later, that it is able to condense at atmospheric pressure, in the coil of condenser 13, "any gases passed there through, boiling at a higher temperature than the circulating gas." The only refrigerant that he mentions in his patent is carbonic acid, and this gas, if the whole scheme were not founded on a fallacy, could, in accordance with his ideas, be used to condense at atmospheric pressure ammonia, methyl chloride, or sulphur di-oxide, but not nitrous oxide or ethylene, much less oxygen or air. To condense air with his apparatus at atmospheric pressure, it would be necessary, on Mr. Tripler's own authority, to begin by pouring into the apparatus liquid argon, nitrogen, hydrogen, or helium. It is probably not necessary to say more by way of demonstrating that this method is not quite "absolutely the same process and principle" as the self-intensive method of refrigeration. To this examination of Mr. Tripler's patent of 1893 it may be added that his production of large quantities of liquid air has not been announced until two years after the self-intensive process used in Germany and England had been published and fully illustrated; that his apparatus appears from the description, V. Scientific American, 2nd April 1898, to follow more closely the method used in England, having only two pressures; and that it produces almost exactly the same quantity of liquid per horse-power hour. It would not have been surprising if it had produced more, since it has thirteen times the power, and thermo-dynamic machines generally, including commercial refrigerators, are much more efficient on a large than on a small scale.

In another American paper, "The Electrical Engineer," for 14th April 1898, there is another account of Mr. Tripler's apparatus, in which it is stated that the expanded gas is returned over the compressed gas merely as an insulation against external heat, and that this arrangement has nothing to do with the production of the cold, which is obtained by expansion through a special valve of peculiar construction not made public. It is curious that, in spite of this, his invention is said to depend upon the process described in his old patents, including the one we have examined, in which there is, from beginning to end, no mention of a special valve. It is curious, too, that a process described as depending entirely on a special form of valve, and as making no use of interchange, should be said to be identical with the self-intensive process in which the interchanger is all-important, while the form of valve has no thermodynamic importance whatever.

Another American paper, the "Engineering News," of 14th April 1898, gives a sectional diagram of Mr. Tripler's interchanger, which fully corroborates the opinion expressed above, that it is a reproduction of the present author's apparatus on a larger scale, with such trifling differences of shape and material as the large scale makes practicable.

In conclusion, as a matter of interest in the historical aspect of the invention, it may be mentioned that drawings exist, dating back to nearly two years before the first publication of the process, shown at the time in confidence to competent witnesses of their authenticity, and clearly embodying all the parts of the apparatus described above. Two of the drawings are reproduced in Figs. 2, 3, 4, 5 of Sheet II., as shown before the end of 1894 to the writer of the recent letters in "Engineering," already referred to (see my letters to that paper for April 15 and 29, and May 6, 1898). Fig. 5 is the simplest possible representation of the process in an analytical diagram, and corresponds to Fig. 4 in Sheet I., but in the former are shown also the pump, the water-cooler in the form of a loop to the right, and the supplementary air-supply. Figs. 2, 3, 4 are a provisional diagram submitted as a basis for consideration of the available power and appliances. Both the analytical diagram and the plan diagram display the combination of all the characteristic features of the self-intensive process shown to the meeting; the long tube of compressed gas, the cooling by expansion through a throttle-valve, and the direct return of all the expanded gas over the compressed gas, with careful provision for good interchange of temperatures.

### DISCUSSION.

The CHAIRMAN, in inviting discussion—observed that although a very short time ago the production of temperatures, such as were involved in the author's process, would have been regarded as of little or no practical importance, they were already employed on the continent for the preparation of narcotics, and were about to be utilised in the production of chlorine by the Deacon-Hurter process. For this reason he had been anxious to bring before the society an invention made by an Englishman, who claimed that his apparatus was superior to and his patent of an earlier date than Linde's. He hoped that in discussing it all feelings of a personal character might be excluded, and that they would deal with it from its purely scientific and utilitarian standpoint. (Some personal points had been introduced during the reading of the paper that were not contained in the printed proof.)

Prof. JAMES DEWAR said that perhaps it would be fitting that he should at once make some remarks on the paper. He had listened to it with great interest, and agreed with the chairman that it was desirable in discussing it that all feelings of a personal nature should be as far as possible excluded. He desired to express his deep regret that Dr. Hampson had not applied to him instead of to his assistant for permission to see the apparatus employed at the Royal Institution. Had he done so, he (Professor Dewar) would have at once made it clear to him why he must refuse that permission, as he had already refused it to many gentlemen of distinction interested in the subject, not from any unwillingness to lay his plans or discoveries before them, but to prevent misconceptions which were certain to arise from such conferences. For the same reason he had himself refused to inspect documents and plans which M. Pictet had voluntarily brought to him. He could only add his assurance that he had not received or utilised in his work on this subject any communications or knowledge derived directly or indirectly from Dr. Hampson. With regard to the subject before the meeting, he was not interested in any particular form of apparatus for producing low temperatures. His one object was to apply those temperatures to research in electricity, magnetism, and other departments of science. That being so, he would discuss the matter from a purely general standpoint, and, he hoped, in a spirit perfectly just to all concerned. In the first place, Dr. Hampson was incorrect in associating any other names with his own (Professor Dewar's) as users of the interceptor which he had described with reference to liquid ethylene. That was one of the earliest additions made by himself to the work of Wroblewski and Olszewski in 1883, and was novel at the time—long before vacuum vessels had been applied to that purpose, though they had been used for an entirely different object. It would, therefore, be absurd to imagine that, when he described in his paper of December 1895 an interceptor of the vapour coming from a liquid, he was dealing with anything but the known fact that one could utilise the cold produced by the expansion of a gas by passing it back again over what he called a regenerator, and in that way produce more economical results. So far as Dr. Siemens's work was concerned, his views were clearly set forth by himself in a discussion on Coleman's paper on "Air Refrigerating Machinery and its Applications," read before the Institution of Civil Engineers, 14th February 1882. Mr. Coleman said in that paper: "Any machine which is worked through the medium of a readily condensable vapour, such as ammonia, ether, methylic ether, or sulphurous acid, has its action limited by the boiling point of the volatile liquid; it is therefore impossible with such machines to get so large a range of cooling in one operation as can be accomplished by air. The low temperatures which Pictet required for the liquefaction of oxygen and hydrogen were obtained in stages, first by the ebullition of liquid sulphurous anhydride in vacuo, producing sufficient cold to liquefy carbonic acid gas at a pressure of four atmospheres, and then in taking advantage of the still greater cold produced by the ebullition of the liquid carbonic acid gas *in vacuo*. There is, however, no reason to suppose that the same or much lower temperatures could not be obtained by the compression and expansion of air in a single operation." In the discussion that followed Dr. Siemens described what he meant by his interchanger. His attention had been called to the subject by having to make a report on the first air-cooling machine of American origin, patented by Dr. Gorrie. Dr. Siemens said:—"The author had, perhaps, hardly given him credit for the labour which he had spent on the subject. He not only devised the temperature interchangers, which was the most essential feature in the whole process, and without which no efficient results could be obtained, but he had fully recognised the antagonistic effect of aqueous vapour in the air dealt with. The loss of effect "by throttled passages" he had given at 10.08 HP. That required some explanation. Dr. Gorrie evidently wished to avail himself of the expansive force of the compressed and cooled gases before they were discharged again; but he did not conceive that the whole effect of the machine depended upon the heat (utilised negatively) in that final expansion; and in order to be sure, as he thought, doling out as it were the compressed air to the expanding cylinder, he introduced a throttle-valve into the compressed and cooled air; and instructions were given to wire-draw that valve as much as possible on the supposition that after all the safe plan of gaining refrigeration would be to expand air through the throttle-valve, that was, through a contracted orifice. Dr. Siemens showed that contraction was the worst thing that could happen, that by merely expanding air without exacting work from it, no depression of temperature would ensue." Further on he said, "That loss in some of the earlier attempts was fully equal to the whole useful effects that could be expected. But the greatest loss of all was incurred in allowing the cold air to leave the apparatus at the minimum temperature. He suggested that a current exchanger should be supplied; that the cold air after it had done its negative work, so to speak, should pass up through spaces surrounding a number of vertical tubes, and that the cold compressed air should pass through the tubes themselves in the opposite direction and exchange temperatures." That paper, then, represented the state of knowledge on the subject in 1882. In 1885 Coleman returned to the subject after Wroblewski and Olszewski had succeeded in producing liquid air, and in a Friday evening discourse at the Royal Institution said, "These machines, in an ordinary way, supply streams of atmospheric air cooled to about 80 degrees below zero F. [– 63 degrees C.]; but, by certain modifications, they can be adjusted to deliver the air cooled much lower, and, in point of fact, to as low temperatures as have yet been produced in physical researches." In the same year, 1885, came Mr. Solvay's work on this subject; and before referring to that he thought it only right to say that Mr. Solvay, as a very old friend of his, had enjoyed his confidence, and been privately shown his work and apparatus connected with this subject for many years. During the whole of that time Mr. Solvay had never suggested to him that he himself had made any researches in the subject until his communication appeared in the Comptes Rendus in 1895; nor had he mentioned that he had taken a patent for producing low temperatures some years before. He (Professor Dewar) was entirely ignorant of Dr. Solvay's work on this subject down to 1895. It appeared, however, that on 6th November 1885 Solvay had filed a patent for "a new or improved apparatus for and process for producing, applying, and keeping up extreme temperatures," and his claims in that patent included one for this very regenerative principle referred to by Dr. Hampson. Solvay claimed that his apparatus was applicable not only for producing extremely low temperatures but extremely high ones. In the former case he claimed to effect his object as follows: "I produce low temperatures by making a series of successive expansions of gas by mechanical power, and by forcing the cold resulting from each expansion, and itself pervading the expanded gas to concentrate itself on leaving the apparatus, upon a special body acting as recuperator, such cold being subsequently continued also systematically and in an opposite direction by the incoming gas intended for the succeeding expansion." In the same patent Dr. Solvay made the following statement: "I employ the cold produced in this expansion department for all known processes, but particularly to liquefy any gas whatsoever and more especially atmospheric air," and he then proceeded to explain that "the process described and the results obtained differ from the usual mode of compression and expansion, from the form or kind of compressions and expansion apparatuses in use; from the form or kind of recuperating linings in use which may be simple tubes or metallic wire gauze directly employed on or in the cold producing pump, the latter being thus devoid of a sheath or an appendix to the piston." It was clear from this that Solvay used what Siemens had used before him, viz., the current outside the tube for cooling the current inside the tube. He was evidently under the impression that he had made this great discovery and did not know of Dr. Siemen's patent, which, in fact, was never completed. In his patent Solvay claimed "the application of the above principle, whatever might be the kind of apparatus employed, as also the materials composing the same and the nature of the gas employed for all purposes, and specially for the fusion of metals and of minerals for producing chemical compositions, or effecting combinations between substances of difficult fusibility for the cooling or liquefaction of gas and the like." It was clear, then, that the principle of such apparatus was known in 1885; but, as he had said, in reference to Dr. Linde's patent, though one could patent a particular apparatus for performing the work, one could not patent the principle involved. The next point he had to touch upon was to clear up a misapprehension which Dr. Hampson had conveyed by one of his quotations that he (Prof. Dewar) had expressed his opinion on this subject at one time in a contradictory sense to that which he had expressed at another. In reference to his paper of 1895 he was in a peculiarly happy position of being attacked by all three of the other inventors concerned, first, Dr. Solvay having seen his experiments, privately said substantially in the Comptes Rendus, "What I saw there was my invention; if I had only used vacuum vessels I should have done the whole thing in 1885." Then they had the critique of Dr. Linde who said, "This is exactly my invention," and now Dr. Hampson was taking the same line. He desired to be perfectly frank in the matter and do all in his power to clear it up. His paper in 1895 referred to a set of experiments quite distinct from this subject. It did not concern the devising of another form of liquid air machine, for which he had already a thoroughly satisfactory apparatus, but to some experiments which he was then conducting with a view to determining the conditions which he would have to deal with in obtaining liquid hydrogen. Referring to the criticism then expressed that he was using the Linde apparatus, he had said distinctly, and he repeated it now, that in his judgment that apparatus was not thoroughly satisfactory for the purpose, both with regard to the yield obtained and the method of obtaining it. In the paper he said: "Coleman pointed out long ago that his air machine could be adapted to deliver air at as low a temperature as had yet been produced in physical research. Both Solvay and Linde had taken patents for the production of liquid air by the application of cold regeneration, but the latter has the credit of having succeeded in constructing an industrial apparatus (the plans of which are not yet published), that is, lowered in temperature to −140°, or to the critical point of air in about 15 hours, and from which liquid air containing 70 per cent. oxygen is collected after that time." Obviously had he known of Dr. Hampson's method at the time he would have referred to it also. In the discussion which followed he had pointed out that "such a process cannot replace the use of 'cooling agents,' when considerable quantities of liquid air have to be produced in a short period of time as in ordinary laboratory work. It is a mistake to attribute to Linde the idea of using the 'cumulative withdrawal of heat, for the first time in his apparatus; but he has succeeded in making a workable industrial machine, and that is a very important step." In the same paper, speaking of his own experiments, he had said, "The efficiency is small, not exceeding the liquefaction of 2 to 5 per cent. of the air passing, but it is a quick method of reaching low temperatures, and easy to use for cooling tubes and collecting a few hundred c.c. of liquid air, especially if the compressed air is delivered at the temperature of −79° before expansion." He had practically repeated the same statement at the Society of Arts meeting on 11th March 1898, at which Dr. F. Linde was present, when, in proposing a vote of thanks to Prof. Ewing he said: "There are many points connected with this work of Dr. Linde's on which I might enlarge if time permitted. The mechanical ingenuity and knowledge of thermodynamics which Dr. Linde has displayed is worthy of all praise. After some 14 years' work I ought to know something about low temperatures, but I must confess that the practicability of such a mode of working has never struck me." He claimed that his own results under the best circumstances gave an efficiency of from 2 to 5 per cent., which he considered a very small efficiency. It turned out that by working with a larger apparatus Linde obtained exactly 5 per cent., viz., ·9 litre per 3 H.P. hour. The yield of Dr. Linde's apparatus was therefore no greater than that obtained by himself in 1885. He had said at that time that he did not consider such results sufficiently efficient, and he still adhered to that opinion when he compared their results with those he produced by the Royal Institution installation. However that might be, it did not affect his view that the principle of regeneration as applied to the production of cold was well-known at that time. What those gentlemen had since done was to construct an apparatus—for which they deserved credit—for getting practical results by that method of working. Dr. Hampson had referred to the paper of Dr. Onnes. He regarded Dr. Onnes as a fair critic, and he had said: "In previous constructions of my boiling glass, I had to supply coils of greater cooling surface immersed in the gas flowing away for the sake of making a liquid jet with gas cooled to little below the critical temperature or even to temperatures in the neighbourhood of the critical. It proved more effective for the construction of the permanent liquid oxygen bath to reckon upon the oxygen being intensely cooled before it is transported to the boiling glass. The spiral was therefore shortened to only such length as seemed necessary to catch the liquid blown away, and to utilise the evaporation of this liquid to cool the adducing coil. Dewar, on the contrary, has not been discouraged by the less economical employment of gas, and armed with his vacuum vessel that leaves the returning gas wholly disposable to cool the coming gas, has directed his attention exclusively to the lowering of the temperature." It was clear from this quotation that Dr. Onnes was aware of the principle involved in this method of working, and if he had been using ethylene his words described exactly what would happen. Dr. Hampson, however, was not satisfied with the use of the term interchanger, but claimed the words regeneration and self-intensification. He would point out, however, that the title of his own invention was "improvements relating to the refrigeration of gases." That was to say, he did not claim to originate this method, but only to improve it. That being so, his provisional specification naturally contained no description of any method of working, but only the following succinct statement: "With this object all the expanded gas surrounds the pipe or pipes of compressed gas through all their length from the point of expansion to the point of normal temperature, and this length of pipe is sufficient to allow of the fullest possible interchange of temperatures between the compressed and expanded gas." From the statement contained in his complete specification it was to be inferred that the Patent Office declined to allow the title first given to the provisional specification unless with it Dr. Hampson supplied a description of the specific apparatus which he claimed to have invented. The object of Solvay was to cool nothing but the air, and that he effected by putting his interchanger in the cylinder itself. When, therefore, he (Prof. Dewar) was told that his regenerative interchange of temperature was a false application of the principle, he could at least reply that he had committed the mistake in good company, viz., with Dr. Hampson himself, who, in his complete specification, said: "I am well aware that the principle of regeneratively interchanging temperatures between currents of gases has long been familiar in multitudes of devices for evaporating and condensing, heating and cooling, but never before has refrigerating apparatus been designed with a view to the transfer (unavoidable leakage excepted) of all the cold of the expanded gas to the compressed gas which is going to be expanded. On the contrary, where heretofore the cold expanded gas has been employed at all to cool the compressed gas, it has been in conjunction with the use also of the cold expanded gas to cool objects external to the apparatus." He must confess himself a little surprised at Dr. Hampson's reference to the use of vacuum vessels, and could not but consider that reference a little unkind, even a little ungenerous. One did not get so much credit in these days for the little one could do towards the science of our time that a fellow worker should be willing to deprive him of it on mere hearsay evidence. Dr. Hampson had no ground, other than hearsay evidence, for stating that Cailletet was the first to use those vacuum vessels. Cailletet himself had never put forth or accentuated any such proposition when discussing Solvay's paper before the Academy. He had himself requested Solvay to ask Cailletet whether he had described the use of such vessels for obtaining low temperatures, and if so, in what paper, and the result was an admission that there was no publication of any kind whatever. Then he had referred to Prof. Onnes, and he also knew nothing about these vessels being used by Cailletet, or any other scientist. It had been stated that they had been seen in connection with researches on marsh gas, but so far he had not been able to discover anything about those researches. On the other hand, Prof. Onnes had himself said: "In the same degree as it becomes of more importance to effectuate adiabatic processes at very low temperatures, the importance of the vacuum vessels of Dewar will increase. It seems to me that they are the most important additions since 1883 to the appliances for low temperature research." He was perfectly satisfied with that statement, and it reconciled him to the fact that others who were quite willing to use his vessels, and, indeed, could not get on without them, gave him no credit whatever for introducing them. He was a little surprised at the ingenious incumbrance which Dr. Hampson had added to his vacuum vessels. It certainly struck him as incongruous to cover a vacuum vessel with several concentric vessels of glass, and he would like to hear from the author what improvement he expected to derive from such an addition. If the vacuum vessel was good the heat transference was practically limited to 5 per cent. of what it would have been without them, and he failed to see what benefit could result from covering it on the outside with this monstrosity. Dr. Linde had done nothing of the kind, but used the vacuum vessel pure and simple. He was willing to give all credit to Dr. Hampson, Dr. Linde, and anyone who effected improvements in these investigations. All he asked was that they should not exaggerate their claims and seek to block the way to other people who were working in the same direction. Dr. Hampson did not appear to realise that anybody else could be working in the same path and utilising the same ideas. It was quite clear, however, from the facts before them that that was precisely the state of affairs in the present case.

The Chairman said that Prof. Dewar had anticipated most of his remarks. He wished, however, to point out that Coleman said that "The statement that is met with in some text books, that no cold can be produced by expanding air without mechanical work being performed, is not strictly correct, as one fraction of a given volume of air can be cooled by blowing the other fraction into the atmosphere; but all such methods of producing cold are, however, from an engineering point of view, wasteful," and not that the principle of expanding air without doing mechanical work was incapable of being utilised. Interchangers or regenerators, by whatever name one might call them, were economisers intended to restitute to one part of a circuit or system the heat or cold produced at another. This principle could hardly be called a novelty, but it had probably nowhere been so perfectly applied as in Dr. Hampson's and Dr. Linde's apparatus.

Dr. W. Hampson, in reply, said that Prof. Dewar, in his criticism of the paper had made an analysis of the patents and the proposals of Siemens, Coleman, and Solvay, and by piecing together portions from one with portions from others had succeeded in proving—if such a result could be called proof—that the principle of the self-intensive process, or as Prof. Dewar expressed it, the regenerative principle, was well-known before he had introduced it. Acting in that way, it would not be very difficult to prove any novelty old. If one searched through the Patent Office records and picked out portions from various specifications and put them together, one could build up or destroy almost any invention. But if one carefully considered each proposal in detail, he would see that the self-intensive principle of combining the refrigeration of gas by its expansion through a small orifice, the return of that expanded gas directly over the tube through which it passed, the bringing down of the compressed gas and the efficient interchange distinctly provided for in the apparatus, was a combination not to be found in the work of Siemens, of Coleman, or of Solvay, nor in any descriptions of diagrams previous to those which he had shown. In the case of Siemens it would hardly be believed that the quotation from Siemens, which he was about to read, was a correct quotation, since he had been so often put forward as the originator of the application of the principle of regeneration or, as he preferred to call it, of interchange. He would, however, refer the audience to Siemens' provisional specification, and to the more elaborate account which he gave in his paper read before the Institution of Civil Engineers. In the first place it would be found that he made no mention of expansion through a nozzle; in fact he never thought of working with anything else but a piston and cylinder. He made no definite provision for thorough interchange. His very elementary diagram showed that he had not grasped the difficulties of the problem of causing two gases to interchange their temperatures efficiently. Then, bearing in mind the temperatures produced by him, it could not be said that he had devised an apparatus for producing low temperatures in the modern sense of the phrase. He actually described his air as bubbling through a liquid solution of brine. Could anyone believe that an arrangement which involved an expanded air bubbling through brine would end in producing the liquefaction of air? Siemens also provided for its passing through large chambers, applying it to the cooling of sick-rooms, &c. Let them imagine the effect upon patients of passing air over them at such a temperature as they now knew to be necessary for producing liquefaction, or what efficiency the cold air would possess after being applied to such an use. In fact, Siemens was not contemplating low temperatures in the modern sense, and described no apparatus which would realise them, and cut himself off from such method entirely by saying "that by merely expanding air without exacting work from it no depression of temperature would ensue." He found it difficult to imagine a man using such apparatus being put forward as the originator of the principle which he had described in his paper that evening. Next, let them them examine the work of Coleman in detail. He, again, never made an apparatus except for similar commercial purposes to those of Siemens. But he did suggest two methods by which air could be liquefied, methods to one or both of which Prof. Dewar had referred. He would refer to them also, and would endeavour to point out their bearing more clearly. Coleman reduced the temperature of air by a refrigerating machine, which he described as working by means of a cylinder and piston. He expected by a single expansion to get the air cooled as low as −94°C. At this temperature carbonic acid gas would liquefy, and

Coleman claimed that the liquefied gases could be used to bring about a further reduction of temperature which would eventually be the means of liquefying air. This was the cascade system pure and simple. Was he to understand that that was what Prof. Dewar referred to as an anticipation of his (Dr. Hampson's) invention? Coleman also made another suggestion, viz., that if a machine could be constructed for delivering the air cooled to below the critical temperature of oxygen (− 120 C.), that gas could then be liquefied by compressing it to 120 atmospheres in a coil surrounded by air so cooled in a cylinder while driving a piston. Was that the method which Prof. Dewar suggested was an anticipation of his invention? If so he submitted that there was nothing of the nature of the self-intensive process in it. With reference to the Chairman's remarks about his quotation from Coleman's paper, he was quoting from Coleman's own words when he said in reference to Joule's first series of experiments, "from this it follows that no satisfactory apparatus can be devised by simply allowing compressed air to expand behind a loaded valve." Dr. Messel's quotation was from another passage, upon which he feared a misunderstanding had arisen. Coleman said that air passing out might cool not the air which had passed out, but that which remaining behind in the cylinder had by its expansion driven out the preceding portion in much the same way as it might drive a piston which was still inside the cylinder. But in neither case was it possible to found the self-intensive system on that of Coleman. Then the system of Solvay had been referred to, and as Prof. Dewar described it, it was at first a regenerative system since the air went back and forth in alternate currents. But in all Solvay's work there was no suggestion of the air expanding through a contracted orifice and then passing over the coil. Solvay had not grasped the importance and difficulty of the problem of thorough interchange, and he carried out his system in such a way that no such effects as were before the meeting could be produced, and he did not produce them.

The Chairman observed that he had not intended to go into these matters in detail, but as Dr. Hampson said that Solvay had not grasped the idea of interchanging, he thought he ought to quote what Solvay actually said. "If a gas somewhat uncondensable, such as hydrogen, for instance, be used for the dilatation any other more condensable gas can be liquefied by causing same to circulate outside the recuperating part of the sheath and quite close thereto; with that view I put round the sheath a second sheath a little larger, forming a round space with the former, along the whole length of which the gas to be condensed must run in an opposite direction to that of the discharge of the expanded gas, that is to say from top to bottom, the gas to be condensed thus being continuously in contact with a colder surface."

Dr. Hampson, continuing, said he did not think that statement, referring to a plan for cooling one gas by means of another, could be put forward as covering the principle of self-intensive refrigeration, which was the foundation of his system. Solvay had neither described nor drawn an interchanger competent to do the work, nor had he even shown that he understood the problem to be worked out. By picking out and putting together these various pieces, Prof. Dewar claimed to prove that the principle was well-known to Solvay and others. If that was the case how came it that such distinguished men as Pictet, Cailletet, Wroblewski, Witkowski, Olszewski, Dewar, and Onnes, who were so anxious to outstrip each other, that any one of them, would almost have given his little finger to be first in the field, should have left this principle altogether on one side? The method was not to be found in any literature or patent published previously to October and December 1895, when for the first time it was seen at work in England, and no system in which the two materials were different inside and outside the pipes could be truly considered an anticipation of it. Prof. Dewar used the phrase liquid ethylene. Was he to understand that there was in the Royal Institution a case in which ethylene was made to flow back over the coil as in the apparatus described by himself (*pointing to diagram on wall representing use of ethylene*).

Prof. Dewar: "No, sir, there is no such apparatus in the Royal Institution laboratory as that which you have pointed to."

Dr. Hampson, continuing, said, if that was the case there was no work in the Royal Institution which was in any sense an anticipation of his own.

Prof. Dewar: "I have said exactly the opposite; that the ethylene gas passes through a nozzle above its critical point, giving thereby liquid by the cooling of the gas over the pipe, and I have read you the passage from Onnes in which he says he has used this not from a jet containing liquid only, but for the purpose of cooling the adducing coil."

Dr. Hampson: "But his description is that one gas circulates in one system of coils, another gas circulating in another system."

Prof. Dewar: "I am not referring to that, and neither is Prof. Onnes."

Dr. Hampson: "It would be an advantage if the passage in which Onnes describes that use of a gas should be brought to public notice."

Prof. Dewar: "I have read it to you to-night."

Dr. Hampson: "I repeat that the paper as I have read it describes two gases not the same."

Prof. Dewar: "Then I can only say that you have read it wrongly."

Dr. Hampson, continuing his reply to the discussion, said that Prof. Dewar had referred to the title of his patent, and argued from the fact that it was described as an "improvement" that he claimed nothing novel in the apparatus. It was not he who was responsible for the title, but the Patent Office, and Prof. Dewar must know that there were thousands of such patents which were all described as improvements in such cases, though they were genuine novelties. Prof. Dewar also made a point against him by quoting the word "regeneratively" out of his patent. He would remind Prof. Dewar that a patent was not a document in which a person set forth all his ideas for the reform of inaccurate expressions; and the only course for him in drawing his specification was to use the words in the sense in which he found them commonly used, though in his paper he felt free to make a protest against what he considered a loose application of such terms. Prof. Dewar had said that he had been unkind in his remarks. He could only say that such was not his intention, and he would be glad there and then to bear testimony to the very great value of Prof. Dewar's vacuum vessels, and to the very large part which Prof. Dewar had taken in improving, developing, and making them popular. But it was a primary principle of science that those who first brought out an invention should have the credit of it when published.

The Chairman: "But Cailletet has never brought out nor published the invention you attribute to him."

Dr. Hampson, continuing, said: The statement was published in 1895-6 by Solvay, and if on subsequent occasions Prof. Dewar in introducing them had referred to the previous use by another scientific man he would not have found other scientific men so keen to point out that Cailletet was the first to use them. It was wrong to say that the statement attributed to Cailletet was founded only on hearsay evidence. Prof. Dewar had laughed at his vessels, and it was a cheap laugh. He could assure him, however, that there were many ways in which the details which provoked Prof. Dewar's amusement were found convenient and useful. Further than that, even if the loss in using a simple vacuum was small, it was a thing to guard against, and he could show Prof. Dewar the good effect of these sections by taking away an external vessel, when it would be found that the surface was so cold that moisture from the air would freeze on it. Vacuum vessels, like other apparatus, were human institutions, and not always perfect. As according to Prof. Dewar's own admission they deteriorated with age, it was an advantage to have an auxiliary means of diminishing the disadvantages resulting from those difficulties. He did not leave a coating of mercury as described by Prof. Dewar, because he wanted to see the liquid all the way down. He thought that was a satisfactory defence of his "monstrosity." He would conclude by expressing his regret if, in making what he considered a simple and direct statement of historic facts, he had done wrong, and especially if it had prevented a fuller discussion on other parts of the subject more germane to its issues.

Prof. Dewar: "I am sure that all present will agree with me in desiring to avoid any personalities, but I had hoped that the author would have expressed his acceptance of the statement made by myself, that no information bearing on his invention was either conveyed to me or utilised by me. That is the point which I have been anxious to impress on the author. It has been stated indirectly that such a communication was deliberately made to me and utilised by me a year and a half afterwards. I consider that after the direct denial that I have given to that suggestion the author should either substantiate or withdraw it."

Dr. Hampson: "In the first place, I will express my regret that I have omitted to refer to that matter. Prof. Dewar will do me the justice to say that I have nowhere published any statement that he had made use of anything I had communicated or of what I have invented. I have, therefore, nothing to withdraw, since I have nowhere suggested that a communication had been passed on to him, and further than that I would reply that a great part of what I have had to say is due to the necessity for pointing out the difference between the method I propose and the methods previously in use at the Royal Institution, and it is therefore necessary for me to bring the distinction out clearly. But I am not to be understood as saying that my proposal was passed on to Prof. Dewar, but am only bringing out the novelty of my apparatus and the distinction between mine and that in use at the Royal Institution."

Prof. Dewar: "That explanation I accept as quite fair and satisfactory to me, and I think Dr. Hampson will now have no difficulty in withdrawing what I read as having been said in a letter to "Engineering," and which I think I am entitled to feel is a reflection upon me: "More than two years ago Prof. Dewar showed in operation at the Chemical Society a process which is practically identical with mine. . . . . . It is also true that $12\frac{1}{2}$ months before that demonstration I communicated my process, then an entire novelty of my own invention, to Prof. Dewar's assistant, Mr. R. N. Lennox." That seems to me a roundabout way of saying that the information was utilised either by the one gentleman or the other. As Dr. Hampson withdraws any intention of charging me with using his apparatus I hope he will now as publicly withdraw the imputation which I have read."

Dr. Hampson: "That was written in reply to a previous anonymous letter quoting an anticipation of my work. In reply to that, I cited a much earlier communication by me to the actual writer of the letter, whom I wished to unmask under his real name, and the statement I made is strictly correct, and must not be read except in the literal meaning of the words. I make no statement in which Prof. Dewar is involved. I only say that this communication was made in the institution where the gentleman named therein is working. I make no insinuations, but simply defend myself against the statement that my apparatus had been anticipated at the Royal Institution."

Prof. Dewar: "Then I hope Dr. Hampson does not imagine that I am the anonymous gentleman who writes to "Engineering." I had no knowledge of Dr. Hampson till now or of his ever having been at the Royal Institution."

The Chairman said there remained one point upon which he wished to touch. In his first communication to the French Academy Comptes Rendus, Vol. 121, 1895, Mr. Solvay undoubtedly stated that the use of vacuum vessels to minimise losses of heat was due to Mr. Louis Cailletet, but in the next volume he makes a correction to the effect that when making the above statement he had not been aware that Cailletet never published that he had used such vessels, nor that Prof. Dewar had used them 23 years before in his calorimetrical experiments. It followed clearly from this that Solvay had been under a misapprehension, and it was more than doubtful if Cailletet ever had used vacuum vessels before Prof. Dewar had introduced them. Private information in his possession confirmed this statement, and he would endeavour to obtain authority to put it before Dr. Hampson. To Prof. Dewar, and to nobody else, belonged the credit of having introduced vacuum vessels to cryogenic science: Dr. Hampson, himself so sensitive as to his own priority, should have been the first to acknowledge this fact. It gave him great pleasure, as he was sure it would to members of the Society, to compliment Dr. Hampson on the excellent apparatus he had constructed, and to thank him for his paper, and for the experiments he had shown that evening. Low temperatures had before now been produced by successive expansions of gas by mechanical means in conjunction with interchangers, but he did not know of any published results nor of anyone who had been bold enough to utilise the extremely small degree of lowering of temperature produced by adiabatic expansion of a gas in conjunction with interchangers to produce this effect before Dr. Hampson and Dr. Linde, and it was their great merit to have combined well-known physical principles and produced a workable apparatus. He regretted that Dr. Hampson had introduced matter of a personal character not contained in his typewritten proof, but now that both sides had been heard he hoped that all controversy of a personal character raging round low temperature research would cease; the scientific world had had more than enough of it.

# Manchester Section.

*Meeting held on Friday, February 4th,* 1898.

MR. GEORGE E. DAVIS IN THE CHAIR.

## THE TREATMENT AND DISPOSAL OF TOWN SEWAGE.

BY DR. J. GROSSMANN.

In considering questions relating to sewage, it is necessary to give sufficient attention to its origin and its components. Thus, sewage from purely agricultural districts will consist of the refuse from domestic sources, from stables of cattle, horses, and other animals, plus a certain amount of water from the general water supply and rainwater. In towns and manufacturing districts we must add to these substances the refuse and waste liquors from factories of all kinds. It is therefore evident that a mixture of such different components as town sewage is, cannot be alike in any two places.

The quantity of refuse from domestic sources determines the amount of sewage proper; the quantity and quality of factory refuse its character; whilst the quantity of water from general water supply and rain determines the relative strength of the sewage.

Neither of these components is a constant factor; each varies. Domestic sewage varies from hour to hour and from day to day; for instance, there is a regular increase in the quantity of soap in the Manchester sewage on Mondays and Tuesdays, which are the usual washing days in Lancashire households. The composition of the water supplied by the town also affects the sewage considerably, and plays an important part in subsequent precipitation processes, especially where lime is used.

Two methods of purification at present hold the field, one, which includes all precipitation processes with subsequent land or other filtration; the other, the bacteriological method with or without previous precipitation. Of the two, precipitation and subsequent land filtration is most in accord with economic principles, for if the precipitated matter is also utilised, most of the useful and valuable products in sewage are recovered. The bacteriological process is wrong in principle, since under the most favourable conditions all the organic matter is at last completely destroyed, the carbon and hydrogen compounds going away in the form of gas, or being reduced to carbon, the nitrogen compounds being utimately reduced to nitrogen; so that, however successful, the bacteriological method could never be looked upon as ideal.

Apart from these considerations, that method will be the best which will yield the purest effluent. Unfortunately there are practically no data to decide upon the quality of different effluents. It is not sufficient to estimate the quantity of organic matter, but we must know the quality. For instance, it would not be enough to say that 1 grain of organic matter is harmless without specifying whether this 1 grain be sugar or strychnine. In the present state of our knowledge we cannot determine the specific organic substances in effluents with anything approaching accuracy, but such being the case, it would be as well to acknowledge it frankly.

In fact, it is even difficult to determine the total organic matter. Instead of attempting to find new methods which will show whether an effluent is satifactory, chemists have been satisfied with such tests as the permanganate and albuminoid ammonia processes. Both of these tests were originally designed for potable waters, and there was justification in using them for that purpose, as they might be, under certain precautions, an index of sewage contamination.

Unfortunately, all the work in sewage purification is based on these tests, so there are at present no reliable data to express what has been achieved in that direction.

A chemical test is the expression in numbers of an absolute fact which should be capable of only one interpretation: thus, if a mixture of iron protosulphate, bisulphite, sugar, strychnine, and tartaric acid were to take a certain quantity of permanganate for complete oxidation, this would not constitute a test, because it would give no clue to either the quality or the respective quantities of these substances. But if it were known that no other oxidisable compounds besides iron protosulphate were present, the quantity of permanganate would represent its equivalent in iron protoxide and would constitute a legitimate test.

The oxidisable matter in sewage is far more complex than the example given above, and permanganate can therefore only be an arbitrary test if it is applied for sewage-testing. To show the complexity of the composition of sewage, it is only necessary to enumerate a few of the substances which are known to result from the fermentation of albuminoids. Such are—according to F. Ficurannaod C. Preusse (Über den Nachirens der organischen Substanzen in Wasser, Ber. 1879, pp. 1906 ff.)—peptones, amido derivatives of one or two basic acids of the fatty series (valerianic and butyric acids, &c.), trimethylamine, and the following substances belonging to the aromatic series:—phenol, cresol, indol, scatol, tyrosin, hydroparacumaric acid, paraoxalphatoluylic acid, hydrocynnamic acid, and alphatoluylic acid. Besides these, sewage contains glycerine, fatty acids, and a large number of alcohols (aldehydes) and acids of the fatty series; constituents of human and animal excrements, and their products of decomposition; refuse from vegetables, &c., &c. The relative action of permanganate on equal weights of different substances is clearly shown in that excellent paper, which seems to have escaped in this country the attention it deserves. Of late, attempts have been made to show that the permanganate test is a criterion as to the liability to putrescence. My experiments in sewage do not confirm this, and I trust that others will examine into this matter carefully. And, after all, if the permanganate test only shows the liability of sewage to putrescence, this can be better shown by incubating a sample. In all cases sewage tests are *post facto* tests; by the time the sample has been taken, and the test made, the mischief, if any, is past repair. The albumenoid test is just as arbitrary, as it has been shown that different substances yield up different proportions of their nitrogen as albumenoid ammonia. So the sooner these two tests are expunged from sewage work the better.

In the absence of reliable tests, we may take it than an effluent will be satisfactory if it causes no nuisance either at the time of its discharge or later on. The conclusion from this is that, according to circumstances, an effluent may be more or less pure. If discharged into a swiftly-running river, from which no potable water supply is drawn, a lesser degree will suffice; if discharged into a stagnant pool, no amount of purification will be sufficient. The general principle in the present state of our knowledge can only be this, that when there is any likelihood of its ever being drawn upon for the supply of potable water, land filtration seems to be absolutely necessary, and should then be carried out under very stringent regulations. The incubating test alone can show us whether the effluent is liable to putrescence in the time during which it is exposed to habitable surroundings. The longer that period is, the more exacting should be the test. In Manchester the effluent remains stagnant in the Ship Canal, so that purification alone is not likely to be sufficient. In such a case I should propose to practically sterilise the effluent with a sufficient quantity of sulphurous acid, 8 grains of which is sufficient to sterilise the bacteria which cause putrefaction, and taking another 8 grains to neutralise the alkali present, and as an excess, 16 grains of sulphurous acid per gallon should be sufficient to keep the effluent in the Canal from putrescing, after this process has been carried on sufficiently long to remove the old stock of water from the Canal. The expense would be about 1*l*. per million gallons, or 9,000*l*. per annum; but it would have the advantage that it would not require a large capital outlay and leave room to adopt a better scheme when such presented itself. Of course, the effluent would have to be carbonated before the sulphurous acid could be added.

We may now consider more closely the two different principles of sewage purification, and give our attention to the bacteriological process, which, stripped of its scientific name, stands revealed as the familiar cesspool of old, only on a gigantic scale. But, whereas in the old days the workings of the cesspool were unknown, we are now beginning to understand a little of the forces which determine its reactions. Many changes formerly attributed to chemical action are now known to be produced by exceedingly small organisms, which are classed among infusoria and bacteria. These organisms have the power of multiplying to a considerable extent, and I have myself found that if Manchester sewage is put into a condition favourable to the development of these organisms, there will be, in the course of a couple of days, as many as 80 millions to the cubic centimetre, and even more. There are a number of different species of bacteria in sewage, the majority of them apparently harmless to human and animal life. But there are also at times to be found pathogenic germs, and always germs which cause putrefaction and the production of offensive odours. Classifying the bacteria again, we may divide them into anærobic and ærobic organisms. The first will flourish most when oxygen or air is excluded; the latter require oxygen for their existence and development. But here again it must not be overlooked that the line of demarcation is by no means sharp; that according to the different conditions of the medium an anærobic germ may become partly ærobic, and *vice versâ*.

The bacteriological school of sewage chemists take advantage of the enormous power of multiplication of bacteria, and by leaving the sewage at rest in suitable tanks which are not exposed to the light and receive but little air, they assume that the anærobic bacteria will in multiplying split up all the harmful organic compounds, and that the effluent after passing over filters will be such as to satisfy all reasonable requirements. Unfortunately, here again, all the principal data are based upon the oxygen and albuminoid ammonia tests, and are, therefore, unreliable. In the absence of really reliable facts, there is too great a tendency to treat the chemical changes wrought by bacteria in such a manner as if they were well investigated and as clear in their reactions as the precipitation of silver solution by hydrochloric acid. Our knowledge of these bacterio-chemical reactions is only in its infancy, and what we know should teach us to be very careful. Statements which were considered facts a comparatively short time ago, are known to be fallacies to-day. A number of bacteria which were once considered to be harmful in themselves, are now known to be harmless, though the products given off and produced by them are known to be amongst the most powerful poisons in existence. How are we to know that these poisons may not be produced in sewage and occur in the effluent which, according to our present chemical tests, may be considered satisfactory? The position taken up by the bacteriological school is altogether too dogmatic and offensive, and is bound to damage a theory which is quite legitimate in its principles, though only in its infancy. Anyone dissenting from them is marked as an interested person. When, to give only one instance, we consider that quinine is a more powerful poison to bacteria than carbolic acid, it behoves us to acknowledge that it is impossible to say what will be the effect of the numerous chemicals contained in town sewage on the development and products from these organisms, and that all our preconceived notions as to what we may consider harmless or harmful constituents are fallacious as regards their influence on bacteriological life.

There is another point which is extremely doubtful, and that is the statement made by that school of chemists, that it will not be necessary to separate the suspended matter before subjecting it to bacteriological tréatment. The amount of insoluble inorganic matter in sewage is not a negligeable quantity. In Manchester sewage, for instance, it amounts to about one ton per million gallons, or 25 tons per day; and such a quantity, considering that it would be deposited on the filters as gelatinous sludge containing about 90 per cent. of water, would amount to 50,000 galls. per day. Such a quantity would, after some time, completely clog the filters, and I, therefore, cannot see how, even if it were otherwise successful and harmless to health, which has still to be proved, the process could be workable in cities like Manchester without previous precipitation. I have previously pointed out that all the nitrogenous compounds would be destroyed, and, although we cannot at present recover these, yet it would be a very doubtful policy to shelve this question and to ridicule all attempts at utilising substances which represent, approximately, a value of 30,000*l.* per day in this country.

Turning now our attention to precipitation processes proper, we may say that they are all based on a fact which is well known to chemists. If we have a turbid liquid containing a small amount of matter in suspension, such matter will often settle out very badly; but if we increase the amount of suspended matter by adding an insoluble substance or by causing an insoluble precipitate to be formed in the liquid, the larger and heavier precipitate will carry the smaller amount of original suspended matter with it, and satisfactory settling will take place. These precipitation processes are too well known to require enumeration; in nearly every one lime plays some part. In every one of these the aim has been to reduce the cost to a minimum, and it will be found that, in most cases, if lime is added to the clear effluent, a further precipitation takes place, showing clearly that instead of precipitating as much of the matter in solution along with the insoluble matter as possible, the minimum amount of chemicals has been used which will just produce clarification.

It seems only rational that whilst treating sewage with precipitants we should aim at removing from it as large a quantity of organic and other matter as practicable. As regards the effluent thus obtained, it is in the absence of correct chemical tests impossible to say how much it is improved, but I have found that when the excess of lime has been removed from it by carbonic acid it will remain free from putrefaction much longer than a partially precipitated effluent.

Whilst these remarks apply to most effluents, it will be best to illustrate them further by laboratory experiments on a specific example, *viz.*, Manchester sewage. At present about six grains of lime and seven grains of copperas are used per gallon, at a cost of less than 15*s.* per million gallons for chemicals. But the effluent will yield a further considerable precipitate on addition of lime. Leaving copperas out of my experiments, I find that 40 to 50 grains of lime are necessary to precipitate Manchester raw sewage completely. The sludge thus obtained settles much more quickly than that produced at present, and therefore will require smaller tanks for settling. This sludge, pressed, dried, and distilled from a small gas retort, yields gas of good illuminating power in large quantities, also ammonia and tar products, and there remains in the retort a mixture which, amongst other ingredients, contains about 7 per cent. of finely divided carbon. This mixture might be, finely ground, used for manure, or it might be removed into a generator and there freed from carbon by blowing air and steam through, and the gases thus obtained utilised for testing or drying purposes. The residue, after the carbon had been removed, contains, besides large quantities of silica, alumina, iron, and some 10 to 15 per cent. of free lime, phosphoric acid equal to about 3 per cent., of which two-thirds is soluble in citrate. I have not been able to collect as yet a sufficient quantity of tar for analysis, and therefore leave it out at present, though I am inclined to think that it is likely to be more valuable than coal tar. On the basis of a number of laboratory experiments, I have made the following calculation of cost, which refers to Manchester sewage, 25 million gallons per day:—

*Expenditure per Day.*

| | £ | *s.* | *d.* |
|---|---|---|---|
| Lime, extra quantity .................... | 32 | 0 | 0 |
| Pressing .................................... | 35 | 0 | 0 |
| | 67 | 0 | 0 |

*Revenue per Day.*

| | £ | *s.* | *d.* |
|---|---|---|---|
| Gas, 750,000 cb. ft. at 1*s.* 3*d.* per 1,000 cb. ft. | 47 | 0 | 0 |
| Ammonia sulphate, 7 tons at 6*l.* ......... | 42 | 0 | 0 |
| Residue, 150 tons at 4*s.* ................. | 30 | 0 | 0 |
| | 119 | 0 | 0 |

This leaves for labour and other charges a margin of 52*l.* per day, or 2*l.* per million gallons. Where the sludge must be pressed in any case, the margin would be 87*l.*, or nearly 3*l.* 10*s.* per million gallons. The quantity of illuminating gas yielded by the dried sludge is equal to that yielded by 75 tons of coal; the quantity of ammonia equal to that of 700 tons of coal. The residue finely ground, should, in proportion, act as well as Thomas Slag, and might be also useful as a drier for manures; whether it will be better for being mixed with carbon or not, must be left to experiment. The value of the phosphoric acid in the residue as such alone is 8*s.* per ton. I have assumed half that value in my calculations. The figures I have given can only be approximate; as they were obtained from a number of small samples of Manchester sewage and sludge, and will only be conclusive when verified by experiments on a large scale; but they are sufficiently important to justify such larger experiments. Considering the enormous strides which the mechanical part of chemical technology has achieved in the last 30 years, the cost of working up sludge should not be prohibitive. Unfortunately sewage chemists have generally not had any practical experience in the management of large chemical works, and are, therefore, apt to overrate the difficulties of these problems, and, as the easiest way out of them, to ridicule all attempts at dealing with this matter.

To sum up, it appears to me that where the exact quantity of lime has been used which is necessary to precipitate the maximum amount of precipitable matter in the effluent, it must be more amenable to further manipulation, and should require but little further treatment. What that treatment should be will depend on local circumstances and the original character of the sewage. In most cases it would be sufficient to acidify it. It also appears to me that the cost of sewage purification can be reduced by recovering the valuable products from the sludge by dry distillation. But the whole question of sewage purification is one which should be taken up by the Government, and in this opinion I am supported by remarks made by the late Professor W. Stanley Jevons in a paper (see the Manchester Statistical Society, April 10, 1867, on the analogy between post office telegraph and other systems of conveyance of the United Kingdom as regards Government control), in which he lays down the conditions under which matters should be controlled by Government, and which appear to me to apply forcibly to the case of sewage purification.

### Discussion.

Dr. Lewkowitsch said the figures as to cost of working the proposed process, and the saving to be effected thereby were so startling that he thought Dr. Grossmann ought to further inquire into the details of the balance sheet. He himself felt so diffident about it that he certainly would not be inclined to invest in the process before he had more information about it. It would be desirable to have the cost of pressing and drying the sludge, and the subsequent carting to the gas works. Although these expenses could be kept at a reasonable figure, the gas-making part—which unquestionably would have to be done at the gas works—appeared to him somewhat doubtful. He had had some experience in making producer gas, and therefore, the idea of making water-gas out of a powder containing 7 per cent. carbon seemed to him more than startling; but, of course, this manufacture was only an alternative, the other being that of getting tar products (as to which he desired more details), and selling the residue as manure to the farmer. Such material, containing 3 per cent. of phosphoric acid, appeared to him valueless, and he thought one would have to pay the farmer a shilling per ton to take it away rather than look upon it as a source of revenue.

Mr. Herbert Porter said that in his own experience of purification of water and effluents, but not sewage, he had found a great deal depended upon the solubility of the lime at the time of mixing. Lime-water contained only 11 grains to the pint. If large quantities of lime were pitched into an already heavy solution the difficulty would be to diffuse it through the entire mass in order to do its work, and there was, therefore, a great temptation to use more lime than was necessary. He should like to ask Dr. Grossmann whether in the treatment of sewage the lime was added in solution or partially dissolved.

Mr. J. Barnes said most people who had had anything to do with sewage purification had always looked with dread on the disposal of sludge. In Accrington it was accumulating at the rate of 70 tons per week. Dr. Grossmann advocated the addition of about three times as much lime as was necessary to produce a clarified effluent. If they considered that two tons of lime per million gallons were added solely for the purpose of getting sludge cake which could be utilised, and if they considered the cost of pressing—taking a low estimate, 2*s.* a ton—it would be equal to 5*s.* or 6*s.* a ton of the dried solids, and then adding to that the extra two tons of lime for producing it, the cost would be from 10*s.* to 12*s.* per ton. He thought they could produce better gas from coal at much less cost, and as an industrial process he did not see how it was going to pay. For his own part he should like to see a sewage process which would dispense entirely with the sludge, and he was of opinion that the septic tank system as carried out at Exeter would, in a great measure, meet the difficulty, for there was no doubt whatever that a large proportion of the sludge was disappearing and had been doing so for the last 18 months.

Mr. H. Grimshaw said, with regard to the suggestion thrown out by Dr. Grossmann that the Society should inaugurate a conference of inquiry into the merits of the respective processes for dealing with the sewage of large towns, he was afraid that by the time they had completed their labours they would find that the whole of the processes had been superseded, and they would have to start *de novo*. As to Dr. Grossmann's demonstration of distilling sludge to produce illuminating gas, this experiment had been attempted before at Rochdale by Mr. J. H. Kidd, but unfortunately it had not been conducted on sufficiently scientific lines to set the matter at rest. It appeared to him that Dr. Grossmann's system of purification was to a certain extent carried out on similar lines suggested by the Chairman (Mr. Davis) some years ago. The Chairman would perhaps call to mind that he proposed passing carbonic acid through the effluent in order to remove the objectionable excess of lime and that he then burnt the carbonate of lime, along with the sludge, into cement. This, he thought, would be much cheaper than using sulphurous acid. With regard to Manchester and Salford Corporations being committed to carry the sludge to sea, he was not altogether decided in his own mind that this was the most satisfactory manner of disposing of it. In Glasgow they were tackling the sludge difficulty in a different way, and they were evidently satisfied with what they were doing. By pressing and drying it they were producing a manure for which they said they could get 1*l.* to 25*s.* per ton on a fairly large scale. He was of opinion that a source of weakness in Dr. Grossmann's process was that the effluent being sterile and passing into the Ship Canal, which was stagnant water, decomposition might be set up again. He hoped soon to have the opportunity of seeing the septic tank system at Exeter, a dark tank which in his opinion was really a cesspool. He knew of an instance where half a dozen houses drained into a cesspool, and after a considerable time the excreta had passed off in the form of gaseous and liquid products to such an extent that there was never much solid matter taken out; and the so-called septic tank system appeared to be simply a cesspool, the overflow from which was passed through aerated filters.

Dr. A. K. Miller said that if, as appeared from Dr. Grossmann's paper, it was proposed to sterilise the alkaline effluent by the addition of sulphurous acid, it would certainly be a wasteful process, as much of the sulphurous acid would become neutralised and the resulting calcium sulphite would have a much lower antiseptic action than sulphurous acid. The author had stated that he *sterilised* his effluent, but he then modified the statement by admitting that the spores were not destroyed. It was perfectly evident therefore that his effluent was not sterile, and, assuming that the effluent remained about fourteen days in the Canal, there would certainly be ample opportunity for secondary decomposition to set in after the so-called "sterilised" effluent had become diluted with Canal water.

Dr. Kleemann said that Mr. Dancer, about six months ago, had submitted a similar process to the Manchester Corporation, although he was not aware that he had patented it; but, so far, nothing had been heard from the Corporation.

The Chairman, before calling on Dr. Grossmann to reply, wished to point out that the process which Mr. Grimshaw had referred to was quite distinct from Dr. Grossmann's as regarded the disposal of the organic matter in the sludge, inasmuch as he (the Chairman) had utilised the organic matter as fuel for helping to burn the other portions of the sludge into cement.

Dr. Grossmann, in reply to Dr. Kleeman, said that his remarks could not possibly have applied to Mr. Dancer, as he was not aware that that gentleman had been engaged on the same subject. In reply to Dr Miller, he (Dr. Grossmann) passed carbonic acid into the effluent before adding sulphurous acid. A certain amount of sulphurous acid would be taken up by the alkali, but he had made allowance for that in his estimate, by placing the cost higher than it otherwise would have been. With regard to Dr. Lewkowitsch's remarks, the main point of difference seemed to be as to the manurial value of the residue, and that question would have to be decided by agricultural specialists; but he might mention that in estimating the value of the residue he had been guided chiefly by the valuable paper published by Prof. Munro on the "Value of Sludge." In reply to Mr. Porter, the lime was rendered partly soluble before being mixed with the sewage. He could not see how it was possible even in the septic tank to make the insoluble matter in sewage completely disappear. His own calculations certainly led to a very different result, as he had shown in his paper. As to carbonic acid being cheaper for ridding the effluent from an excess of lime, he used carbonic acid in the first instance and acidified afterwards with sulphurous acid. Unless sulphurous acid were used the effluent would still be slightly alkaline, in which condition it was most favourable for the development of putrefying bacteria, but these putrefying bacteria could not live in an acid effluent containing sulphurous acid.

*Meeting held on Friday, April 1st, 1898.*

MR. GEO. E. DAVIS IN THE CHAIR.

## THE OXYGEN TEST FOR SEWAGE AND EFFLUENTS, AS CARRIED OUT IN THE LABORATORY OF THE MERSEY AND IRWELL JOINT COMMITTEE.

BY J. CARTER BELL, A.R.S.M.

(*This Journal*, 1898, 11—13.)

### Discussion.

The **Chairman** said that if the permanganate test was to continue as a process, some general agreement must be made as to how the details should be performed. The effect of dilution, the strength of the various standard solutions, the indicators, the time occupied in making the test were all necessary subjects for discussion. A chemist could not say he used a particular "test" if he varied it in the most minute particular. He had found every modification of Tidy's test to give concordant results upon the same samples, if conscientiously carried out, whether by the Joint Committee's method, by Mr. Bell's method, or by any other. But when the results of these modifications were compared with each other they did not agree; so that whenever the permanganate test was employed, the exact procedure in all detail must be specified and adhered to.

The one great drawback to the ordinary permanganate test, was that it was performed in an *acid* solution, whereas a sewage effluent was always *alkaline*. Permanganate when used at a boiling temperature in an alkaline solution, acted far more completely upon the soluble organic matter than in a cold acid solution, and gave a much nearer account of the total organic matter than Tidy's process.

This alkaline process was called Wanklyn's moist combustion method, and the following figures showed the relative amounts of oxygen absorbed in grains per gallon when sewage effluents were examined by this process and those of Mr. Bell and the Mersey and Irwell Joint Committee :—

| — | J. Carter Bell. | Joint Committee | Wanklyn. |
|---|---|---|---|
| 1 | 2·0 | 2·4 | 16·6 |
| 2 | 2·0 | 2·5 | 16·0 |
| 3 | 3·5 | 4·5 | 18·0 |
| 4 | 1·8 | 2·6 | 19·6 |
| 5 | 1·4 | 1·9 | 15·0 |
| 6 | 2·0 | 2·9 | 27·0 |

It was a remarkable fact that the amount of organic matter over which disputes arise did not amount to much more than the one-tenth part of the total organic matter, the remainder of which was not recognised by the acid permanganate test. What was this remainder, and was it liable to putrefy ?

Dr. **Pankhurst** said that if the oxygen test could not distinguish between putrescent organic matter and non-putrescent, then it was of no value. The test, as applied by the Joint Committee up to the present time, was altogether arbitrary, but if it had any value at all, a uniform method of applying it should be adopted by all chemists.

Mr. **Frank Scudder** said, that chemists had for years been applying the permanganate test to sewage and sewage effluents; the test had even been used by Mr. Bell himself. In the Salford sewage trial in 1895, on being asked if he adopted the recognized process, and applied it with all care and accuracy, Mr. Carter Bell replied "Yes."

In cross-examination Counsel put the following question to Mr. Bell. (*Q.*) You used the permanganate test ? (*A.*) Simply because others used it. The Judge: Just tell me what about the three minutes' tests ? (*A.*) I did not do that, I do not consider it is any use, and I did not do it. (*Q.*) What about the 15 minutes' test ? (*A.*) That I did not do ? (*Q.*) The only test you do is the four hours' test ? (*A.*) Right.

The object of using these various time-tests was to differentiate the quality of the organic matter and in order to make this point clear, he (Mr. Scudder) divided the quality of the organic matter found in the Salford sewage effluents into three divisions as follows:—the three-minutes-test showed the putrid matter decomposing permanganate at once with acid. Angus Smith said this test measured the organic matter decomposed or putrid or at least certain gases which it left behind capable of decomposing $KMnO_4$.

15 mins. *minus* 3 mins. = 12-mins.-test showed matter readily putrefying and rapidly decomposing permanganate with acid. Angus Smith classed this as organic matter readily decomposed and probably ready to become putrid.

Four hours *minus* 12 mins. *minus* 3 mins. = 225 mins.-test showed matter capable of putrefying, although slow to decompose.

The following table differentiated the quality of the organic matter as estimated by the permanganate test.

*Salford Sewage Effluents.*

Quality of organic matter as measured by permanganate showing oxygen absorbed in grains per gallon after :—

| Sample. | 3 mins. | + | 12 mins. | + | 225 mins. | = | 4 hours. |
|---|---|---|---|---|---|---|---|
| No. 1 | 0·87 | + | 1·07 | + | 1·31 | = | 3·25 |
| „ 2 | 1·13 | + | 1·07 | + | 1·52 | = | 3·72 |
| „ 3 | 0·64 | + | 0·81 | + | 1·42 | = | 2·87 |
| „ 4 | 0·66 | + | 0·96 | + | 1·50 | = | 3·12 |
| „ 5 | 0·43 | + | 0·52 | + | 1·17 | = | 2·12 |
| „ 6 | 0·56 | + | 0·65 | + | 1·24 | = | 2·45 |

From these results a valuable differentiation of the organic matter was obtained, provided there were no inorganic bodies present such as ferrous salts, nitrites or sulphites which decompose permanganate of potash. In the Salford effluent it was proved by analysis that these bodies were absent, consequently they had a true measure of the quality of the organic matter. In 1895 Mr. Bell declared the three minutes' test to be of no use, but he now proposed to make use of it, for he said "Even Dr. Tidy when he brought this oxygen process before the Chemical Society was disposed to register the reduction which occurs in three minutes in the absence of FeO and $H_2S$ as due to nitrites, neither nitrites nor even salts of iron are harmful in an effluent, therefore it would be fairer, in reporting upon an effluent, to make some reduction for the three minutes' test." Of course Dr. Tidy was referring to the case of drinking waters and not to sewage effluent, for he stated that "during the period of the three minutes' test, all nitrites will be oxidised, whilst the organic matter will remain unchanged." As Dr. Tidy truly said, "the permanganate test, like every other chemical test, must be used with intelligence," and when they found as pointed out by Dr. Frankland that a sewage effluent, free from nitrites, reduced in three minutes as much permanganate as Thames water did in three hours, and as Dr. Odling reported, with reference to the samples of Salford sewage effluent brought in question by Mr. Bell, "the lowest of the results would suffice to condemn the effluent several times over. Indeed, the result of Scudder's three minutes' test exceeded somewhat that allowed by the Thames Conservancy for a three hours' test." It was, therefore, unreasonable to suggest that any deduction ought to be made for the three minutes' test, when nitrites and ferrous iron were absent. He did not agree with Mr. Bell when he said that salts of iron were not hurtful in an effluent. He had known cases where considerable injury and expense had been caused to manufacturers using river water, into which a sewage effluent containing salts of iron was discharged.

In order to account for the discrepancy between the results obtained by Mr. Bell and himself, a series of experiments was conducted following the details of the method as carried out by Mr. Carter Bell on the one hand, and by himself on the other.

*Comparison of the two Methods.*

The results of the examination of sewage effluents varying in organic impurity following the two methods were given in the following table :—

TABLE 1.

*Permanganate Test, Four Hours Ordinary Temperature (results expressed in grains per gallon).*

| | F. Scudder's Method. | J. Carter-Bell's Method. | Difference. |
|---|---|---|---|
| | C.c. | C.c. | |
| Sewage effluent | 70 | 20 | .. |
| Distilled water | 0 | 50 | .. |
| Sulphuric acid | 10 | 10 | .. |
| Permanganate | 50 | 10 | .. |
| Effluent, No. 1 | 0·28 | 0·19 | 0·09 |
| " 2 | 0·45 | 0·40 | 0·05 |
| " 3 | 0·75 | 0·58 | 0·17 |
| " 4 | 0·78 | 0·73 | 0·05 |
| " 5 | 0·95 | 0·80 | 0·15 |
| " 6 | 0·96 | 0·84 | 0·12 |
| " 7 | 1·10 | 0·95 | 0·15 |
| " 8 | 1·14 | 0·95 | 0·19 |
| " 9 | 1·46 | 1·15 | 0·31 |
| " 10 | 1·99 | 1·50 | 0·49 |
| " 11 | 2·24 | 1·72 | 0·52 |
| " 12 | 2·46 | 1·90 | 0·56 |
| " 13 | 2·59 | 2·02 | 0·57 |
| " 14 | 2·59 | 2·03 | 0·56 |
| " 15 | 2·84 | 2·18 | 0·66 |
| " 16 | 2·83 | 2·27 | 0·56 |
| " 17 | 3·31 | 2·48 | 0·83 |
| " 18 | 3·32 | 2·58 | 0·74 |
| " 19 | 3·68 | 2·83 | 0·85 |

From these figures it would be observed that Mr. Bell's results were invariably lower than his own, especially as the effluents became stronger in organic matter.

In his own procedure the sewage effluent was operated upon direct, and not diluted with distilled water as in Mr. Bell's procedure before adding an acid solution of permanganate. Moreover, his method involved the use of a larger volume of permanganate solution, and it acted on a stronger solution of organic matter than in Mr. Bell's process.

*Effect of using a larger volume of $KMnO_4$ Solution.*

The results given in Table 2 showed that the use of a larger volume of $KMnO_4$ solution did not account for the great discrepancy between his results and those of Mr. Bell.

TABLE 2.

*Permanganate Test, Four Hours.*

| | C.c. | C.c. | Difference. |
|---|---|---|---|
| Effluent | 20 | 20 | .. |
| Distilled water | 50 | 50 | .. |
| Acid | 10 | 10 | .. |
| Permanganate | 10 | 15 | .. |
| Effluent, No. 1 | 0·19 | 0·20 | 0·01 |
| " 2 | 0·58 | 0·66 | 0·08 |
| " 3 | 0·80 | 0·81 | 0·01 |
| " 4 | 1·50 | 1·66 | 0·16 |
| " 5 | 2·53 | 2·69 | 0·16 |

*Effect of Dilution.*

The following results given in Table 3 corroborated the deduction drawn from Table 1, that previous dilution of the sewage effluents with distilled water gave lower results than acting directly upon the effluent with an acid solution of $KMnO_4$.

TABLE 3.

*Permanganate Test, Four Hours.*

| | C.c. | C.c. | Difference due to dilution. | C.c. | C.c. | Difference due to dilution. |
|---|---|---|---|---|---|---|
| Effluent | 20 | 20 | .. | 70 | 70 | .. |
| Distilled water | 50 | 0 | .. | 0 | 175 | .. |
| Acid | 10 | 10 | .. | 10 | 10 | .. |
| Permanganate | 10 | 10 | .. | 50 | 50 | .. |
| Effluent, No. 1 | 0·19 | 0·19 | 0·00 | 0·28 | .. | .. |
| " 2 | 0·40 | 0·47 | 0·07 | 0·45 | 0·44 | 0·01 |
| " 3 | 0·58 | 0·65 | 0·07 | 0·75 | .. | .. |
| " 4 | 0·80 | 0·88 | 0·08 | 0·95 | .. | .. |
| " 5 | 0·95 | 1·14 | 0·19 | 1·10 | 1·00 | 0·10 |
| " 6 | 1·50 | 1·87 | 0·37 | 1·99 | .. | .. |
| " 7 | 1·72 | 2·01 | 0·29 | 2·24 | 1·69 | 0·55 |
| " 8 | 1·90 | 2·11 | 0·21 | 2·46 | 1·99 | 0·47 |
| " 9 | 2·02 | 2·17 | 0·15 | 2·59 | 2·14 | 0·45 |
| " 10 | 2·83 | 3·34 | 0·51 | 3·68 | 2·99 | 0·69 |

*Effect of using a larger volume of Sulphuric Acid Solution.*

In any effluent absorbing over 2 grs. of oxygen per gallon it was found better to add 20 c.c. acid, as the permanganate solution then remained clear and free from any precipitation of hydrated manganese oxide. The results were not, however, influenced by this modification.

TABLE 4.

*Permanganate Test, Four Hours.*

| | C.c. | C.c. |
|---|---|---|
| Effluent | 70 | 70 |
| Acid | 10 | 20 |
| Permanganate | 50 | 50 |
| Effluent, No. 1 | 0·78 | 0·77 |
| " 2 | 0·96 | 0·98 |
| " 3 | 1·14 | 1·11 |
| " 4 | 1·46 | 1·43 |
| " 5 | 2·84 | 2·84 |
| " 6 | 3·31 | 3·34 |

From these experiments he came to the conclusion that the cause of the difference between the analyses of the effluents taken from the Salford sewage works was due principally to the fact that Mr. Bell diluted the effluent with distilled water before applying the permanganate test. By so doing the test was applied to a much weaker solution of organic matter than when the effluent was treated direct with the acid solution of $KMnO_4$. Consequently from a scientific point of view, it was preferable to adopt his (Mr. Scudder's) procedure. This was confirmed by Dr. Odling who stated that "it is quite in accordance with experience in my laboratory that previous dilution of the effluent brings out the estimation of oxygen absorbed low," and he went on to say, "I support your results and process."

Mr. Bell suggested that discrepancies might arise from the following causes:

(1) Temperature (2) Time between collection of samples and analysing; (3) end reaction not sharp enough if too much sewage be present; (4) the rate at which the hyposulphite is run in; and (5) the presence of suspended matter decolourising the $KMnO_4$ at the bottom of the flask.

(1) As regards temperature, as both Mr. Bell and himself conducted their tests at the ordinary temperature of the laboratory, the great discrepancy between our results cannot be accounted for thus. The effect, however, of temperature had long been fully recognised and established, and the results given by Mr. Bell in table headed "chemicals

used for one million gallon of sewage" bore out generally his experience of the effect of temperature. From this table of Mr. Bell's he had calculated the percentage purification effected, and the results were interesting inasmuch as they showed that whether 60° or 80° be the temperature adopted, the results were in ratio, and therefore the conclusions to be drawn from the results must necessarily be the same. On the other hand high temperatures such as 212° F. gave most erratic results and difficult to interpret. Moreover, errors of a recognised character appeared, and in consequence the use of a high temperature was condemned.

2nd. As regards time between collection of sample and analysing.

The rapid changes which the organic matter in a sewage effluent underwent by keeping, was most important to note. Mr. Bell cited the case of a sample of settled sewage rapidly generating $H_2S$ from the decomposing organic matter. On the other hand the Massachusetts Board chemists recited a case on page 461 of the Report of the State Board of Health for 1894 of a sample of fresh sewage, which, when allowed to stand for 24 hours decreased in organic nitrogen (Kjeldahl) 51 per cent, the oxygen consumed 25 per cent., whilst the free ammonia increased over 100 per cent.

In the laboratory of the Mersey and Irwell Joint Committee considerable time and attention had been given to the study of the changes which sewage effluents underwent by keeping under varied conditions, and the results were of great value in judging of the liability of an effluent to undergo secondary decomposition. He need however quote only the results of one experiment of the hundreds that had been made to illustrate the value of studying the organic changes that occur on keeping sewage effluents.

*Sample of Tank effluent from the Manchester Corporation Sewage Works. After Lime and Copperas treatment.*

The sample was well mixed and filled into bottles holding about 450 c.c. Other bottles (half Winchesters) were about half filled with 450 c.c. sewage effluent, leaving 600 c.c. of air space (the stoppers were put in). Another portion of the sewage was diluted with an equal bulk of tap water and filled into bottles (to stoppers).

One half of each set was put into the incubator, other half being set on window ledge of laboratory.

No. 1. Bottle half filled.

No. 2. Bottle completely filled.

No. 3. Bottle ½ sewage, + ½ tap water.

| Date analysed. | | Ammonias. | | | | Oxygen absorbed Test | | | | Organic Nitrogen, Kjeldahl. Process. | | Samples on Window Ledge of Laboratory. | Samples in Incubator. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Free. | | Albuminoid. | | 3 Minutes. | | 4 Hours. | | | | | |
| | | 60° F. | 75° F. | 60° F. | 75° F. | 60° F. | 75° F. | 60° F. | 75° F. | 60° F. | 75° F. | | |
| 11/11/96 | Original | 2·15 | .. | 0·229 | .. | 0·95 | .. | 3·26 | .. | 0·482 | .. | Neutral chlorine, 25·6 grains per gall. | ... ... |
| 11/11/96 | ½ water + ½ sewage | 1·19 | .. | 0·137 | .. | .. | .. | 1·72 | .. | 0·332 | .. | ... ... | ... ... |
| 16/11/96 | No. 1 | 2·29 | 2·33 | 0·243 | 0·232 | 0·95 | 0·85 | 2·69 | 2·29 | 0·50 | 0·61 | Unchanged in appearance | Fungus on surface, slight smell. |
| | No. | 2·61 | 2·85 | 0·241 | 0·201 | 1·01 | 1·29 | 2·37 | 3·27 | 0·54 | 0·46 | Commencing to turn dark at foot, and had faint smell. | Black sediment, slight smell. |
| | No. 3 | 1·31 | 1·28 | 0·165 | 0·106 | 0·63 | 0·70 | 1·53 | 1·56 | 0·27 | 0·22 | Clear and colourless, with little sediment. | Clear, but sediment turning black. |
| 19/11/96 | No. 1 | 2·38 | 2·59 | 0·212 | 0·156 | 0·88 | 0·67 | 2·73 | 1·76 | 0·41 | 0·43 | No smell, sediment grey coloured. | Fungus on surface, slight smell. |
| | No. 2 | 2·66 | 3·15 | 0·187 | 0·140 | 1·02 | 1·50 | 2·91 | 3·57 | 0·41 | 0·39 | Getting dark tinge, sediment black, slight smell. | Dark-tinged liquid, sediment black, $H_2S$ present. |
| | No. 3 | 1·26 | 1·19 | 0·089 | 0·078 | 0·34 | 0·72 | 1·55 | 1·56 | 0·31 | 0·25 | Sediment grey, liquid clear, not putrescent. | Sediment black, liquid dark tinge, $H_2S$ present. |
| 23/11/96 | No. 1 | 2·31 | 2·31 | 0·249 | 0·212 | 1·00 | 0·70 | 2·56 | 1·30 | 0·47 | 0·30 | Slight putrescent smell, grey sediment, fungus on surface. | Liquid clear, grey sediment, fungus on surface. |
| | No. 2 | 2·45 | 3·15 | 0·201 | 0·162 | 1·14 | 2·28 | 2·67 | 4·44 | 0·32 | 0·29 | Sediment black, putrescent smell. | Sediment black, liquid dark, $H_2S$ and putrescent smell. |
| | No. 3 | 1·33 | 1·22 | 0·106 | 0·028 | 0·71 | 1·34 | 1·61 | 3·86 | 0·17 | 0·16 | Liquid clear, little grey sediment, hairy fungus. | Sediment black, with black fungus, $H_2S$, and putrescent smell. |
| 26/11/96 | No. 1 | 2·24 | 2·35 | 0·285 | 0·190 | 0·82 | 0·67 | 2·33 | 1·42 | 0·54 | 0·40 | Sediment brown and grey, fungus on surface, no smell. | Sediment brown, fungus on surface, no putrescent smell. |
| | No. 2 | 2·45 | 2·94 | 0·179 | 0·168 | 0·98 | 2·60 | 2·69 | 4·01 | 0·56 | 0·57 | Sediment black, slight putrescent smell. | Black sediment, liquid dark, putrescent and $H_2S$. |
| | No. 3 | 1·40 | .. | 0·112 | .. | 0·62 | .. | 1·58 | .. | 0·26 | .. | Sediment grey, liquid clear. | No sample. |

3rd. As to the end reaction not being sharp enough if too much sewage be present.

This is not his experience, and no difficulty had arisen in getting a sharp end reaction using 70 c.c. of the Salford sewage effluent. The results given in Table 3 where 20 c.c. and 70 c.c. of effluent respectively were operated upon were as nearly as possible the same.

4th. As to the rate of adding the $Na_2S_2O_3$ solution.

The titration of an iodine solution was one of the simplest and most accurate of volumetric manipulations, but it could not be satisfactorily carried out in a wide mouthed vessel as used by Mr. Bell. A narrow stoppered bottle must be used. Moreover it was important to have an excess of KI present, otherwise free iodine escaped from the liquid.

5th. As to the presence of suspended solids.

It was not necessary to *constantly* shake the solution as suggested by Mr. Bell, an occasional shake being sufficient, and that only in the case of sewages heavily charged with suspended solids.

The permanganate test as carried out in the laboratory of the Mersey and Irwell Joint Committee had proved of great value in the examination of sewage effluents. It was not solely relied upon in forming a judgment of the quality of an effluent and its fitness to be discharged into a stream. Other scientific data must be taken into consideration in addition. There had been considerable divergence of opinion expressed from time to time as to the propriety of the permanganate test, and as to the limits of impurity allowable in sewage effluents (*see* this Journal, Jan. 1897, p. 16 *et seq.*).

The following table showed the results of the examination of 307 sewage effluents submitted by the Mersey and Irwell Joint Committee to Sir Henry Roscoe for analysis during the past 12 months.

*Permanganate Test, Four Hours.—Results of the examination of sewage effluents submitted by the Mersey and Irwell Joint Committee. January to December* 1897, *from* 54 *Sanitary Authorities representing a population of* 1,684,002.

| — | — | Percentage. |
|---|---|---|
| Effluents classed as good and fair ........ | 220 | 71·7 |
| " " unsatisfactory ....... | 47 | 15·3 |
| " " bad......... .... ... | 40 | 13·0 |
| Total tests ............... | 307 | 100·0 |

Effluents that absorbed oxygen from—

| | |
|---|---|
| 0·07 up to 1 grain per gallon .............. | 211 |
| 1·0 grains to 1·25 grains per gallon........ | 21 |
| 1·25 " 1·50 " " ........ | 10 |
| 1·5 " 2·0 " " ........ | 14 |
| 2·0 " 2·5 " " ........ | 14 |
| 2·5 " 3·0 " " ........ | 8 |
| 3·0 and upwards......................... | 29 |
| | 307 |

| | | |
|---|---|---|
| Good and fair effluents— | | |
| From precipitation alone ............ | 7 | |
| " filtration ...................... | 213 | |
| | | 220 |
| Unsatisfactory effluents— | | |
| From precipitation alone ............ | 16 | |
| " filtration......................... | 31 | |
| | | 47 |
| Bad effluents— | | |
| From precipitation alone ............ | 34 | |
| " filtration.......................... | 6 | |
| | | 40 |
| Total ............... ..... | | 307 |

Of the 40 bad effluents 27 were from seven sanitary authorities, who admitted that these effluents were bad. The remaining 13 samples were from the Salford sewage works, and Mr. Bell contended that they were satisfactory inasmuch as when they were kept in closed bottles for a period varying from seven weeks to ten months considerable purification had taken place as measured by the permanganate test. Mr. Bell quoted analyses of some of the official samples taken by the Mersey and Irwell Joint Committee during the year 1897. On reference to his own note books he found that many of these effluents referred to by Mr. Bell stank after being kept at a temperature of 75° F. for seven days.

With reference to the action of phenol on the permanganate test, Mr. Bell quoted the case of a sewrge effluent having been condemned as a bad effluent by him (Mr. Scudder), and pronounced by himself to be a good one. On reference to this particular case, he found that on May 11th, 1897, a sample of effluent from the Reddish sewage works was submitted to him (Mr. Scudder) for analysis by the Mersey and Irwell Joint Committee, and he reported to them that the four hours' oxygen test was 6·7 grains per gallon, and that "with regard to this effluent the amount of sewage impurity was insignificant, the high oxygen absorbed figure being due to the presence of a highly volatile reducing substance, which could not be identified owing to the small quantity of liquid. The free and saline ammonia was 1·26 grains per gallon, the albuminoid ammonia 0·05 grains, chlorine 35 grains per gallon. Further samples are necessary for investigation."

In a subsequent sample submitted on June 17th, the oxygen absorbed was 7·06 grains per gallon, and he reported that "the amount of sewage impurity was insignificant, the albuminoid ammonia was 0·03 grains, the organic nitrogen 0·15 grains, and the chlorine 52·5 grains per gallon. The presence of phenol accounts for the high oxygen absorbed, and indicates that manufacturing impurity is present although of a harmless character. A fair effluent."

The high oxygen absorbed figure did not lead him to condemn the effluent, and its value in determining a source of pollution which no other test indicated could not be overlooked.

As further showing the action of tar acids on the permanganate test, Mr. Bell quoted analyses made by him (Mr. Scudder) of effluents from the treatment of Salford sewage by the Bacillite Company's process, which were "branded by the oxygen test as harmful." Mr. Bell stated that "these effluents remained bright and clear for weeks, showing no signs of secondary decomposition, yet this oxygen test classified them all as bad effluents." As average daily samples of the effluents resulting from the treatment of the Salford sewage by this particular process were collected on October 15th, 1895, and continued until October 30th, 1895, and submitted for analysis and report, he was able to say that the permanganate test was not the only one that branded these effluents as harmful. Moreover, these effluents while invariably clear and free from suspended solids when first collected and possessing a faint tarry smell, became on standing a comparatively short time turbid, deposited, solid matter (chiefly carbonate of lime and organic matter), and gradually underwent putrefaction, evolving a stink, having first the odour of sewer gas, and eventually, in the majority of cases, that of sulphuretted hydrogen. The samples were kept in stoppered bottles, and at a temperature of 75° F., conditions favourable to secondary decomposition.

Mr. Bell said that waters containing antiseptics would be most beneficial to the Ship Canal. This he did not agree with, because in the first place the amount of phenol or cresol which was necessary to prevent the development of a stink was large and the expense exorbitant; secondly, as Angus Smith pointed out, disinfection was not a magic art performed by a small piece of some substance which removed all evils at once, and the greater the amount of these tar acids used the greater the amount of sulphuretted hydrogen when there was much water. They produced a decomposition which Angus Smith called "the putrescent odour of carbolic acid," and which resembled the odour of sewer gas.

Mr. Bell quoted experiments on the action of permanganate of potash on such bodies as phenol and sulphate of quinine, and his results required some explanation. Applying the three minutes' test to a solution of phenol (1 grain to the gallon), Mr. Bell stated that the Mersey and Irwell method gave a result 0·00004 as against 0·87 by his own method, the difference being some thousands per cent. The figure (0·00004) must be wrong, inasmuch as if 70 c.c. of the phenol solution were taken the volume of $KMnO_4$ solution used would only be $\frac{1}{25000}$ c.c., which was not measurable by a burette. Moreover, this difference of some thousands per cent. was not borne out by the figures given in Mr. Bell's last table.

The results of his own experiments were as follows:—

*Phenol and Sulphate of Quinine.*

| — | Oxygen required for | | |
|---|---|---|---|
| | Three Minutes. | | Four Hours. |
| | Adding $KMnO_4$ direct. | Adding $KMnO_4$ in excess. | |
| Phenol, 1 grain per gall. ........... | 1·00 | 1·12 | 1·73 |
| Sulphate of quinine, 5 grains per gall. | 0·22 | 0·36 | 1·20 |

Mr. Bell said that the three minutes' test, as conducted by him (Mr. Scudder), was going back to the original process of Forchammer, and quoted Tidy as saying, with reference to this method: "I am free to admit that, conducted in this way, the permangate process is irregular, inaccurate, and wanting in delicacy." Now, Dr. Tidy's remarks did not apply either to the original process of Forchammer or to his (Mr. Scudder's) method, as reference to the original paper by Dr. Tidy in 1879 would show (J. Chem. Soc. **35**, 67). Dr. Tidy was referring to Wanklyn's want of accuracy in describing the method of performing the experiment with $KMnO_4$ in his book on water analysis.

Dr. D. B. Hewitt, as a member of the Mersey and Irwell Joint Committee, said he had been acquainted for many years with the permanganate test, and he believed it was used by Dr. Angus Smith, who was a conscientious and able chemist, but in addition to that he remembered a paper read before the Manchester Literary and Philosophical Society by Dr. Burghardt, in which he set out very fully a great number of tests made by permanganate, extending from the upper regions of the River Irwell down to Mode Wheel and the Barton Locks, and he relied very strongly indeed on the permanganate test as a means of indicating the putrefactive character of sewage. A statement had been made that should be made perfectly clear. It was said that there had been some difficulty in obtaining from the Joint Committee a satisfactory description of the mode of making their test. It was perfectly correct that at one period there might have been some difficulty in obtaining information. In courts of law contending parties were not very willing to show their hands, and he believed in the Salford case the chemists were very ingenious in their explanations. It had been said that a lime effluent would not decompose, but he had seen the effluents both from Manchester and Salford looking very clear and bright, and the moment they touched the waters in the Ship Canal there was a precipitation which resulted in a certain amount of solid and organic matter being deposited in the canal, which formed sludge, and which the canal authorities had to take out. It had been frequently stated that the effluents from Manchester and Salford were much better than the canal water, but it had been proved beyond doubt that the ship canal was much worse below the outlets of the Manchester and Salford effluents than above them. Now, whether the permanganate was the proper test or not, it was not the test solely relied upon by the Joint Committee, although it had been used from the beginning for the purpose of establishing certain ratios; and the ratios of Mr. Bell were no better than the ratios of Mr. Scudder. Mr. Bell knew that every time a complaint was made by the Joint Committee they sent not only the results of the permanganate test, but the albumenoid test and the amount of alkalinity. He admitted there was a time when the Joint Committee would not give everything that was asked, but he was quite sure that if any chemist wished to go to the Committee's laboratory and discuss the matter in a friendly way, they would be very welcome. He found that Mr. Bell, as analyst to the Cheshire County Council, advised that the effluents measured by the same test as used by him at Salford, were not fit to enter the Cheshire rivers, while the effluent at Salford was good enough for the ship canal. He (Dr. Hewitt) was pleased to hear the Chairman say that he had tried the permanganate test in several ways, and he quite followed him that it was very likely, when carried out in different ways, very different results were obtained, but Mr. Scudder's results gave useful comparative figures or ratios. He thought Mr. Carter Bell had done a great service to the State in bringing this matter forward and having it discussed by a number of skilled chemists, and one of the objects of that Society was to compare notes and come to some agreement as to a standard method to be adopted.

Mr. H. Grimshaw said he did not wish to defend the imperfect way in which this test had often been applied, and he thought it was not altogether creditable to them as chemists that they had not come to some settlement as to a standard method to be adopted throughout the country. Notwithstanding so much had been said against the test, when they had some hundreds or thousands of tests to make, some easy method of analysis had to be adopted, and certain allowances made for discrepancies in the results. No two chemists seemed to have settled down to the same *modus operandi,* and they were all agreed that, when this was the case, the test was valueless. If they could only come to some agreement that an effluent, however produced, would not subsequently become offensive, then they might agree to a basis upon which the oxygen test could be applied, so as to show that an effluent would not be liable to secondary decomposition, and might therefore be allowed to pass into an industrial river.

Mr. R. Pettigrew said that the permanganate test in capable hands was, in fact, the best comparative test, and in submitting a large number of sewages each month to the Joint Committee (who were not all chemists) such a test was a great guide and enabled them to understand, in conjunction with the remarks of the Chemical Adviser, the position of the various Local Authorities much better than if complete analysis were submitted in each case. When 20 or 30 sewages had to be done in a day, it was, on the face of it, of no special value to make full analyses of first-class effluents, and, after long experience, it was found that effluents which had an oxygen absorbed value of not over one grain per gallon four hours test, were not usually liable to undergo putrefaction. Hence this test was first applied (both three minutes and four hours) and all which came out below one grain per gallon four hours test were laid aside as satisfactory, and those above this figure were more fully examined into and classed accordingly. There had been statements about the permanganate test and manufacturers effluent, but to such this test (unless in the case of brewers', fellmongers', and such like effluents) was seldom applied.

A great deal had been spoken about nitrites and ferrous salts and their action on the permanganate test. Those who spoke surely did not realize the position of these two compounds. He had seldom, unless during heavy rains, found nitrites present in tank effluents, and in filtered effluents they would be at once detected by observing the ratio between the three minutes' and four hours' test; moreover, every sample delivered was tested separately for nitrites.

With regard to ferrous iron, suppose 10 grains per gallon of ferrous sulphate were used for precipitation purposes, and that the whole of this iron escaped in the ferrous sate, no precipitation taking place, then in 70 c.c. (the amount taken for the permanganate test) of this liquid there would be 0·031 grains of iron as (Fe) which would make the estimation of the oxygen absorbed only 0·28 grains per gallon too high. But as the amount supposed to escape was absurd, it was plain that ferrous iron could have no bearing on the question. No test they knew of for sewage analysis gave absolute figures and they did not want that, but only the measure of putrescence or liability to putrescence. The permanganate test in skilful hands showed this in most cases better than any other single test, and he who neglected the permanganate process did not use the best methods he had at hand.

Dr. J. Grossmann said that there were many substances likely to be found in sewage which were not of a noxious character, and yet consumed a certain amount of the permanganate solution in making the test, and again processes might be used involving the employment of sterilizing substances which also would consume permanganate solution. Although the test was highly praised in certain quarters, owing no doubt to its being easy of execution, he did not agree with Mr. Pettigrew in the opinion that the chemist who did not employ it "did not use the best methods he had at hand."

Mr. A. Shearer said that the most characteristic constituent of domestic sewage, viz.: urea, had no reducing action upon permanganate under the condition of the "oxygen consumed" test even at 110 F. Mr. Sisson mentioned to him that he had found that urea, when boiled with strong alkaline permanganate, yielded abundance of ammonia. He repeated the experiment with the same sample of urea and obtained similar results. Thinking that possibly the urea might have been prepared from potassium cyanate and ammonium sulphate, he prepared with great care some pure urea from urine. From this he made a dilute solution and added a quantity equal to 5 millegrammes along with a small quantity of pure carbonate of soda (about 15 centigrammes) to 500 centimeters of water and distilled as described by Wanklyn in his text book. He obtained small but quite appreciable quantities of free ammonia, and then added alkaline permanganate and obtained in each fraction of 50 c.c. very considerable quantities of ammonia, the fourth fraction containing considerably more than the first. He then diluted the contents of the flask with ammonia free water and continued the distillation, more ammonia distilled and although the flask finally frothed over and spoiled the experiment, in all 0·4 millgrammes of ammonia was obtained.

The fact that ammonia was yielded in the way described was most surprising to him, seeing that Wanklyn stated most emphatically that no ammonia was yielded by urea under the above conditions.

With respect to the oxygen consumed test, he had recently made a number of experiments with a view to ascertaining what variation in the results took place when the conditions as to temperature, dilution, and duration of the test were altered. Without going into details, his results showed him that the most important differences occurred when the temperature varied, as much as 40 per cent. difference in the figures being obtained when the temperatures varied between 60° and 90° F. Dilution he found made very little difference in the result; time of course being an important factor. One speaker had called the test arbitrary. He agreed with him. It was both arbitrary and empirical, and its value as a means of determining the amount of pollution was greatly detracted from owing to the many variations and eccentricities of the test. It would be almost better if chemists would confess the failure of chemical tests alone as a means of determining the character of sewage effluents. The stability or otherwise of an effluent could be most readily determined in his opinion by placing the portion to be tested under conditions of temperature favourable to the development of any bacteria which might be present, and keeping it, say for a few days, when the sense of smell would determine accurately whether or no any objectionable change had taken place.

Mr. J. Barnes said that several years ago, when he first decided to try the permanganate test for sewage and sewage effluents, he knew of no recognised or published method. The tests he had to make were not for publication but for his own guidance, so he adopted what appeared to him to be the simplest method. The oxygen process described in the books was for the purpose of indicating and to a certain extent measuring minute quantities of organic matter which might be present in drinking water. It did not appear to him advisable to dilute the sewage with pure water down to the level of a bad drinking water in order that it might be treated by an extremely dilute solution of permanganate. The permanganate which he had used was ten times as strong as that recommended for testing drinking waters, and the sewage or effluent was not diluted with water previous to testing, unless the sample was a very bad one. 2½ hours was the time adopted because it was of a convenient duration to fit in with a morning's work, and the temperature of 70° F. was found to be one very easily maintained either in Summer or Winter by means of a tank of water. There was only one standard solution, the titre of which need be accurately known, and that was the permanganate used for the final titration, and which contained $KMnO_4$ equivalent to 0·5 milligram of oxygen per cubic centimetre. Now, this was the method he had always used, and the details had already been described in the paper referred to by Mr. Bell. Since then he had made further experiments upon the effect of diluting and also upon the effect of varying the degree of acidity and of temperature. He had also examined the influence of chlorides both in the presence and absence of ammonia. The details of these experiments he hoped to be able to lay before the Society at some future time, but he might mention the conclusions which he had so far been able to arrive at. The presence of chloride would only vitiate the results when there was present more than a certain amount of acid, or when the temperature was too high. In the presence of ammonia it was found that nearly all the chlorine was reconverted or retained as HCl. when the process was conducted at the temperature obtained by the boiling water bath, there being presumably a corresponding liberation of nitrogen. The numbers obtained with different degrees of dilution varied to some extent, still the variation was not such as to make the process an unreliable one. The temperature ought to be kept constant to within one or two degrees of the solutions frequently agitated during the period allowed for oxidation. The amount of organic matter taken should not be more than would deoxidise one-half of the permanganate. If these points were attended to there was no reason why different chemists should obtain very varying results.

Mr. J. Carter Bell, in reply, said that he began to use the permanganate test some 24 years ago, but as the test was an empirical one, so much depending upon the details in carrying it out, and as his methods were in existence long before the Mersey and Irwell Acts came into operation, he thought his process should have had a little more consideration. Some eight years ago, when the Salford sewage was undergoing treatment by the Electrical Purification Company, he had agreed with Sir Henry Roscoe, the chemical adviser to the company, that the oxygen test should be conducted for three minutes and three hours at laboratory temperatures, and he thought he should have been informed when any change had been made in the procedure.

Mr. Pettigrew's and Dr. Hewitt's remarks were, he (Mr. Bell) considered, very valuable, and to the point, as from the former it was easy to see the *modus operandi* followed in judging sewage effluents, and Dr. Hewitt had smoothed the way for a reasonable settlement of the question. He could not, however, quite understand the difference in behaviour of the effluents in the Mersey and Irwell laboratory and in his own. Effluents that had been reported as "bad" and "putrid" by the Joint Committee, had neither decomposed nor emitted any putrid odour in his (Mr. Bell's) laboratory, so that he could only come to the conclusion that in the Joint Committee's laboratory, the samples had been subjected to extraordinary conditions. He doubted the fairness of subjecting effluents to a high temperature in a stagnant atmosphere such as that of an incubator.

Mr. Scudder had quoted Dr. Angus Smith very often, but he doubted whether he was sufficiently accurate in the expression of the late doctor's views. The late Dr. Angus Smith patented a process for disinfecting sewage with a mixture of the sulphites and carbolates of magnesia and lime which, with his vast experience, must be considered practicable and effective. A disturbance of the oxygen test can be well imagined wherever this process should be used.

The figure (0·00001) mentioned by Mr. Scudder at the close of his remarks referred to a gramme and not to a grain—in grains per gallon it should read 0·04. He hoped the discussion would not have taken place in vain, for it proved that if uniform results were desired all chemists must work by precisely the same methods.

*Meeting held on May 6th,* 1898.

MR. GEORGE E. DAVIS IN THE CHAIR.

## NOTE ON THE CONSTITUTION OF INDICAN AND SOME DERIVATIVES OF INDIGOTIN.

BY LEON MARCHLEWSKI, PH.D., AND L. G. RADCLIFFE.

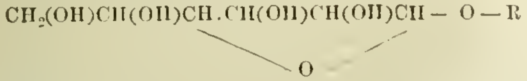
Since E. Schunck's classical researches on indican very little has been done in order to clear up the constitution of this substance. Considering the great importance of this glucoside from a technical point of view, and remembering that only by a thorough knowledge of its constitution can the danger which threatens the production of natural indigo be possibly averted (we refer, of course, to the great improvements achieved in the preparation of artificial indigotin), it may be permissible to call your attention to a few facts and theoretical considerations, which may, in conjunction with the well-known discoveries of Schunck, throw some light on the constitution of indican.

The chemistry of indican is, of course, closely dependent on the chemistry of the glucosides, and it is therefore necessary to call attention to the constitution of glucosides in general.

Glucosides are, as this name already indicates, condensation products of at least one molecule of a hexose, pentose, or carbohydrate generally, with a molecule of another substance of acid or basic character, amongst which may be found phenolic substances, amido compounds, and carbohydrates. The glucoside of levulose, for instance, is cane sugar.

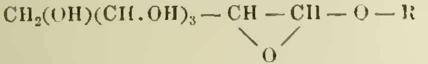
The mode of combination of the radical of the carbohydrate with the other molecule constituting a given glucoside was clearly shown by one of us four years ago (L. Marchlewski, J. Chem. Soc., London, 1893, 1137). It was found that simple glucosides—that is, such as are composed of one molecule of a hexose and one molecule of another substance, which latter is not a carbodydrate—do not contain aldehyde or ketonic groups, provided that the substance combined with the hexose does not possess such groups. In other words, the aldehyde group which, according to certain authorities, exists in free glucose is not present in the glucosides; consequently those atoms which form in ordinary glucose under certain circumstances the aldehyde group, are arranged quite differently in glucosides.

The constitutional formula which explained this behaviour of glucosides was as follows:—



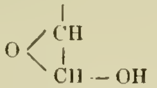
$$CH_2(OH)CH(OH)CH.CH(OH)CH(OH)CH-O-R$$

in which R represents a phenolic or basic body combined with the glucose residue. Later on it was found necessary to modify the above formula to some extent; the following one has, therefore, been proposed (Berichte, 1893, 2928):—

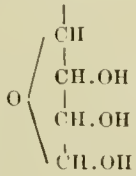


$$CH_2(OH)(CH.OH)_3-CH-CH-O-R$$

This new formula found an excellent support in the remarkable researches of Lobry de Bruyn and his co-workers, concerning the action of alkalies on glucose and similar carbohydrates. It was found that glucose by the action of alkalies can be partly converted into mannose and levulose, but that no traces of galactose could be detected; and as it is well known that these three carbohydrates belong to the same stereochemical group of substances—so far as the configuration of the atoms attached to the four neighbouring carbon atoms is concerned, the only difference being in the configuration and constitution of the groups connected with the two first carbon atoms—it will follow that the observed interconversion of the three carbohydrates is due to the movability of the atoms attached to the first two carbon atoms of the normal chain, the intermediate changes being of the molecular group:

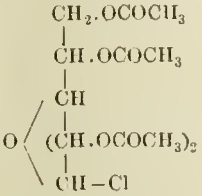


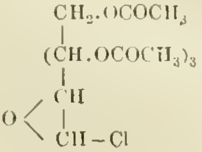
and not—



because in the latter case it would be expected that galactose would be found amongst the transformation products of glucose.

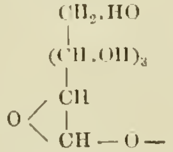
There is, however, still another and probably more important argument in favour of the acceptance of the new formula for the glucosides. It is well known that aceto-chlorhydrose, *i.e.*, the reaction-product of acetyl chloride and glucose, can be condensed with sodium salts of phenols or carbohydrates, producing artificial glucosides, and the products so obtained are identical with certain natural products. We can, therefore, take it for granted that aceto-chlorhydrose is principally constituted on the same lines as glucosides, and therefore one of the following two formulæ will represent its constitution, as other formulæ are not possible:—





Between these two we may easily decide by studying the transformation products under the influence of alkalies. If amongst them we find galactose, then the first formula for aceto-chlorhydrose will be the correct one; but if galactose is proved to be absent, we shall have to accept the second formula.

The experiments (which have already been mentioned in a note presented to the Academy of Science in Cracow) have proved that galactose has not been produced, judging from the absence of mucic acid, which should have been formed by the oxidation of the transformation products of aceto-chlorhydrose under the influence of alkalies. We consider, therefore, the second formula for aceto-chlorhydrose to be the correct one, and consequently the new formula for glucosides is to be preferred to the old one. There cannot, therefore, be any doubt that the group—

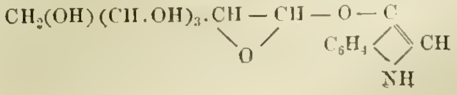


is present also in indican, and it remains to discuss the question as to what is the nature of the second component, which, in conjunction with the glucose radical, constitutes the molecule of indican.

We frequently find the belief expressed that indican is simply the glucoside of indigo-white. This view might, at first, appear not improbable: the formation of indigotin from indican might have been easily explained by assuming that indican is decomposed by acids or by enzymes into glucose and indigo-white, the latter being oxidised to indigotin. But this view is certainly not correct, and it is quite incomprehensible how it could have originated in the face of the careful researches published by Schunck and and Römer (Ber. d. deutschen Chem. Ges., 1879, 2311).

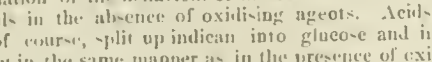
These savants have shown that by decomposing indican with hydrochloric acid in the absence of oxidising agents, a substance is produced which by subsequent oxidation cannot be converted into indigotin; the decomposition products of indican by acids cannot, therefore, be indigo-white and glucose. Further, Schunck and Römer have shown that indigotin is formed from indican by the action of hydrochloric acid in the presence of an oxidising agent, and that these conditions—the presence of a hydrolysing agent and of an oxidising agent at the same time—are necessary for the production of indigotin. These observations are, we think, of the greatest importance, and have led us to construct a theory regarding the constitution and decomposition of indican, which we now beg to submit.

We think that indican cannot possibly contain the whole molecule of indigotin in any form whatsoever; that the formation of indigotin is caused by a condensation process going on under favourable conditions simultaneously with the hydrolysis of the indican. We further believe that the parent substance or origin of the indigotin is indoxyl, and that the constitution of indigotin might be expressed in this case by the formula—



in which a molecule of indoxyl appears to be condensed with one molecule of glucose with the elimination of one molecule of water, this latter being formed from a hydrogen atom from the glucose and the hydroxyl group of the indoxyl or *vice versa.*

This formula, as will be seen, explains without difficulty the properties of indican, so far as the formation of indigotin and glucose under the influence of acids and oxidising agents is concerned. Moreover, it also gives a satisfactory explanation of the behaviour of indican under the influence of acids in the absence of oxidising agents. Acids alone will, of course, split up indican into glucose and indoxyl exactly in the same manner as in the presence of oxidising agents; but the indoxyl in the free state will be apt to condense with any aldehydic or ketonic compound which happens to be present, or even with the second decomposition product of indican, *viz.*, the glucose. It is very possible that, under these conditions, a different condensation product will be obtained, which may also contain the radical of glucose and indoxyl, but arranged in a different manner to that of indican. In other words, we may expect the formation of an indogenide of glucose which will be isomeric with indican, and possess the formula—



$$CH_2OH.(CH.OH)_4.CH = C\left\langle\begin{matrix}CO\\NH\end{matrix}\right\rangle C_6H_4$$

a substance which will be distinguished by great stability, like other indogenides, and which will probably resist the action of hydrolysing agents to a much greater degree than indican.

Finally, this hypothetical formula for indican will also give some explanation of the formation of indrubin from indican under certain conditions; inasmuch as one of the constituents necessary for the synthesis of indrubin, *viz.*, the indoxyl, is already present, and the other, the isatin, can easily be produced by a more energetic action on indican.

We are not yet in a position to give you any positive proofs of the correctness of our views, and we are afraid that the analytical researches to establish this end will probably present great difficulties; but it is not improbable that synthetic methods may clear up and establish this point, and we may say that preliminary experiments seem to justify our hypothesis.

The assumption that indican is the glucoside of indoxyl is also very probable from physiological considerations. The formula proposed is analogous to that of indoxylsulphuric acid, *viz.*, a sulphuric acid ether of indoxyl, which, as is well known, appears amongst the secretions of the animal organism, being most probably a product of the decomposition of albumenoid substances. It is, therefore, not improbable that certain species of plants decompose their albumen in a similar way, that the degradation product of albumen appears in the form of indoxyl, which is afterwards, so to say, neutralised by condensing with glucose—a process very often resorted to by plants in order to remove useless products, which appear as final stages of transformation of matter in their organism.

We venture to think that this explanation is the more probable, as it has been shown that plant and animal organisms very often do work up their albumen on analogous lines; they produce, for instance, from albumen substances like phylloporphyrin and haematoporphyrin, which are chemically closely allied.

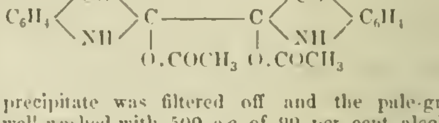
## II.

In the proceedings of the Manchester Literary and Philosophical Society, 1892, vol. V., O'Neill describes a crystalline substance obtained by the oxidation of indigo suspended in glacial acetic acid, and subsequent hydrolysis of the product so obtained, with caustic soda. The substance mentioned separated from the alkaline solution on the addition of an acid, *viz.* hydrochloric acid.

He states that this substance possesses an acid character, and that its formula is $C_{32}H_{28}N_4O_{12}$. It has, however, been shown (L. Marchlewski, "Memoirs of the Academy of Science in Cracow, 1897") that this is incorrect; that the formula representing the molecular weight of this interesting acid is, when air-dried, only $C_{16}H_{14}N_2O_6$, which is the doubled formula of ordinary isatic acid. Hence it was proposed to call O'Neill's acid, di-isatic acid. Subsequently we undertook jointly to further investigate the nature of di-isatic acid, and, if possible, to clear up its constitution. We have not, however, succeeded in doing this as yet, but, still, have ascertained a few facts which may have general interest.

We prepared the di-isatic acid according to O'Neill's method, only slight alterations being made.

The indigo, 100 grms. was mixed in a strong dish with from 1,500 to 2,000 grms. of glacial acetic acid, and to this cold mixture was added slowly, through a fine sieve, 25 grms. of permanagnate of potash, the whole being vigorously stirred during the process. After the expiration of about half an hour the indigo was oxidised to a pale-green micro-crystalline substance, which possibly possesses the formula—



This precipitate was filtered off and the pale-green residue well washed with 500 c.c. of 90 per cent. alcohol, containing a little acetic acid. It was dried on a porous plate in the air at the ordinary temperature, or at a gentle heat. The yield is that 100 grms. of 85 per cent. of indigo gave about 145 grms. of the air-dried acetyl compound.

The preparation of O'Neill's acid from this acetyl compound was carried out in the following manner:—20 grms. of the acetyl compound were treated in the cold with 200 c.c. of caustic soda, containing 80 grms. per litre. The mixture was allowed to stand for 24 hours, with occasional shaking; then filtered, and the filtrate acidulated strongly with hydrochloric acid. The O'Niell acid is precipitated as a curdy yellow precipitate, which becomes hard and crystalline on standing. This was filtered off and the residue extracted with boiling water. On cooling the liquors the O'Neill acid crystallises out, and after several recrystallisations from boiling water with the aid of animal charcoal, it is obtained in colourless crystals.

The crystals dissolve freely in cold methyl and ethyl alcohols and acetone; are practically insoluble in chloroform, carbon, disulphide, and ether.

This acid contains water of crystallisation, and, on heating to 140—149° C., appears to soften, probably due to the loss of water; but it does not fuse until 226—227° C., when it becomes dark-brown, and is evidently decomposed. The yield is about 10 per cent. on the weight of the acetyl compound used. This small yield is accounted for by the fact that a large quantity of indigo is regenerated from the acetyl compound by the treatment with caustic soda.

O'Neill had already made elementary combustions of this acid and found, as stated above, that its composition corresponds exactly to that of the hypothetical isatic acid. Analogous results were obtained and published by one of us (*loc. cit.*), in this case, the acid used was in its air-dried form.

However, the results we have obtained point out that the acid in reality represents a more complex body than was formerly supposed to be the case, and that it crystallises with water, so that combustions of differently dried samples lead to different formulæ. For instance, a sample dried at 110—115° C. was found to lose 8·17 per cent. of water, and the combustion of this dry acid gave the following figures:—

Took 0·0967 grm., gave 0·2298 grm. $CO_2$,
0·0434 grm. $H_2O$,

and these correspond to—

C ............................64·81 per cent.
H ............................ 4·98 „

For the estimation of nitrogen—
Took 0·1500 grm., gave 12·9 c.c. of moist N at 736·7 and 14° C.; this corresponds to—

N ............................9·87 per cent.

The values do not agree with the formula $C_{16}H_{14}N_2O_6$, the percentage composition of which is—

C ..........................58·18 per cent.
H .......................... 4·24 „
N .......................... 8·48 „

It will be seen, however, that the results obtained will agree with the original empirical formula proposed by O'Neill, and subsequently confirmed by one of us, if we assume that di-isatic acid loses nearly two molecules of water in the drying process at 110—115°, being converted in an anhydrous acid of the formula $C_{16}H_{20}N_2O_4$, for which the following values are calculated :—

C ..........................65·31 per cent.
H .......................... 3·40 „
N .......................... 9·52 „

According to this assumption, the amount of water lost during drying should be 10·91 per cent. It will be seen that these calculated values and those obtained experimentally agree passably ; the slight existent differences may be due to the incomplete elimination of water from the samples. We tried, of course, to desiccate the acid at even higher temperatures, but found that the prolonged action of an elevated temperature caused a partial decomposition of the substance.

We have succeeded in proving the correctness of our assumption (*i.e.*, that di-isatic acid is really di-isatin (crystallising with two molecules of water) in another manner.

During the re-crystallisation of the acid it was noticed that a portion of the acid became practically insoluble and lost its crystalline form, being converted into a white powder. It was thought that this powder might be the same acid free from water of crystallisation. The powder was air-dried on a porous tile, and then a portion of this dried at 120° C. for six hours. The loss was only 0·15 per cent., so that it evidently contained no water of crystallisation as does the ordinary crystallised acid. This anhydrous substance possesses an acid character similar to the crystallised hydrated acid, and by acidifying its alkaline solution we obtained a crystalline precipitate, which, after re-crystallisation from water, possessed all the properties of the hydrated acid.

Moreover, we made quantitative titrations of the anhydrous acid and found that it neutralised relatively more alkali than the crystallised acid, which has already been shown (L. Marchlewski, *loc. cit.*) to be a monobasic acid, and that the values obtained in the first case point to the formula $C_{16}H_9NaN_2O_4$, and in the second to $C_{16}H_{13}NaN_2O_6$.

### *Titration of the Anhydrous Acid.*

0·5 grm. of this substance was dissolved in 25 c.c. decinormal NaOH in the cold, and titrated back with decinormal $H_2SO_4$ ; it was found that 0·5 grm. of the anhydrous acid had neutralised 17·3 c.c. of decinormal NaOH, thus 100 grms. of this acid would neutralise 7·95 grms. of Na.

In another experiment, in which the acid was boiled with the caustic soda for 15 minutes, then cooled and titrated back with the acid as usual, 100 grms. of the acid would neutralise 7·68 of Na, mean, 7·815 grms. of Na. If the reaction took place with the crystallised acid, according to the equations—

$$C_{16}H_{14}N_2O_6 + NaOH = C_{16}H_{13}NaN_2O_6 + H_2O$$

100 grms. of acid would neutralise 6·969 grms of Na. If the reaction took place with the dehydrated acid, *viz.*—

$$C_{16}H_{14}N_2O_6 - 2H_2O = C_{16}H_{10}N_2O_4$$

according to the equation—

$$C_{16}H_{10}N_2O_4 + NaOH = C_{16}H_9NaN_2O_1 + H_2O$$

100 grms. of acid would neutralise 7·82 grms. of Na. Found by experiment 7·815 grms. of Na.

### *Titration of the Crystallised Hydrated Acid.*

0·5 grm. of this substance was dissolved in 20 c.c. of NaOH in the cold, and titrated back with decinormal $NH_2SO_4$. It was found that 0·5 grm. of the titrated acid had neutralised 15·8 c.c. of decinormal NaOH, thus 100 grms. of the crystallised hydrated acid would neutralise 7·26 grms. of Na. According to the above equation, 6·969 grms. of Na.

The anhydrous acid (di-iastic) is a white powder, freely soluble in methyl and ethyl, alcohol, and acetone ; insoluble in chloroform, carbon disulphide, and ether.

The silver salt of the hydrated acid was made in the usual manner, *i.e.*, the acid was neutralised with ammonia and precipitated with silver nitrate, well washed and dried on a porous plate and then in a desiccator. This silver salt was found to contain 25·68 per cent. Ag.

If the formula of the silver salt was $C_{16}H_{11}AgN_2O_5$, *viz.*, $C_{16}H_{13}AgN_2O_6 - H_2O$, it would contain 25·77 per cent. Ag. The bromine compound of the crystallised acid was prepared in the following manner :—

About 0·7 grm. was dissolved in 40 c.c. of 50 per cent. acetic acid, and excess of bromine was dissolved in glacial acetic acid added, the mixture being kept at the ordinary temperature and allowed to stand for 48 hours. On pouring into water, a voluminous white precipitate was thrown down ; this was filtered off, thoroughly washed, and dried on a porous plate.

### *Estimation of the Bromine (Carius' Method).*

Took 0·1563 grm. gave 0·1195 AgBr., 32·5 per cent. Br.
„ 0·1610 „ „ 0·1215 „ 32·15 „ „
Mean, 32·32 per cent. Br.

The following bromine compounds are possible :—

$C_{16}H_{12}Br_2N_2O_6$ would contain 32·78 per cent. Br.
$C_{16}H_8Br_2N_2O_1$ „ „ 35·30 „ „
$C_{16}H_{11}Br_3N_2O_6$ „ „ 42·32 „ „
$C_{16}H_7Br_3N_2O_1$ „ „ 43·00 „ „

The formula of the above bromine compound is therefore $C_{16}H_{12}Br_2N_2O_6$.

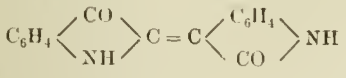
It is a white powder which melts slowly and evidently decomposed. It is freely soluble in cold methyl and ethyl alcohols, acetone, and ether ; insoluble in chloroform and carbon disulphide ; difficultly soluble in cold caustic soda, and soluble in hot sodium carbonate.

We consider the empirical formula and molecular weight of O'Niell's acid to be established, and our efforts will now be directed towards the solution of its constitution.

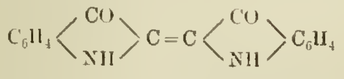
## III.

It is well known that doubt exists in the minds of many chemists as to whether the various artificial indigos are really identical in chemical constitution with natural indigo. The authors were of opinion that the above oxidation products might be utilised for deciding this point.

The indogenide of isatin, indirubin, has recently been reinvestigated by Schunck and Marchlewski (Ber. d. deutschen Chem. Ges., 1895, 539), who have shown that it has the constitution—



as compared with indigotin—



The authors thought that although indirubin and indigotin differ but slightly in their constitution, they might possibly show a considerable difference in their nearer oxidation products, and if this was the case then any similar difference in the constitution of artificial indigos from that of natural indigo might also be shown by the oxidation products being different from those of the natural product ; and if no such difference existed in the case or artificial indigos, and did in the case of indirubin, then the artificial indigos might be regarded as identical with the natural.

The artificial indigo from "Kalle salt," the recently introduced artificial indigo of the Bad. Anilin u. Soda Fabr., synthetic indirubin and almost chemically pure indigotin from natural indigo were studied.

The Kalle salt was converted into indigo blue by boiling with caustic soda, then filtered, and the blue thoroughly washed with boiling water until free from alkali. Took for the experiments 2·5 grms. of indirubin, B.A.S.F. indigo, Kalle indigo, and natural indigo, and mixed each with 40 grms. glacial acetic acid, and 0·8 grms. of finely powdered and sieved $KMnO_4$. The oxidations were carried out in flasks side by side. The B.A.S.F. and Kalle indigos seemed to oxidise the quickest, but the rubin appeared only to become redder. After all the samples had stood for one hour they were filtered. The oxidation products of the three blues seemed to have the usual appearance, *viz.*, a greenish crystalline mass; the rubin was not appreciably altered, though the filtrate was as brown as in the other cases. The residues were allowed to dry in the air. Took 1·5 grms. of each of the four air-dried acetyl oxidation products and treated with 15 c.c. of 2N caustic soda; allowed to stand as usual with occasional shaking for two hours; filtered off the regenerated indigo and acidified the filtrated with hydrochloric acid; allowed to stand for the precipitates to harden; and then filtered off. The three blues yielded the usual crystalline precipitate in fair quantity, but a mere trace of precipitate of a quite different appearance was produced from the indirubin. From this we conclude that indirubin is not affected in the manner as indigotin, either natural or synthetic—which latter behave in a manner quite identical; and that the artificial and natural indigotins may be considered to be identical.

Whilst studying the comparative oxidations of indigotin and indirubin it occurred to us that we might decide the question as to whether indirubin became converted entirely or partially into indigotin during an alkaline reduction process. It is well known that acid-reducing agents convert indirubin entirely into indigotin. With this end in view the following experiments were made:—

Synthetic indirubin and crystallised natural indirubin were reduced at 70—75° C. with alkaline hydrosulphite; both reduced with the greatest ease. To ascertain the point at which the conversion took place (if at all), small quantities were filtered of each and drops allowed to oxidise on filter paper. At the end of four hours no appreciable conversion could be detected; at the end of 24 hours both were filtered; and small quantities of cotton wool soaked in the liquors. The wool was removed after about 10 minutes, and allowed to oxidise in the air. After some hours it had assumed in each case a fine, reddish purple blue. These samples of cotton wool were then extracted with boiling absolute alcohol, and it was found that the whole of the colour was removed from the fibre, and on carefully examining the now white wool no traces of indigotin could be detected. However, the liquors themselves were next oxidised by bubbling a slow stream of oxygen through them, and the precipitated colours filtered off and thoroughly washed. Each precipitate was then extracted with hot alcohol to remove the indirubin, and it was found that both the regenerated synthetic and natural indirubins left a very small quantity of indigotin—exceedingly small in the case of the chemically pure natural indirubin (kindly presented to us by Dr. Schunck). It is, then, certain that a slight conversion does take place. The experiments were made, in this case, both on the synthetic indirubin and the natural indirubin, because recently an attempt was made to throw doubt both on the constitution of indirubin and on the identity of the natural indirubin with the synthetic compound (*vide* Bucher, Chem. Zeit., No. 97, 1897; Marchlewski, Chem. Zeit., No. 103, 1898), so it was thought better to make the experiments on both, only to find in the result further proof of their identity. We have moreover, investigated the action of "the glucose reduction" process on indirubin in three experiments. Synthetic indirubin was used and was compared with refined natural indigo. We took 0·4 grm. of indirubin and indigotin and the requisite quantity of reducing caustic paste (1 part of colour in 200 of the paste). Both were put through exactly the practical conditions of working on a large scale. The indirubin yields a purple shade which, on boiling with alcohol, lost the purple colour and yielded a light *blue* stripe. Here again, then, there was a partial conversion into indigotin.

It will here be interesting to mention one or two experiments made with a view of ascertaining the relative fastness of indigotin and indirubin. The printed samples were soaped at 60°—70° C. for 10 minutes, when it was found that the indirubin purple shade had lost much more than the indigotin, this indicating that indirubin was not so fast to washing as indigotin. We also tried the effects of discharging with the potassium chromate method, and have found that the indirubin is not so easily discharged as indigotin. We desire to acknowledge our indebtedness to Mr. J. Rhodes, who kindly carried out the printing trials on a works scale.

We think, therefore, that we have shown that the various artificial indigos are identical in constitution with natural indigo, and that the conversion of indirubin into indigotin is not readily brought about by alkaline reducing agents. On the other hand, the quantities of indirubin present in natural indigo are very small, and in the face of the convertability of indirubin into its blue isomer, it is to be doubted whether one is entitled to ascribe to indirubin such an important rôle as is so frequently done.

The above researches were carried out partly at the research laboratory of Messrs. Claus and Rée, Clayton, Manchester, and partly at the laboratory of the Manchester Technical School.

In conclusion we desire to express our thanks to Mr. S. Sinnatt for his assistance in some portions of the above work.

### DISCUSSION.

Dr. SCHUNCK, F.R.S., said he might be considered not only the father but the godfather of the substance treated of by the authors of the paper, and therefore he might be expected to say a few words on the subject. The paper confirmed the results of his own researches, which were published more than forty years ago. During that time great progress had been made in chemical science, and the views of chemists at the present day must therefore be very different to what they were then. In his opinion there was a very high degree of probability in the views set forth by the authors of the paper as regards the constitution of indican, and they would explain many of the facts which had so far remained unexplained. At the same time it must be confessed their view was entirely hypothetical, inasmuch as no one had obtained glucose and indoxyl from indican. Any hypothesis set up to explain certain facts must explain them clearly and without any doubt. Such doubts as still remained could only be cleared up by a thorough re-examination of the subject. It was not within his power to take up the matter again, but he considered it a promising subject for any younger chemist to take up. At the same time he would tell anyone doing so that the subject would be found a very difficult one, on account of the fugitive character of the substance or substances to be dealt with. If a compound were to be discovered which, on treatment with acids, should be split up into indoxyl and glucose, it would still remain open to doubt whether the substance was strictly identical with indican or not.

Dr. GERLAND endorsed the statement made in the paper that indigo-red was very readily converted into indigo-blue, and he believed this had been previously proved by Dr. Lehne.

Dr. MARCHLEWSKI, in reply to Dr. Schunck, called attention to the researches of Van Lookeren Campagne, who claimed to have proved that the glucose like sugar, originally called by Dr. Schunck indiglucin, was identical with glucose. These researches led him (Dr. Marchlewski) to believe that indican was a true glucoside, and that consequently the constitution of indican, so far as the constitution of the glucose radical contained in it was concerned, might be considered settled. He admitted that the statement that the indigotin-producing group of indican was indoxyl, was purely hypothetical, but at the same time he believed that only the proposed hypothesis explained satisfactorily the results of Dr. Schunck's fundamental researches.

## THE VAPOURS FROM HEATED SULPHURIC ACID.

BY HERBERT N. MORRIS.

Of late years many processes for the concentration of sulphuric acid up to 94—98 per cent. monohydrate have been introduced, based upon the principle of surface evaporation, as distinct from distillation. By this I mean, that instead of fractionally distilling the acid, as is done, for example, in platinum stills, the acid is heated in open vessels placed directly or indirectly in a fire flue; and the vapours from the vessels are exposed more or less to the flue gases or the atmospheric air.

Amongst such processes I may mention those of Garroway, Negrier, Webb, Veitch, Levinstein, Dyson, &c., some of which have come into very considerable use, especially in this country.

In an experience of several years, working one of these processes (Levinstein's), I have made a number of observations, some of which I think are worthy of bringing to the notice of this Society. With the kind permission of Mr. Levinstein, at whose works the experiments have been made, I beg to bring before you the following:—

I have noticed, and others have had similar experience, that in concentrating by the evaporation process, and in particular in concentrating to a high percentage of monohydrate, a quantity of white vapours are given off, which do not necessarily contain a large amount of acid, but which are undoubtedly difficult to absorb or condense.

I have seen the vapours from one concentrating apparatus (bringing 30 tons per week of acid, sp. gr. 1·75 up to 95 per cent. monohydrate) cooled to 15° C., through a series of pipes immersed in cold water, then passed through a coke scrubber tower moistened with water; emerging from the tower into the chimney, white like steam, scarcely any visible change having taken place by the treatment. It is hardly necessary to say that the acidity in the vapour was finally very minute.

### *The Formation of the White Vapours.*

In concentrating dilute sulphuric acid up to 96 per cent. monohydrate by heating in a *closed* retort connected with a condenser, practically no white fumes are evolved. All the fumes are easily condensed by cooling.

If, during the concentration of the acid, *dry* air be passed very slowly over the surface of the liquid in the retort, little or no effect is seen, but a rapid stream of air causes immediate evolution of dense white fumes, which do not disappear in the condenser, but pass in white volumes into the atmosphere. Moist air has the same effect as dry, *viz.*, very slow current no effect, a rapid current dense, white fumes. Carbon dioxide has the same effect as air.

The following experiment is worth referring to in detail:—

806 grms. of acid, sp. gr. 1·62, were placed in a retort connected with a long condenser, attached to a glass receiver, from which a vertical tube was carried to the top of the room. The retort was gradually heated and *dry* air was passed over the surface of the acid. At as low a temperature as 135° C. slight white fumes came out of the end of the apparatus into the atmosphere, but none were visible in the retort. At 170° white fumes appeared in the retort and passed through into the air. At 200° dense white fumes filled the retort, the condenser, receiver, and outlet tube, and passed into the air. The temperature was maintained at 200° for 3½ hours, dry air being passed over the acid during the whole of the time. The vapour was quite cold in the receiver. The vapour escaping into the air in the earlier part of the experiment scarcely affected moist litmus paper. A piece of neutral paper immersed in the fumes turned red after five minutes. At the end of the experiment the acid in the retort was found to contain 94·9 per cent. monohydrate. The total loss by volatilisation was 8·53 per cent., of which, 1·35 per cent. had been condensed in the apparatus, the remainder passing into the air.

It is interesting to note from this experiment that sulphuric acid may be concentrated to 95 per cent. at as low a temperature as 200° C., possibly lower, but owing to the difficulty of condensing or utilising the fumes evolved, it is at present impracticable.

If fuming sulphuric acid, *e.g.*, acid containing 40 per cent. sulphuric anhydride be melted in a retort and a current of dry air be passed over the surface, some volatilisation takes place but no white fumes are formed. If, however, the air contain moisture, dense white fumes are formed which behave in every way like the white fumes above referred to, from the concentration of sulphuric acid, and I have taken them to be identical.

I am not aware that anything has been published showing the exact composition or condition of existence of this vapour. No definite compound or mixture seems to have been detected and examined, but it is generally assumed that sulphuric acid is decomposed or dissociated by heat into sulphuric anhydride and water. Supposing our vapour to be a mixture or compound of $SO_3$ and $H_2O$, a re-combination might be expected to take place on cooling, which appears to be the case when the acid is heated in a vessel out of contact with air or other gas.

I cannot now enter into a discussion on the probable constitution of this vapour. A number of experiments have been made, and I hope to be able to bring the subject up during the next session and deal with that point more in detail.

The question of great importance to manufacturers is:

### *How can these White Vapours be efficiently Absorbed or Condensed.*

I have already referred to one instance of an attempt to deal with them by cooling and water absorption.

In the numerous experiments I have made, the fumes obtained by passing moist air over fuming sulphuric acid and those obtained from the evaporation of sulphuric acid on a large scale have been taken as identical.

The white vapour after cooling has been passed in a thin stream through water, sulphuric acid of various strengths, caustic soda solution, sodium carbonate, lime water, ammonia, &c., also through tubes or bottles packed with lumps of the solids of several of the above. In all these cases little or no visible effect was produced.

It may be suggested that I passed too rapidly through the various absorbents, and that the bubbles of vapour were not allowed to come sufficiently in contact with them. I readily admit this, but if the rate of passage be too slow, we might just as well pass the vapour into an empty chamber or vessel and allow it time to condense, *i.e.*, to have surface condensation only, not absorption. Ten gallons of the vapour were aspirated into a glass carboy and allowed to stand. Condensation gradually took place and in a half to three-quarters of an hour appeared to be complete. On shaking up with a solution of alkali or hydrogen peroxide the acidity was determined.

Again, a thin stream of the white vapour was brought to meet a stream of ammonia gas, so arranged that the vapours impinged directly upon one another in a large glass receiver, and thence passed through two 4-litre flasks containing a small quantity of water, then through a long condenser, and finally out at the end of a long vertical tube into the atmosphere. A small quantity of a white deposit of ammonium sulphate was formed, but the white fumes passed on into the atmosphere, even when a large excess of ammonia vapour was present.

The fumes were passed rapidly through a 10-per cent. solution of barium chloride. They were not visibly affected, and only after 15 minutes did the solution become appreciably turbid.

Other experiments made on a large scale by passing the vapour through a tower packed with coke, showed that water had little effect, but that steam reduced the acidity, but not the whiteness, to a minimum.

Some other curious facts connected with this vapour were noticed. On a large scale the fumes from the concentrating apparatus pass through a circular pipe, 12 ins. diameter. I have found that if the gas be aspirated rapidly into a receiver and allowed to condense, a certain amount of acidity is found. If, however, another sample of the gas be aspirated *slowly* from the same place, either at the same time or immediately before or after, a much smaller acidity is found. This has been proved conclusively by numerous experiments, *viz.*, quick aspiration of the gas gives more acidity than slow aspiration. The following are some figures obtained :—

| Slow Aspiration. | Quick Aspiration. |
|---|---|
| Grains $SO_3$ per cub. ft. | Grains $SO_3$ per cub. ft. |
| 0·62 | 1·26 |
| 0·70 | 1·11 |
| 1·86 | 2·64 |
| 0·71 | 1·17 |

There appears to be also a difference in the acidity of the gas drawn from the centre of the flue and that drawn from near the circumference if the aspiration be slow. If, however, the aspiration be rapid there is no appreciable difference.

Whether these properties are peculiar to this gas or apply to other mixed gases I am not prepared to say; but the results are undoubtedly important to those who have these fumes to deal with, and whose exits are under Government control.

From the results above recorded, two conclusions may be drawn :—

1. That in order to prevent the formation of the white fumes, both the atmospheric air and the furnace gases should be entirely excluded from contact with the vapours from evaporation.

2. That the simplest method of completely condensing the fumes when once formed is to pass into one or more large chambers and allow time for condensation.

As in any of the processes for concentrating sulphuric acid on the evaporation principle it is practically impossible to exclude every trace of air or of furnace gases, the formation of white fumes cannot be entirely prevented. To reduce the acidity to a minimum it is only necessary to pass through a scrubber tower moistened with steam. This does not, however, get rid of the white fumes which pass into the chimney as a *difficultly condensible steam*, and to the uninitiated inhabitant of the neighbourhood is an imaginary source of nuisance.

It would be impracticable to erect large chambers in which the vapours might have space and time to condense. The cost would be out of all proportion to the benefit to be derived from the saving of the small quantity of acid.

As an experiment on a large scale I have tried the following :—

Both air and furnace gases were excluded as much as possible and the vapours then drawn, by means of a fan or blower, into the first or leading sulphuric acid chamber of the series, supplying in this way part of the steam necessary for the process, and allowing the space of the whole chamber system in which to condense.

The vapours from two concentrating plants (each boiling about 20 tons of 75 per cent. acid to 95 per cent. per week) were passed into the first of a series of chambers for more than two months without materially affecting the working process, and I should have continued the experiment had the fan been able to resist the action of the damp acid vapours. This is a point in which serious difficulty lies, together with the means of exclusion of foreign gases, and the matter, therefore, still remains in an experimental stage.

In conclusion I wish to express thanks to my assistant, Mr. W. Barlow, A.I.C., by whom the details of many of the experiments have been carried out.

## New York Section.

*Meeting held at the College of Pharmacy,*
*on Friday, April 22nd, 1898.*

PROF. CHAS. F. CHANDLER IN THE CHAIR.

## PROCESS FOR THE PRODUCTION OF PHOTOGRAPHIC PRINTS ON TEXTILE FABRICS BY MEANS OF ADJECTIVE COLOURS.

BY STEWART F. CARTER.

Probably the first experiments made in the production of photographic prints on vegetable fabrics, by means of dyestuffs, were those suggested by the characteristic sensitiveness to actinic light of certain substantive colours, when in the diazotised state; notably primuline.

In this case, the fabric was first dyed with primuline, diazotised and exposed under a negative, the print being then developed by passing through suitable solutions of of phenols or amines, such as alpha and betanaphtol, metaphenylene diamine, etc. according to the shade desired.

More recently the observation of E. Kopp, that fabrics prepared with "Kalles indigo salt" (ortho nitro phenyllactoketone) and exposed to light, no longer gave a blue on treatment with caustic soda, led to the utilisation of this fact, in experimental production of blue prints on cotton or linen fabrics.

So far, however, there seems to be no record of any attempts to utilise the fast adjective colours in this direction.

The following process, which suggested itself to me in October 1894, and gave satisfactory results on the first trial, will doubtless be of interest from both a photographic and tintorial point of view :—

As a basis for obtaining the necessary mordant the ferroprussiate process, with certain additions is used.

A fine cotton or linen fabric, perfectly bleached, is treated with the following solution and then dried in subdued light :—

| | | |
|---|---|---|
| *a.* | Water, 200 c.c. ......................<br>Citrate of iron and ammonia, 75 grms.. | yielding<br>240 c.c.<br>sp. gr. 1·1525 |
| *b.* | Water, 200 c.c. ......................<br>Potassium ferricyanide, 75 grms...... | yielding<br>235 c.c.<br>sp. gr. 1·160. |

Equal parts of *a* and *b* are mixed together when required for use, and the fabric treated as above. It is then exposed under an ordinary negative, and after sufficient exposure has been given the print is well washed in cold water, when the ordinary "ferro-prussiate" or "blue' print," is produced.

The print is now worked in a cold bath of—

Water, 1 litre,
Caustic soda (sp. gr., 1·350), 2·5 c.c.,

until the blue is quite decomposed and the resulting ferric hydroxide precipitated on the fabric. It is now washed well in hot water to free from the sodium ferrocyanide formed and then entered in a second fixing bath of—

Water, 1 litre.
Sodium phosphate, 3 grms.

in which it is worked at a temperature of 170° F. for about three minutes. Then follows a good washing, first in cold and then in hot water (160° F.), and, after rinsing in cold water, the print is ready for dyeing.

The dye bath depends on the colour which is desired to be produced, and consists of one of the so called alizarine colours, the choice being those which admit of the production of good "whites" on the unmordanted portions of the print and fast and pleasing shades.

For general purposes the best and most striking results are obtained with resorcine green (dinitro resorcine).

The dye bath is composed of—

3 to 5 grms. of Resorcine Green,
1 litre of water.
5 c. c. of glue size

or an equivalent quantity of gelatine previously dissolved.

The bath is heated to about 160° F., the glue size added, and the print worked in it for two or three minutes, then the dyestuff is added and the bath gradually brought up to about 180° F., the print being kept in motion all the time. Dyeing proceeds rapidly, and when the shade is considered full enough to show a strong picture, the fabric is removed from the bath and well washed in boiling water, till all loose dye is removed. It is next worked in a bath of neutral soap at 160° F., till the white portions show up pure, then hot-watered again, and finally rinsed in cold water and dried, preferably by placing between folds of clean cotton and passing a hot iron over.

The print so produced is fast to light and washing.

Whilst, as already stated, Resorcine Green is perhaps the most suitable and pleasing dye to use, other dyes may be employed. Gallocyanine, for instance, gives violet and blue shade; alizarine gives the well-known and extremely fast purple; by means of alizarin brown very pleasing sepia effects are obtained; and so on, according to the desired end in view, all the prints being fast to light and washing. Of course, if desired, natural dyestuffs, such as logwood, might be employed, but the alizarine colours give the most striking and permanent results.

The fixing bath of sodium phosphate causes the dyed shade to come up with good bloom, and without this treatment I have usually found the resulting shades to be more or less flat.

The addition of glue and gelatine to the dye bath has the effect of keeping the white portions of the print from becoming soiled in dyeing. A fact sufficiently well known to all acquainted with the arts of dyeing and calico printing.

### Discussion.

Mr. M. Toch said that this process or rather a process similar to this was published by him in the "Photographic Times Annual" of 1895, at which time he thought that work of this character was original with him, but he now saw, from the paper just read, that similar work was done in 1894. He said his own process was something different, and rather difficult to work. He proceeded on the theory that bi-chromate of potash was sensitive to light, and at the same time formed lake compounds with the wood extracts. He made solutions from the extracts of hypernic, logwood, quercitron, &c., and sensitised fabrics and paper with these mixtures and bichromate, and then printed in the usual way, after which the prints were washed, with good permanent results. He published the whole process at some length, but found, from the numerous inquiries received that it was difficult to impart the process to others unless the operation were optically demonstrated. There was some uncertainty in the result if the water used were too hot or the wood extracts too old, and so he abandoned the subject after a while, on finding collotype printing cheaper and better.

Mr. Alfred J. Cohn said that the process seemed like the primulin process described a number of years ago, whereby prints were obtained in various colours by the reaction yielded by primulin with various phenols, resorcin, eikonogen, &c., the colours ranging from yellow to deep black. Some years ago he had experimented in the preparation of a black print paper that would yield a positive from a positive, and that would be developed by simple washing in water. The problem presented was to prepare an aqueous solution capable of being applied to paper, and which would be decomposed by light, but which would be blackened by contact with water. He solved the problem with a solution containing ferric chloride, oxalic acid, hydrochloric acid, sodium chloride, tannin and some gum. Paper coated with this solution had a yellow colour, which was bleached to a white by sunlight, but where protected, retained its yellow colour, and on washing yielded very sharp black prints. The prepared paper would not keep well for any length of time, soon becoming brittle. Moreover, the background, originally white, was apt to acquire a purplish colour in time, due probably to a reaction between the ferrous oxalate, resulting from the decomposition by light with the tannin present. The oxalic acid seemed to have the power of preventing the reaction between the ferric chloride and the tannin, or at least of retaining it in so loose a condition that the mere addition of water was quite sufficient to upset the unstable compound and yield a black, inky colour.

Dr. Schweitzer said that the production of pictures on primulin was based on the principle that primulin when diazotized was very sensitive to light, and wherever the light affected it the diazo compound was decomposed, and could not develop any more colours when combined with a phenol or amine. But in this process the mordant was decomposed and, when the fabrics were subsequently immersed in the dye bath, the dye did not attach itself on these parts on which the mordant had been destroyed.

## SIMPLE QUANTITATIVE DETERMINATION OF DIASTATIC POWER.

BY JOKICHI TAKAMINE.

A simple and quick quantitative determination of diastatic power is of considerable importance in the various industries such as in the manufacture of alcohol and alcoholic beverages; especially when diastatic substances are used in a green condition, such as green malt and green taka-koji, a quick method of determination becomes imperative.

There are various methods of determining diastatic power in use, such as Lintner's, Junk's, O'Sullivan's, and others. While each method has its merits, yet, on the whole, they are complicated and require trained hands, and take a long time in their execution. The method I propose is one that I have been daily using with good results in the distillery, &c., where diastatic substances are used in green condition and quick method only is practical. While this is probably not as accurate as some of the known methods, yet it is delicate enough for all practical purposes, in fact it is remarkably accurate compared for its simplicity.

This method firstly, is based upon the remarkable stability of the diastatic power of taka-diastase. A sample of taka-diastase probably over four years ago was tested from time to time up to the present by Junk's test, and its diastatic power was found practically permanent and stable. Secondly, this method is based upon the fact that (within certain limits) the speed of the diastatic transformation of starch paste is directly proportional to the quantity of the diastatic substance used. Or, in other words, time taken for diastatic transformation varies inversely as the quantity of enzine.

The first step in the method consists in standardising or determining the diastatic power of a given sample of taka-diastase by a known established method once for all; or still better prepare a batch of taka-diastase whose diastatic strength represents an even number of diastatic units by known method, such as say 300 or 500 times its own weight of starch in thirty minutes according to modified Junk's test (modified Junk's, Bull. of Pharm., 52—54, Feb. 1898), or say 300 or 500 diastatic units by Lintner's test (Lintner's J. für Prakt. Chem. (2), **34**, 378—394). The second step in the method consists in making a color; metric scale of diastatic strength, which is done by allowing different volumes of solution of taka-diastase of known strength to act upon equal quantity of starch paste and comparing the diastatic power of the substances to be tested to the scale thus formed by the colorimetric indications produced by the action of iodine solution upon the converted starch paste.

The diastatic power thus comparatively tested can be expressed by simple calculation in the terms of standardised taka-diastase, or Lintner's or Junk's terms, or any other terms that may be desired.

### *Standard Taka-Diastase Solution.*

Make 1 per cent. solution of standardised taka-diastase, dissolve 1 grm. of standardised taka diastase in 100 c.c. of water. (Fresh solution ought to be used).

### *Starch Solution.*

Make 5 per cent. solution of neutral potato starch; boil 800 c.c. of water (distilled or rain) in a suitable wide mouth vessel. Into the water pour the milk of starch made by the mixing of 50 grms. of starch with 200 c.c. of water, and boil for two minutes.

### *Iodine Solution.*

Take 1 grm. of iodine and 2 grms. of potassium iodide. Put the two in a beaker, add a little water, say 5 c.c., and after the solution dilute to 250 c.c.

### *Apparatus required.*

(1.) Quart glazed bucket.
(2.) One shallow tin pan 2 ins. deep, 8 ins. in diameter.
(3.) Two graduated $\frac{1}{10}$ c.c. pipettes.
(4.) Eight glasses of about 150 c.c. each, with file-mark at 100 c.c.
(5.) Ten strong test tubes.
(6.) A measure of 100 c.c.
(7.) Two white plates.

### *The Materials to be Tested.*

If it be thick liquid such as malt extract, dilute a known weight of it with known volume of water, so that it can be measured by pippette. If it is solid, such as ground malt, steep the known weight in known volume of water, and take known volume of the extract for examination. Or known weight, say one half to one gramme of solid material, may be taken alone, with a small quantity of water.

It is essential to know or guess approximately the diastatic strength of the substance to be tested, so as to regulate its degree of solution in order that its diastatic power compares somewhere within the scope of the scale of comparison.

### *Process of Testing.*

Pour into each of the eight glasses the hot starch paste up to the 100 c.c. mark. Place them side by side in shallow pan of warm water of a temperature varying from 40°—60° C. (according to the article to be tested), in the form of a circle. From the pipette add say 10 c.c. of solution to be tested to the first glass, on top of the starch after cooling to a proper temperature. Then in quick succession, add taka-diastase solution in the following proportions to each of the seven other glasses:—

| | C.c. |
|---|---|
| Into the 2nd glass | 1 |
| ,, 3rd ,, | 2 |
| ,, 4th ,, | 3 |
| ,, 5th ,, | 4 |
| ,, 6th ,, | 5 |
| ,, 7th ,, | 6 |
| ,, 8th ,, | 7 |

Then each glass is stirred up with the test tube as a stirring rod in quick succession. Keep stirring them, one after another, until the starch paste all turns limpid.

At this stage it will be observed that the stronger the diastatic power the quicker the liquifaction of the paste.

When the contents of the glasses become liquefied, take out of each glass in succession a drop of the liquid by means of the stirring test tube, and drop them on a white, dry dinner plate, in the order of the glasses. When there are eight drops of equal size on the plate drop on each drop a drop of iodine solution. Then with your finger spread each drop on the plate to the size of about a silver dollar. The drops from the second to the eight glass will form a colorimetric scale of colours ranging from blue to purple and reddish brown. Observe which portion of the colorimetric scale that of the substance to be tested corresponds to. By repeating this iodine test two or three times in succession within the first ten minutes one can easily determine which portion of the scale the coloration by the substance to be tested corresponds to. Suppose the colour corresponds somewhere between the third and the fourth, and then we can assume it at 3·5, and calculate the diastatic strength in the terms of the starch converted or the sugar formed; or, if further accuracy of the test is desired, a scale of starch glasses containing taka-diastase solution of 3 c.c., 3·0 c.c., 3·4 c.c., 3·6 c.c., 3·8 c.c., 4 c.c., may be put up and compared with 10 c.c. of the given solution in the same manner. In this way more accurate results may be obtained. Instead of having only one specimen at a time several samples of substances may be tested at once.

When great accuracy is not required the following modified method will be found convenient:—

Into each of a dozen test tubes of about 20 c.c. capacity, pour in from a graduated pipette 15 c.c. of water. Make a fine mark on the tubes for that quantity. After draining the water from the tubes, fill each tube to the mark with starch paste while hot. This is conveniently done by sucking up the starch paste into a long glass tube (say 12 ins. long and $\frac{1}{2}$ in. inside diameter) narrowing at its lower end, from which allow the hot starch paste to drop into the test tubes. Several dozen of these test tubes may be filled at one time with starch, as they will keep for at least a couple of days. These test tubes are put in a row on the test tube stand. Into the first tube a known number of drops of the substance to be tested is added. Into the second and succeeding number of tubes and increasing numbers of drops of taka-diastase solution. And then shake each tube vigorously one after the other in quick succession (two tubes can be shaken at a time.

When the contents of each tube are liquefied take out of each tube with a rubber-headed dropper (one drop of uniform size from each tube) one drop, which put into the white plate in the order of the tubes shaken. And carry out the iodine test in the same manner as above-mentioned.

By selecting droppers with uniform tips a fairly accurate result can be obtained and will answer for most purposes.

The iodine tests now in use, such as Junk's, are based upon a chronic point at a given time and a given temperature. These three points must be closely observed to get good results, while in this method mere comparison of colour is absolutely necessary, which can be done by anybody except one colour-blind.

### DISCUSSION.

Mr. SAARBACH said that the process was based on the proposition that the diastatic power was inversely proportional to the time of action; but this proposition was only true so long as some starch in excess was present, and the inversion had not gone so far that the amount of maltose formed was proportionally not too large. Had the author fixed for his comparative test a certain time limit for the action of his diastase, as the test might give varying results when the time was prolonged?

Mr. J. TAKAMINE in answer to Mr. Saarbach replied that he had mentioned in his paper that this test was to be observed within about ten minutes from the start of the experiment. If longer time for the colorimetric observation were required, a larger excess of starch should be used, or the same result could be obtained by using a smaller quantity of diastatic substance. Another way of prolonging the duration of the colorimetric re-action was to cary out the test at a lower temperature, say at the ordinary temperature. He had chosen a 10—15 minutes' test, for that was about the duration of the conversion allowed in the cookers at an alcohol distillery. By taking 200 c.c. of 5 per cent. starch solution instead of 100 c.c., the time of re-action could be prolonged to about 30 minutes, which gave ample time for the close observation of the test.

## ON THE TECHNICAL ANALYSIS OF ASPHALTUM AND ASPHALTES.

BY S. F. PECKHAM.

FOR the purposes of this paper I include Trinidad pitch—parianite—with the bituminous rocks or asphaltes.

The object of the technical analysis of solid bituminous minerals is not to determine the ultimate chemical composition of any of the complex compounds that may be mixed together to form the material under consideration, hence the method employed is more or less empirical. It may be safely assumed that the bitumen present in the specimen has a specific gravity of about 1, hence the specific gravity of the specimen will vary with the amount of mineral matter present. It is of some importance to learn the behaviour of the specimen between a temperature of zero and 212° F.; that is, to ascertain whether the specimen becomes very brittle at low temperatures, or very soft at high temperatures. Flowing tests depend entirely upon the standard specimen selected, and are of no value except when applied to different specimens of the same variety of material in comparison with each other.

The determination of the petroleum ether soluble, the turpentine soluble, and the chloroform soluble give results at the same time both absolute and relative. These results are of little value when taken alone. They were suggested originally by an attempt to imitate the process described in Allen's "Commercial Organic Analysis," and it was for some time supposed by Miss Linton, myself, and probably by others, that a similarity existed in the materials dissolved by these several menstrua from different varieties of bitumen. I have for some time been reaching a conclusion that this is not the case.

In 1892 I distilled, under pressure, some California petroleum, and obtained a "cracked" distillate. I removed from this distillate the lighter portion and placed the residue remaining in the retort in a Becker glass, where it has been ever since. A solid material has gradually accumulated in a ring just below the surface of the liquid, and an addition of petroleum ether to the liquid produces a brown precipitate resembling ferric oxide when precipitated from its solutions. A precipitate having the same appearance can be thrown down from a turpentine, chloroform, or benzol solution of Trinidad pitch. I, for a long time, supposed they were the same substance—asphaltene—but I have lately discovered that they are entirely unlike. What relation, if any, they may bear to each other has not yet been determined. The precipitate from the California distillate appears to be wholly organic; that from the turpentine solution of Trinidad pitch appears to consist largely of a compound of alumina; while that from the chloroform soluble of the same bitumen appears to be a thio-salt of iron. Technically these solutions, with their precipitates, bring into consideration certain problems concerning solid bituminous minerals of the highest importance. The results of an analysis that sets forth the specific gravity, melting point, flowing test, petroleum ether, and carbon disulphide solubles, with perhaps the total amount of nitrogen and sulphur, tells very little concerning the application to any particular purpose of any given specimen of bitumen. The term "organic matter not bitumen" is misleading, for in many cases the volatile matter so determined is not organic at all.

That which is of primary importance in the analysis of an asphaltic mineral is to learn what proportion of the mineral consists of hydro-carbons first, soluble in petroleum ether; second, soluble in boiling spirits of turpentine; third, soluble in chloroform. If the volatile and combustible matter that appears as "organic matter not bitumen" is large in amount, it is of scarcely less importance to know exactly of what this material consists. Next in importance is the total amount and manner of combination of the sulphur; next the total amount and manner of combination of the nitrogen; and lastly, the total amount and manner of combination of the iron and alumina. The amounts of silica, lime, and magnesia should be known as guide in preparing the street mixtures, but they are of no significance in the determination of the specific qualities of the bitumen.

After several years' experience I am satisfied that the simplest as well as most satisfactory method of procedure is as follows: The solvent should be freed from water. The petroleum ether and chloroform should be agitated with strong sulphuric acid and distilled over caustic lime with steam. The turpentine should be shaken with freshly burned lime, poured off, and rapidly distilled.

The filters that I use are those prepared by Baker and Adamson, 11 cm. in diameter, which have been washed with both hydrochloric and hydrofluoric acids. These filters are not appreciably affected by the solvents. Three of them are balanced, and two are placed in separatory funnels, into which are weighed such portions of the bituminous mineral as will assure about half a gramme of bitumen. The filter is filled with petroleum ether, which is allowed to run through slowly until the colour has perceptibly diminished, and then the stop cock is closed between each addition of solvent for 15—30 minutes, until the solvent passes colourless. It is well to leave the funnels to stand over night at the end, but not at the beginning of the operation. The filters are dried in the air, then in an oven, at about 150° F., and weighed. Very close work can be done in this way. The solution may be tested with fuming nitric acid, or may be evaporated, and the residue treated with nitro-sulphuric acid, or 10 per cent. sulphuric acid for basic oils. The dried filters are returned to the funnels and exhausted as rapidly as possible with boiling spirits of turpentine. The turpentine should not be allowed to cool in the filters. When the exhaustion is complete the filters are washed with petroleum ether by filling the closed funnel above the filter to complete submergence and allowing the filter to digest for 30 minutes. A second digestion will usually completely remove the turpentine. The filters are then dried and weighed. The turpentine solution may be diluted with several times its volume petroleum ether and the precipitate examined by treatment with nitric acid, &c.

The dried filters are again returned to the funnels and after being exhausted with chloroform are again dried and weighed. The chloroform may be distilled off and the residue washed in petroleum ether and tested.

The dried filters are again returned to the funnels, and after exhaustion with 10 per cent. hydro-chloric acid, are washed, dried, and weighed. The loss is calcium and magnesium carbonate and calcium sulphate, if any is present. The calcium, magnesium, and sulphuric acid may be determined in the acid solution if desired, also iron and alumina, if any are present.

The filters are cut in small pieces and deflagrated with sodium carbonate and potassium nitrate, and sulphur, as pyrites, silica, iron and alumina, are determined in the fused mass.

A new portion of the bituminous mineral, containing at least ½ grm. of bitumen is deflagrated with sodium carbonate and potassium nitrate, and in the solution of the fused mass silica, sulphur, iron, and alumina may be determined, or lime and magnesia, if they are present. Nitrogen may be determined by any method desired.

A portion is dissolved in pure benzol and the solution washed with 10 per cent. dilute sulphuric acid. The acid solution is neutralised with sodium nitrate, when nitrogenous basic oils will be recognised by their odour. Another portion is exhausted with chloroform and the residue extracted with ammonium hydrate for the peat acids.

Free sulphur will be found in the petroleum ether solution. Organic compounds of alumina will be chiefly found in the turpentine solution. Very few solid bitumens are free from at least a trace of these curious and little-known substances.

"Organic matter not bitumen" consists of coke, peat acids and humus, partially decayed woody fibre and sulphur. If the bitumen contains ferrous sulphate, the sulphuric acid will be lost here and the iron will appear in the residue saturating the filter ash as colchotha, as is not infrequently observed.

It is very apparent that if a technical analysis of asphaltum involves the work laid out in this scheme, that the method indicated some time since by Mr. A. W. Dow is wholly inadequate. That such is the case has long been well known to a number of us who fail to regard ability to recognise the distinction between so called lake and land Trinidad pitch, as the paramount object of a technical analysis of sold bitumens. This scheme is applicable to any and all solid bituminous minerals with which I have become acquainted.

A very careful examination of the relative values of the different methods of determining sulphur in solid bitumens, and particularly late analyses of mixtures containing known quantities of sulphur, has demonstrated that the method in use by myself and my son, which was described in a late number of this Journal, determines *all* of the sulphur present. The values obtained are not too low, but are correct to within the usual errors of manipulation and personal equation. The great value of this method over any other, when considered in reference to the other determinations that are made in connection with it, cannot fail to impress anyone giving the subject close attention.

The relative significance of the results obtained through the use of this scheme and their proper interpretation are reserved for a later communication.

# Scottish Section.

*Meeting held in Glasgow on Tuesday, April 5th, 1898.*

MR. G. T. BEILBY IN THE CHAIR.

## THE PROGRESS OF AGRICULTURAL CHEMISTRY.

BY C. M. AIKMAN, D.SC.

Dr. Aikman traced the development of agricultural chemistry during the last 60 years, and pointed out that while Sir Humphrey Davy and others had carried out experiments on the influence of certain physical and mechanical properties—*e.g.*, heat and water retaining and absorbing powers on the fertility of the soil—little was know regarding its composition and function. He referred to the work of Liebig, Boussingault, Lawes, and Gilbert, and stated that De Saussure first drew attention to the importance of the mineral constituents of plants, though Liebig first successfully combated the "humus" theory then prevalent, advancing in its place his "mineral" theory, which latter led the way to the intelligent use of fertilisers. Though bones were used last century for fertilising purposes, the precise nature of their action was unknown. Liebig considered that the value of fertilisers was due to the phosphates and potash which they contained, and though he at first regarded nitrogen as a necessary ingredient, he subsequently concluded that the amount which reached the soil, dissolved in rain water, was more than sufficient for the needs of the plant, and that the mineral ingredients only were important. The Rothamsted experiments demonstrated that the supply of nitrogen was the most important factor in regulating plant growth and in determining soil fertilility.

Dr. Aikman then proceeded to discuss the trade in fertilisers. He referred to the use of bones as the earliest artificial manure, and to the production and application of superphosphate, patented by Sir John Lawes in 1842. The various sources of mineral phosphates and the use of potassic manures were also noticed, and the following data concerning guano were given. Guano was in use as early as the 12th century, but the sources have been for some time exhausted. More than five million tons have been used in this country since 1840. In 1850, nearly a quarter of a million tons were imported, and in 1855 about double that quantity was exported from South America. The following figures, showing the steady increase in the export of nitrate of soda, were quoted: 800 tons in 1830, over 10,000 tons in 1840, over 100,000 tons in 1865, over 300,000 tons in 1875, about 500,000 tons in 1885, and in 1890 over one million tons. The use and sources of sulphate of ammonia were also referred to.

Dr. Aikman next discussed the question of soil fertility, and pointed out that while research had shown that, generally speaking, three substances—*viz.*, nitrogen, phosphoric acid, and potash—were alone important in determining a soil's fertility, the knowledge of the quantities of these ingredients present was insufficient, as the condition of the fertilising matter was an important factor, and the dissolving power of plant roots was only approximately known. He referred to the experiments made by Dr. Bernard Dyer to determine the acid-dissolving power, which showed that the sap acidity of plants was equal to that of a 1 per cent. citric acid solution. Regarding the assimilation of nitrogen, he stated that as this was absorbed by the roots in the form of nitrates, an explanation of the process of nitrification was essential, and he referred to the work of Schloesing and Muntz, Warington, Winogradsky, Munro, and Frankland in this connection. In conclusion he discussed the question of the availability of atmospheric nitrogen, and referred to the results of Hellriegel and Wilfarth, which showed that certain plants—among which were peas, beans, clover, &c.—were able to assimilate the nitrogen of the air through the agency of certain bacteria inhabiting nodules in their roots. Reference was made to the application to leguminous crops of pure cultures of these nitrogen-fixing organisms, in the form of nitragin and of alinite to grain crops.

### DISCUSSION.

The CHAIRMAN said that he might mention the coke ovens as one very important source of ammonia which had not been alluded to. The production of ammonia from these was extensively carried on in Germany, was rapidly gaining a footing in England, and had also started in Scotland.

Dr. A. P. AITKEN said that he had been much interested in the clear and succinct account Dr. Aikman had given of the progress made in the application of chemistry to agriculture and the side industries which it had opened up. He did not know that there was anything that he could add to the very fitly-put description given; but Dr. Aikman had asked him specially to refer to one particular subject, which he did with some reluctance, namely, the subject of *nitragin*. He had been making experiments with that substance, in order to see whether the very flattering testimonials which the makers of it provided to those who bought it could be borne out by actual experience. It was rather early yet to say what was the result of these experiments, seeing that they had not yet terminated; but he was sorry to say that, so far as they had gone, they had been of a negative kind. He thought at the time this substance was boomed in Europe, that it was somewhat prematurely launched on the public, and that it would have been better if the promoters had had another year of actual experiment, to prove the value of it on a practical scale. He did not say that it had not been of any value, but so far as his experience went, the value had not been apparent. They might have better news of it when the results of the experiments now going on were obtained, but he could not say that he looked very hopefully to them. It was important to note, regarding the nitragin organism, that those who discovered it and those who had worked with it told them that it was found in all soils—that a soil without it had not yet come under their observation. There might, perhaps, be a case of a light soil in a hot climate where it might be so burned down from the surface as to kill the organism; and in such a case the microbe *Bacillus radicicola*, of which nitragin was a pure cultivation, might have been killed out; but it was not likely to happen under ordinary conditions. If the conditions of the field were such as to be suitable for the growth of a microbe of that kind, it would grow, and therefore it would not be necessary to import it. On the other hand, if the conditions of the field were such that it would not grow, importing it would do no good. What they wanted to know was, the conditions of soil and environment which made the microbe grow in the best way. If they discovered these, this knowledge would be valuable. In regard to the question of utilisation by plants of the nitrogen of the air, he would like to add one thing to what had been said. Amongst the first who experimented on this subject was M. George Ville, at the experimental station at Vincennes, near Paris, and he, first of all, long before Hellriegel, discovered that plants did utilise the free nitrogen of the air. M. Ville made very elaborate experiments. He had carefully read the published results of his experiments, and had been quite satisfied with the genuineness of them. He thought that although his work did not meet with that amount of acceptance which it deserved at the time, it was right that they should keep in memory the remarkable set of experiments he had made. He was thinking while Dr. Aikman was reading his paper, how curiously things had turned round. There was a time when it was thought that the first and the most important thing in regard to agriculture was filth; thereafter it was thought to be organic matter or humus; then, for a long time, it was considered to be mineral matter; and then it was thought by some enthusiasts to be all micro-organisms. Now it was recognised that all those things had their truth, and that every one of them, in its own position, held its importance in contributing to the fertility of the soil. As regards humus, a number of experiments had recently been made which very strikingly exhibited the fact that their forefathers, who had such faith in it, were not so far wrong as we, in these days of nitrate and phosphate, are apt to imagine. Dr. Frank, of Berlin, had showed, as regards some forest trees, how very dependent they were on humus—that they did not take their nitrates and phosphates from the soil directly, but depended for their nourishment upon fungi inhabiting the humus about their roots, and that the nutritive matter collected by the fungus was poured, as organised sap, into the tree, of which it was practically the nurse.

Mr. A. Macdonald said that he did not know that Sir J. B. Lawes had used spent char so early as Dr. Aikman had stated, but he thought that his own firm had been the first to dissolve and use this article. Dr. Aitken had put them right in the matter in stating that the humus of the agricultural elements had as much right to be considered as the purely mineral parts in manure, and he thought that was a point many chemists were apt to overlook. He was sure that organic matter had very much more to do with agricultural chemistry than chemists gave it credit for. Another matter which chemists forgot was the mechanical condition of manure. In comparing mineral phosphates with organic phosphates, the *bulk* or volume of the mineral phosphates is very much less than phosphates from an organic source, and the same thing applied to a certain extent to nitrogen. He thought therefore the mechanical condition should be taken into consideration by agricultural chemists when estimating the efficiency of a manure. In illustration of the argument, if pure rock sulphur were ground into a powder, it would be found that the volume of it when compared with an equally pure sulphur, *viz.*, flowers of sulphur, would be enormously less; in fact, 3 cwt. of ground sulphur could be filled into a barrel which could contain only 1 cwt. of flowers of sulphur. The same thing applied to carbonate of magnesia. The difference between the volume of a mineral carbonate of magnesia and that of precipitated carbonate of magnesia was very great, and everyone knew the difference in efficiency, especially in medicine, between ground sulphur and flowers of sulphur, and between native carbonate of magnesia and precipitated carbonate of magnesia. These differences were not due to chemical condition (for all might be equally pure), but solely to the different states in which the atoms or molecules existed, or, in short, to their mechanical condition. There was an interesting experiment which he once made bearing upon this subject of mechanical condition, and it might also be considered as having some relation to organic matter in manures as well. He was at that time troubled about the economical disposal of a by-product in the liquor obtained from bone boiling. This bone liquor was concentrated until it contained nitrogenous matter equal to about 7 per cent. ammonia. It was a gluey, sticky mass, and it was difficult to find a market for it; it was customary to add a little of it to manures while in process of dissolving, but even in this way it could only be used sparingly, for fear of making the resulting product sticky, and therefore unusable as a manure. He thought if he took a certain quantity of bone charcoal and mixed it with an equal weight of this concentrated bone liquor, in course of time the superfluous moisture would be evaporated, and the whole mass would be obtained in a condition fit for easy use. 50 tons of bone charcoal and 50 tons of bone liquor were mixed together, and left to lie for three or four months. At the end of that time the whole of the nitrogen, or most of it, had been converted into ammonia, and the mass completely lost its gluey character. A similar experiment was made with Belgian phosphates instead of bone charcoal. This second mixture was laid down in March 1891, and though seven years had passed since that time, the mixture lay to this day pretty much in the condition in which it was laid down. He attributed the change which had taken place in the one mixture and not in the other to the different mechanical conditions of the phosphates used, and probably also to the fact of the one being of organic origin while the other was of mineral origin.

Mr. J. W. Biggart suggested that the difference between the action of the bone charcoal and that of the Belgian phosphates in Mr. Macdonald's experiments was caused by the high absorptive power of the former for gases, in virtue of which the char absorbed oxygen from the air and oxidised the organic matter with formation of ammonia.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Desiccating various Substances, An Improved Mode and Apparatus for.* A. Huillard, Paris, France. Eng. Pat. 15,231, June 25, 1897. Date claimed under Internat. Conv., Jan. 7, 1897.

An apparatus for desiccating pasty products, and especially tanning extracts, consisting of an endless travelling band guided by rollers and passing through chambers heated by steam pipes. At a convenient part of the apparatus the band dips into a tank containing the material to be dried, and the thin layer adhering to this band is carried through the chambers, being afterwards brushed off in a dried condition and falling into a suitable receiver. In some cases the heating chambers are unnecessary, and the band then travels through the open air.—E. G. C.

*Compressed Gas, Impts. in Means for the Storage of.* H. J. Barbier, Paris, France. Eng. Pat. 10,796, April 30, 1897.

Relates to arrangements for minimising the danger due to the accidental bursting of cylinders for storing compressed gases. The cylinder is divided into compartments by interposing pierced diaphragms, or by constricting the tubular shell at intervals, and each cylinder may be connected to a primary collector formed of a stout tube provided with hollow screw plugs, these collectors in turn opening into a main collector. The heads of all the connecting screws and bolts are situated outside the apparatus, to facilitate the grouping together of the elements or cylinders. In a modified arrangement for reservoirs of small capacity, the cylinders are mounted in a plate having passages leading from the filling orifice to each cylinder.—E. G. C.

*Filter Presses, Impts. in, and in Apparatus for Opening and Closing the same.* G. M. Park, Blackburn. Eng. Pat. 6480, March 12, 1897.

"In apparatus for filtering sewage, sludge, &c." there is claimed "the combination with a filter press kept closed against the pressure of the sewage," &c., "by means of a

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[*] Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

central screw, or one or more nuts externally applied, and produced by worm or worm wheel acting in conjunction with spur or bevel gear,"—"a tumbling or disengaging worm whereby the worm wheel can be turned quickly by hand." Also a "dovetailed hook sleeve having in its upper part a roller, ball or bobbin, so that any plate can be easily withdrawn as desired." In addition there is also claimed "a spring slip coupling" "to slip when the required pressure is attained.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Acetylene, The Purification of.* P. Wolff. Chem. Zeit. **22**, [28], 281—282.

Mysterious purifying materials for acetylene are frequently recommended on the ground that they abolish the usual smokiness of the acetylene flame. Since the tendency of acetylene to form soot on its combustion is due to polymerisation of the acetylene, and can only be avoided by the use of special burners, no purifying material can prevent the formation of soot. Ferric hydrate, or Laming's material, or other agents such as are used for the purification of coal-gas, may be used to remove ammonia and sulphuretted hydrogen from acetylene, but they do not remove phosphuretted hydrogen, which is by far the most objectionable impurity in it. Lunge and Cedercreutz have recommended the use of chloride of lime for the removal of phosphuretted hydrogen by oxidation (this Journal, 1897, 1016). Earlier experiments made by the author showed that small explosions occurred when acetylene was passed through a dry purifier containing chloride of lime, and these appeared to arise from chloride of nitrogen produced from the ammonia in the gas and the chlorine of the bleaching powder. It is therefore necessary to remove the ammonia, before passing acetylene over chloride of lime. This may be done by an efficient washer, and chloride of lime will then remove phosphuretted hydrogen and any sulphuretted hydrogen that has escaped the washer. Chlorine and moisture may then be removed in a third purifier charged with lime. Acetylene thus purified has merely a faint ethereal odour, and to facilitate detection of leaks in services, it may often be desirable to odorise it, either by means of volatile compounds having a powerful odour, such as carbylamine or amyl acetate; or by passing it over calcium carbide, which will thoroughly dry it, and at the same time liberate traces of crude acetylene, which will odorise the bulk while not appreciably contaminating it.

Neither copper nor its alloys are attacked by pure acetylene, but impurities in acetylene quickly produce brown stains on polished brass fittings. These stains are due chiefly to copper phosphide, and not to copper acetylide, as was at first feared. Purification of acetylene is necessary to prevent corrosion of the fittings, contamination of the air of rooms where it is consumed, and deficient illuminating power.—J. A. B.

*Calcium Carbide, Analysis of.* H. Bamberger. Zeits. f. angew. Chem. 1898, [11], 213.

*See under* XXIII., *page* 494.

*A Gas Balance ["Econometer"] for Indicating Continuously the Proportion of Carbonic Acid Gas in the Flue of Furnace Gases.* J. Franklin Inst. 1898, **145**, 205.

*See under* XXIII., *page* 489.

*Cerium, Determination of, in Presence of the Rare Earths.* G. von Knorre. Zeits. f. angew. Chem. 1897, [22], 717.

*See under* XXIII., *page* 491.

*Of Coal Gas, Alleged Injury to Vegetation through Leakages into the Soil.* O. Pfeiffer. J. f. Gasbeleucht. **41**, 137.

*See under* XXIV., *page* 504.

### PATENTS.

*Production of Artificial Light, Applicable for Lighthouse and Ordinary Illumination; Impts. in Apparatus for the.* J. R. Wigham, Capel Street, Dublin. Eng. Pat. 4125, Feb. 16, 1897.

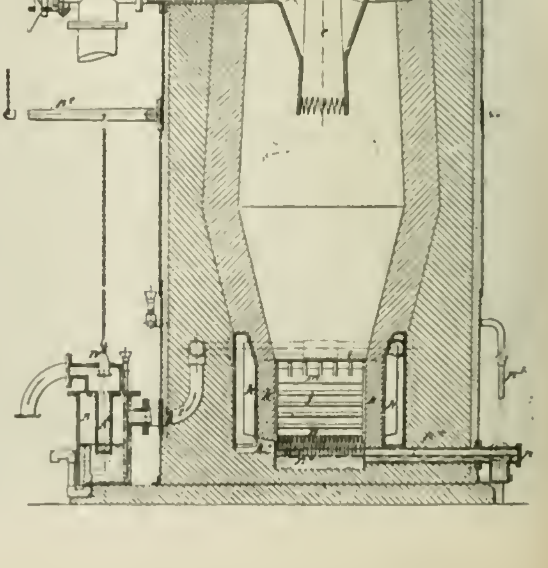
An apparatus for producing hydrogen or acetylene is formed of a cylindrical receptacle with a horizontal perforated partition half way up, upon which the zinc or carbide rests, and with semicircular openings it its bottom edge. Surrounding this is an inverted funnel having holes around its bottom edge, and a suitable valve and outlet for gas at the top. Outside all is a cylindrical vessel to hold the exciting liquid. When hydrogen is produced it is burned in an incandescent burner; when acetylene is produced it is burned in any ordinary burner.—R. S.

*Gas Retorts, An Improved Method of and Means for Removing "Scurf" or Retort Carbon from.* C. Billington, jun., Wolstanton, Stoke, Staffordshire. Eng. Pat. 7435, March 23, 1897.

This invention is supplementary to that described in Eng. Pat. 7286 of 1896, according to which the "scurf" is oxidised and removed by a forced draught of air through the retort. In the present invention an air-draught is produced and forced through a central metallic nozzle into the retort, in combination with a draught or current of air induced to flow through a metallic pipe or casing surrounding the central nozzle. The draughts are produced by means of a mechanical blower, &c., which is worked by an electric or other suitable motor fitted or attached to a travelling or fixed bed or foundation. In a modification, the air from the nozzles is forced through a suitably perforated tube introduced into the retort.—R. A.

*Production and Purification of Gas, and Apparatus therefor; Impts. relating to the.* J. A. Bellon-Lencanchez, Paris. Eng. Pat. 8513, April 2, 1897.

*Gas Producer.*—The grate is formed of two opposite stepped gratings, *i*, and a horizontal grating, *n*, the capacity of the grate being varied as desired, by merely raising the horizontal grating, the bars of which are supported on metal bars, *n′*, which can be placed in any of a vertical series of holes in the supports, *k*. A regulating water-feed is provided, wherein the water, which may come from hollow bars, *m*, passes down a tube, $p^3$, to a box, *p*, connected by a horizontal pipe, *p″*, with the ashpit bed, the box having a sluice operated by a screw and hand-wheel, so that the



excess water flows off by the sluice. The air-feed is regulated by the rise and fall of the gasometer bell, which is connected by a chain, &c. to a lever, B$^2$, in such a manner that as the bell rises the lever falls, and *vice versâ*. The lever, B$^2$, is connected by a chain to an air regulator cone, B$^1$, which opens and closes the escape orifice on the air tube leading from the bellows. A water seal, B$^3$, is provided in the regulator to prevent the return of the gas should the bellows stop working. The air is heated by circulation in spaces, *h*, on two opposite sides of the furnace, provided with heat-conducting ribs or projections. The gas escapes through a central gas duct *e*, having a lower serrated edge. This duct communicates with two tubes, *e*$^1$, designed for facilitating cleaning, and having two openings, *e*$^2$, in communication with the upper part of the producing apparatus. The whole of the gas passes out by the general conduit, *c*.

*Gas Scrubber.*—The gas enters the scrubber through a serrated tube dipping into water, above which is a perforated grating upon which the coke rests. A double current of water falls down the column from a distributor above.

*Purifier.*—This consists of a casing wherein are mounted, so as to be partially immersed in liquid, a series of vertical and vertically movable gratings adapted to be readily dismounted, and formed of waved or undulating bars breaking line with one another. The tars run down the gratings in the concave portions of the bars, while the gases circulate horizontally around the said bars, entering at one side of the casing and leaving at the opposite side.—R. S.

*Enriching Gas of Low Illuminating Power, Process for.* O. Imray, Southampton Buildings. From E. Pilous, G. Ottermann and Co., and A. Michaelis, all of Vienna. Eng. Pat. 23,237, Oct. 9, 1897.

Gas of low illuminating power, obtained by the dry distillation of house, market, or street refuse or sweepings, sewage matter, petroleum, oil or wood waste, and other organic residues and waste products, is passed, either before or after purification, into a carburetting vessel containing calcium carbide, either alone or mixed with hygroscopic salts, such as sodium carbonate. Acetylene is slowly and uniformly produced and taken up by the gases.—R. S.

*Automatic Igniters for Illuminating Gas, Process for the Manufacture of.* M. Bernstein, Berlin. Eng. Pat. 27,652, Nov. 24, 1898.

Organic materials, such as web, fibres, filaments, &c., are mixed with inorganic materials, such as oxides or salts of calcium, barium, or strontium, or of the earth metals, as aluminium, chromium, beryllium, zirconium, yttrium, erbium, cerium, lanthanum, didymium, or thorium. The mass formed is then saturated with a solution of platinum or palladium, or a mixture of the two. The whole is then dried, and the organic matter burned off, leaving a very porous body suitable for the purpose. Three methods of applying a mass of this material to incandescent gas burners, are shown.—R. S.

*Hydrocarbon Vapour Burners, Impts. in and relating to.* T. W. Fletcher and Fletcher, Russell, and Co., Ltd., Warrington. Eng. Pat. 10,111, April 23, 1897.

The burner, which is applicable for heating injection furnaces, &c., is provided with "a helical or conical coil of suitable tubing," one end of which, at the base of the coil, communicates with the hydrocarbon supply. The outward end of the coiled tubing returns, preferably outside the coil, to a point at or beyond the base of the latter, where it is provided with a nozzle or nozzles so directed that the volatilised hydrocarbon issuing therefrom will blow axially through the coil, burning inside or beyond it, air being drawn in at the base. The coil, which may be arranged either horizontally or vertically, is surrounded by a sheet-metal or other casing, which is contracted at one end into a burner nozzle as usual, and which may be provided with a receptacle for spirit or naphtha, for use in the preliminary heating of the coil. The arrangement of the coil and the burner as a whole are claimed.—H. B.

*[Incandescence or Heating] Burners for the Production of Non-Luminous Flames, Impts. in and connected with.* M. Graetz, Berlin. Eng. Pat. 8755, April 6, 1897.

In a lamp, of the type mentioned in A. Graetz's Eng. Pat. No. 2295 of 1897 (this Journal, 1898, 141), there is here claimed the combination with the wick-tubes of a perforated cylinder, closed at its top, and arranged upon the inner wick-tube so as to project beyond the wick; a deflecting ring fixed on the outside of the cylinder, below the top of the latter; and a cap surrounding the wick-tubes, and having its upper part divided into two concentric collars. The inner collar is contracted till its edge is "comparatively near to the edges of the wick-tube and the ring," and it extends up to a plane located between the plane of the tops of the wick-tubes and the plane of the ring. The outer collar extends up to and above the plane of the ring, and as its main purpose is to form an auxiliary rest for the incandescence mantle, it may be dispensed with if the lamp is to be used for heating purposes only.—H. B.

*Incandescent Wick Burner for Liquid Fuel, An Improved.* J. Spiel, Berlin. Eng. Pat. 10,801, April 30, 1897.

In a lamp with a circular wick, the burner tube is surrounded by a cylindrical cap, the upper edge of which is turned inwards at about right angles, and extends inwards over the burner top, leaving a central opening of the same diameter as the inner wick-tube. A central rod supports a deflecting disc of the usual shape at a short distance above the circular opening. Air inlets at the base of the cap allow air to pass upwards between the cap and the outer wick-tube, then inwards at right angles across the top of the wick, and out beneath the deflector. The top of the outer wick-tube is slightly widened, and rises a little above the level of the wick. The outer wick-tube is fitted with any known raising and lowering device, in order that the distance between its upper edge and the inner surface of the cap may be regulated.—H. B.

*Incandescent Bodies, A Process for Manufacturing* Moritz Bernstein, Berlin. Eng. Pat. 29,796, Dec. 16, 1897.

Bodies composed of vegetable fibres, asbestos, firebricks, &c. (see next abstract), are to be "impregnated" with indium, erbium, cerium (or mercury), decipium, &c., "in a liquid or pulverised state." The second claim is: "the use of erbium mixed with indium and other bodies in the manufacture of incandescent bodies, substantially as described."—H. B.

*Incandescent Bodies, A Process for Manufacturing.* Moritz Bernstein, Berlin. Eng. Pat. 29,797, Dec. 16, 1897.

The claim is: "A process for manufacturing incandescent bodies by impregnating with a mixture of (1) Zinc, zirconium and calcium, or (2) Zirconium, beryllium, barium (or cerium), in suitable proportions, in a liquid or pulverised state, bodies or tissues of vegetable fibre, hair, or asbestos, calcined or non-calcined clays or earths, especially firebricks, steatite, porcelain-clay, and also natural or artificial meerschaum, or combinations of the said products."—H. B.

*[Incandescence] Petroleum Lamps, Impts. in or relating to.* Max Bernstein, Berlin. Eng. Pat. 496, Jan. 7, 1898.

The lamp has a circular wick, and above the wick-top is supported by a central rod, a hollow head in which the vapours arising from the wick, and the central current of air, are to mix, afterwards passing out through perforations in the head, and burning inside a mantle as usual. By means of a toothed wheel acting on the central rod, the burner head may be vertically adjusted. The head carries on its under side a short tubular projection, which may telescope within the inner wick-tube, and a horizontal flange, so that by racking the head up or down, the vapour stream entering the head may be regulated and at last wholly cut off. There is claimed the arrangement at the mouth of the wick-tube of the adjustable head above described, either with or without a slotted lever device for raising the burner gallery, for convenience in lighting the lamp.—H. B.

*Incandescence Mantles, A Burner for Burning off.* W. Mackean, Westminster. Eng. Pat. 3193, Feb. 11, 1898.

The burner consists of (1) a lower, upright Bunsen burner, giving a flame which spreads somewhat outwards, and (2) an upper, circular Bunsen burner, the flame of which issues downwards, in the form of an inverted cone, from perforations in a tubular ring. The mantle is held in a position to be acted on more or less by either flame, and is moved up and down as required.—H. B.

*Petroleum, Impts. in the Manufacture of Fuel Blocks from.* H. Hansen, Christiania, Norway. Eng. Pat. 10,900, May 1, 1897.

The blocks are manufactured by combining refined petroleum, caustic soda, thick turpentine, pine resin, margarine, and cocoa-nut oil in stated proportions, the proportion of petroleum being between 75 per cent. and 85 per cent. of the total weight of the ingredients. The margarine, cocoanut oil, and about one-third of the caustic soda are made into soap, which is well dried and granulated. The turpentine and resin are then melted together and mixed with the soap granules, after which the petroleum is added and the mixture heated to about 105° C. The rest of the caustic soda is then added, and the whole is stirred until it attains the consistence of porridge. The mixture is kept at a temperature of 100° C. until it becomes quite thick, after which the temperature is raised to about 105° C., and maintained at that degree for about an hour. The material is then moulded into large blocks or slabs, which, after cooling, can be cut into smaller blocks.—R. A.

*Heating and Evaporating Liquids, Impts. in Apparatus for Effecting Interchange of Temperature between Fluids, or for.* S. Frank, Frankort-on-the-Main, Germany. Eng. Pat. 12,114, May 15, 1897.

Attemperating cells or chambers are constructed of pairs of similar plates, the edges of which are curved or bent to form flanges, these latter being connected by screws or other fastenings. Flanged openings are formed by stamping or punching, and, when the cells are brought together, these flanges fit over one another so as to form a continuous passage for the fluid. The plates are prevented from buckling by nipples arranged to coincide and bear against each other. These nipples are also formed by the stamping process, and owing to their arrangement, they are constantly washed by the fluids in the cells, and are therefore not liable to overheating.—E. G. C.

*Heat for Industrial Purposes, A Process of Transmitting.* G. F. Dinsmore, Boston, U.S.A. Eng. Pat. 1310, Jan. 17, 1898. Date claimed under Internat. Conv. June 18, 1897.

The invention is especially applicable for fusing refractory material, and consists in superheating fluid in a coil, and allowing the jet or stream therefrom to attain a high velocity. The jet is led into a closed retort, where it impinges upon the material to be treated, and its kinetic energy is converted into heat.—E. G. C.

*Calcium and other Carbides, and By-Products obtained thereby: Improved Manufacture of.* A. J. Boult, London. From R. Memmo, Rome. Eng. Pat. 24,077, Oct. 19, 1897.

Claim I. "In a furnace for the manufacture of carbides, particularly of carbide of calcium, a movable bottom insulated from the rest of the furnace, so as to prevent the formation of lateral arcs between the walls and the carbons," &c. II. . . . "the arrangement of carbons for forming star-shaped or ring-shaped arcs for using alternating polyphase currents," &c. III. . . . "finely pulverising the materials to be treated in an electric furnace, mixing them with any adhesive liquid or substance, pressing them into blocks and drying the bricks thus obtained, and finally breaking them into small pieces and introducing them in this shape into the furnace," &c. IV. . . . "the formation and collection of water-gas," &c. A mixture of slaked lime and powdered carbon is mixed with some adhesive substance so that it may be pressed into compact bricks and dried, then broken up. V. . . . "the combination and arrangement of parts."—J. C. R.

*Calcium Carbide, An Improved Process and Apparatus [Continuous Furnace, &c.] for Producing.* H. L. Hartenstein, Bellaire, County of Belmont, U.S.A. Eng. Pat. 28,226, Nov. 30, 1897.

The process is "characterised by the fact that the operation upon the limestone is continuous, and that it is at no time allowed to become cool during the treatment. A further distinguishing characteristic is the combined effect of the electrical current and of the solid and gaseous fuel as heating agents upon such limestone, whereby the same is highly impregnated with carbon," &c. "The limestone, feeding down into the lower chamber of the furnace by gravity, reaches its base in a highly heated condition, where it is subjected to the electrical current, thus conserving heat; while, on the other hand, the heat produced in the smelting operation, due to the combined action of the electric current and the combustion of the solid and gaseous fuel [ordinary illuminating gas is suitable], is not wasted, but, on the contrary, ascending through the mass of crude limestone, assists in the raising of its temperature, and, therefore, in conserving heat energy."

Claim I. is for the "process of carburetting calcium, which consists in calcining limestone and then forcing into the calcined mass, while in a heated condition, a carbonaceous material by the aid of a combustible gas under pressure, and subjecting the mass to the action of an electrical current," &c. II. . . . "mixing raw limestone with a solid fuel and firing the same, whereby to drive off its carbon dioxide, then forcing into the calcined stone a pulverised carbonaceous material [finely pulverised coke], and simultaneously treating the mass with an electrical current," &c. IV. . . . "firing the mass in the presence of an air blast," &c. Other claims relate to apparatus and the furnace, which is shown as a calcining chamber supported in an elevated position with a track arranged beneath, with a base portion movably mounted on said track, the electrodes mounted in the walls of the movable portion (of which there are duplicate sections), and pipes or chambers opening into the chamber thereof adjacent to the electrodes.—J. C. R.

*Acetylene Gas Apparatus, Improved.* G. Jacobi, Dresden. Eng. Pat. 2309, Jan. 28, 1898.

A vessel containing liquid carbonic acid is connected with the top of a closed water reservoir and with a pipe leading therefrom, which sprays the water on to carbide in a generating vessel. The carbonic acid forces the water at uniform pressure into the generator, and also breaks up the stream and distributes it over the carbide. A purifier, condenser, and gasometer are connected to the generator.
—J. A. B.

*Acetylene, Apparatus for Generating.* H. O. Mareks, Berlin. Eng. Pat. 2602, Feb. 1, 1898.

The bell of a gasholder has attached to it a rod with cam-shaped slot, in which slides a roller attached to a rod on which is pivoted a catch, which engages in a toothed bar attached to horizontal rods which pass into the upper part of a generating chamber and support cartridges or definite charges of carbide. The bottom of this chamber contains water, and the fall of the gasholder bell operates the mechanism described, and thereby withdraws the rods to such an extent that one cartridge is dropped into the water. The acetylene generated passes into the holder, and when the bell again falls, another cartridge is dropped from the rods.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Manufacture of.* M. Wagner, Nogent-sur-Marne, France. Eng. Pat. 29,960, Dec. 17, 1897. (Under Internat. Convention.)

The apparatus is devised to produce acetylene under a pressure of from 20 to 100 cm., for heating purposes. Carbide, contained in a series of perforated compartments in a closed vessel, is attacked by water admitted at the base of the vessel from an expansion chamber, in which the level of the water is kept constant by means of a supply from a feed cylinder and an overflow cock. The height at which this cock is placed determines the pressure at which the water is supplied to the generator, and therefore the pressure of the gas. The gas passes from the top of the generator to a purifier immersed in the water in the expansion chamber, and thence to the burners. A gasometer may be added to receive surplus gas.—J. A. B.

*Acetylene Gas for Private and Public Lighting, Machine for Producing.* R. Quatannens-Moens, Dixmude, and E. Carrcer-Dilger, Bruges, both in Belgium. Eng. Pat. 1665, Jan. 21, 1898.

Carbide dropped from a distributor opens by its weight a counterpoised valve and falls into water in a closed vessel, whence the generated gas passes through water in an annular tank surrounding the generator, to a bell gasholder, and thence through a bubbler and purifier, to the burners. The distributor is a cylinder having 20 quadrantal compartments, which are filled with carbide, and discharge their contents on to the valve above the generator in turn as the cylinder is rotated in consonance with the rise and fall of the bell, which is provided with levers actuating a ratchet wheel on the shaft of the cylinder.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating and Burning; chiefly Designed for Use on Velocipedes, Auto-Cars, Boats, and the like.* L. Chambault, Paris. Eng. Pat. 19,951, Aug. 30, 1897.

A lamp, termed "La Marveilleuse," is described in several modifications by the aid of 19 figures. A water reservoir is placed above a carbide container, and various means for regulating the admission of the water to the carbide are used.—J. A. B.

*Acetylene Gas Generators, Impts. in.* E. J. Dolan Philadelphia, U.S.A. Eng. Pat. 20,011, Aug. 31, 1897. (Under Internat. Convention.)

In communication with the gas-exit pipe of a generator, in which a cage containing carbide is so placed that it dips beneath the surface of water to a slight extent, is a chamber containing a diaphragm, the under side of which is exposed to the pressure of the gas in the exit pipe. The diaphragm, by its rise and fall with the pressure of the gas, actuates, through levers and a pawl, a ratchet wheel attached to the shaft of the carbide cage, which is thus rotated, and a fresh portion of its contents is exposed to the water.
—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production and Storage of.* G. Trouvé, Paris. Eng. Pat. 12,110, May 15, 1897.

Calcium carbide is decomposed by water which enters from a surrounding reservoir through a hole in the base of the carbide container. The acetylene produced passes to a bell gas-holder through a pipe in which is a cock, which is linked by levers and an extensible arm to the bell, so that the cock is closed as the bell rises. The increasing pressure of the gas in the carbide container forces the water therefrom, while acetylene formed after the cock is closed, passes through a bye-pass into the bell, until consumption causes the descent of the latter and therewith the re-opening of the cock.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* F. Dresser, Liverpool. Eng. Pat. 13,081, May 27, 1897.

The apparatus consists of a vessel, the upper part of which is divided into two compartments by a vertical partition. One compartment is closed at the top, and is provided with a gas outlet. The apparatus is filled with water, and canvas bags containing carbide, and attached by a cord to a rod, are introduced through the open compartment beneath the vertical partition into the closed compartment, in which they rise to such height as the length of the cord permits. The gas generated displaces water, which overflows from the open compartment. Enclosing the carbide in bags is said to retard the reaction, and to yield purer gas than when the action is unrestrained.—J. A. B.

*Oil-Vapour Burners, An Impt. in.* J. S. V. Bickford, Camborne. Eng. Pat, 2332, Jan. 28, 1898.

Four pipes, "somewhat inclined and forming a square," are connected together at three of the corners by means of a bend or elbow. At the open corner, the free end of one of the pipes is connected with a vertical pipe which rises from a boss in a base plate and receives the oil supply from a reservoir or otherwise. The other pipe, having a free end at the open corner of the square, is jointed to a vertical pipe, which passes down some distance, then bends inwards and upwards, being provided with a vertical jet pipe directly under the centre of the square. Immediately over the jet pipe there is supported on two opposite sides of the square a deflecting metal plate. The square of pipes having been heated by a temporary fire, oil injected into them issues as vapour at the central jet, the flame impinging upon the deflecting plate, and heating the square of pipes so that the vaporisation is continuous.—H. B.

*Reverberatory Furnaces, Impts. in.* J. Q. Rayner, Kulebaki, Nischnij-Novgorod, Russia. Eng. Pat. 3039, Feb. 7, 1898.

The vault of the furnace is formed with two dome-like cavities or chambers, one on each side of the centre line of the hearth, the portion of the vault between the cavities being preferably flat. The patentee states that this construction ensures a more thorough mixture of the combustion gases and air, and a more uniform distribution of the heat to the hearth.—R. A.

*Superheater for Steam, An Improved.* I. Grubinski, Warsaw. Eng. Pat. 3638, Feb. 14, 1893.

A superheater is formed of buckled circular plates bolted together at their edges, and partly separated by an annular disc of smaller outside diameter. Between each buckled plate and the central disc there are curved angle irons dividing up the enclosed space into a number of spiral passages. The steam to be superheated enters at or near the centre of the apparatus, and after passing through one set of spiral passages outwards, escapes round the edge of the central dividing annulus, and passes by the second set of spiral passages inwardly towards the centre, and away by the outlet pipe.—E. G. C.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

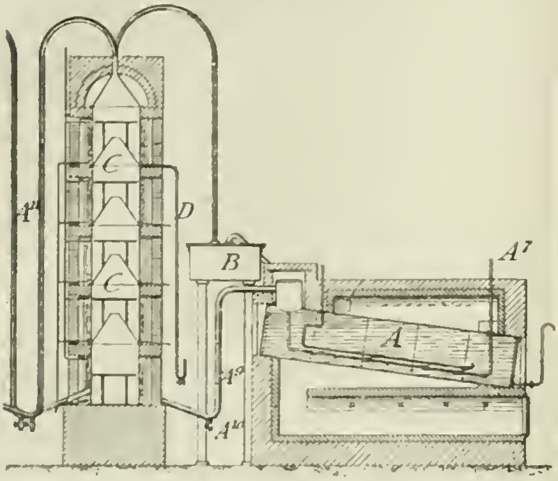
### PATENTS.

*Charcoal, Impts. in or connected with the Manufacture of.* W. A. G. von Heidenstam, Skonvik, Sweden. Eng. Pat. 9656, April 15, 1897.

The charcoal is made from wood, wood chips, peat, or other suitable material, by feeding and pressing the prepared raw material in and through a heated retort, so that the material moves through the retort in a continuous stream, and issues at the end as hard charcoal. The retort is provided with suitable pipes for withdrawing the byproducts at the stages at which they are generated. The charcoal may be cooled by cooling the end of the tube through which it issues.—R. A.

*Distilling, Fractioning, and Cracking Mineral Oils [Petroleum], and Obtaining Various Products therefrom; A Method of, and Apparatus for that Purpose.* V. I. Ragosin, St. Petersburg. Eng. Pat. 1411, Jan. 18, 1898.

The mineral oil is supplied through the pipe $A^7$ to the still A, and is mixed with benzene supplied from the tank B, the benzene dissolving the oil and lowering its boiling temperature. The benzene may also be vaporised by passing it through a boiler on its way to the still. The mixed vapours are conveyed through a pipe $A^9$, to the lowest of a set of condensers C, arranged in a suitable tower, any liquid which primes over into the pipe $A^9$ being drawn off through the cock $A^{10}$, and the liquid condensed in the condensers C being drawn off through the pipes D. From the first tower the uncondensed vapour is conveyed through the pipe $A^{11}$ to a second condensing tower, and so to a third, and back through the series, the benzene vapour ultimately remaining, being collected in the tank B. The pipes D from the condensers are led through a cooler to receptacles, in which the liquids can be kept separately or mixed as desired. In another arrangement the vapours from the still are passed into a cracking vessel, and subjected to the action of heat before passing into the condensers. Any liquid products of the cracking, such as liquid paraffin wax, can be run off by a suitable cock, or run back into the still. A travelling brush, which is worked by a worm rotated by an external handle, is employed to clear the carbon deposit from the bottom of the still.—R. A.



*Ortho-nitro-chloro-benzene and Pure Para-nitro-chloro-benzene, Impts. in the Manufacture or Production Simultaneously of Pure.* G. W. Johnson, London. From The Chemische Fabrik Griesheim, Frankfort-on-the-Maine, Germany. Eng. Pat. 2480, Jan. 31, 1898.

The mixture of *o*-nitrochlorobenzene with *p*-nitrochlorobenzene obtained by nitrating chlorobenzene, is submitted to systematic fractional crystallisation and fractional distillation. Under a pressure of 753 mm., *p*-nitrochlorobenzene boils at 238·5 C., and at 113° C. under a pressure of 8 mm., whilst o-nitrochlorobenzene boils at 245·5 C. at 753 mm., and at 119 C. under 8 mm. pressure. The *p*-nitrochlorobenzene collects in the first fractions, and the *o*-nitrochlorobenzene in the last, the two isomerides being precipitated from these fractions by fractional crystallisation.—T. A. L.

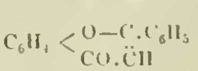
## IV.—COLOURING MATTERS AND DYES.

*Orchil Fermentation.* F. Czapek. Centr.-Bl. f. Bakter. u. Parasitenk. **4**, [2], 49—52; Chem. Centr. 1898, **1**, [11], 684.

It is stated that the formation of the red colouring matter from different varieties of lichens, is due, in addition to the purely chemical action of ammonia, to the activity of micro-organisms present in the liquid (as putrescent urine) used for furnishing the ammonia. A decoction of lichens, treated with putrescent urine, was converted in 3—4 weeks at 20—25 C. into a red colouring matter, which gave all the reactions of orcein. If, however, the mixture be sterilised at 100 C. for half an hour, no formation of orcein occurs, even if ammonium carbonate be added, or the mixture be allowed to stand in contact with ammoniacal air. Chloroform also arrests the formation of colouring matter. Sterile samples can be successfully infected by inoculation from samples which have formed orcein, if care be taken that the liquid, or the air above the liquid, contains ammonia. The active microbes do not appear to adhere to the lichen, but are furnished by the putrescent urine; they were isolated from the latter by a particular method of culture. Cultures grown on alimentary soil from sugar-peptone agar and ammonium carbonate (1 per cent.) were particularly active. The ferment occurs in the form of short rods similar in appearance to the hay bacillus. The pure cultures inoculated in sterile fermenting liquids of the above-mentioned kind, produced orceïn in 3—4 weeks at 25° C. The bacillus acts on the "lichen-acids," with formation of orcin, which, in the ammoniacal liquid, is converted into orcein. It is stated that there are other specifically different fungi, which, under the same conditions, will also cause the formation of orcin. The organisms isolated from putrescent urine, are altogether different from the urea ferments, and they resist ammonium carbonate to such a degree that this property may be used for their isolation. It is stated that besides fermentation, there are also oxidation and reduction processes.—A. S.

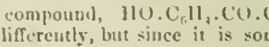
*3-Oxyflavone, Synthesis of.* T. Emilewicz and St. v. Kostanecki. Ber. **31**, 696—705.

Flavone—



$$C_6H_4 < \begin{matrix} O-C.C_6H_5 \\ CO.\ddot{C}H \end{matrix}$$

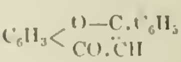
which is the parent substance of chrysin and other yellow vegetable dyestuffs, contains the same carbon chain as benzal-acetophenone, $C_6H_5.CH:CH.CO.C_6H_5$, and the authors have studied the *o*-hydroxylated derivatives of the latter with a view of synthesising flavone or its derivatives. On converting the acetyl derivative of 2-hydroxybenzal-acetophenone, $1.2.HO.C_6H_4.CH:CH.CO.C_6H_5$, into the dibromide, and treating this with alcoholic potash, it yields α-cumarylphenylketone—



and not flavone. There still remained the possibility that the isomeric compound, $HO.C_6H_4.CO.CH:CH.C_6H_5$, might behave differently, but since it is somewhat difficult to prepare, its ethoxy derivative, obtained by condensing benzaldehyde with resacetophenone ethyl ether—

$$1.3.4.C_6H_3.C_2H_5O(OH)COCH_3,$$

was employed. This, after acetylation, was converted into the dibromide and treated with alcoholic potash, when a substance having the composition $C_{15}H_9(OC_2H_5)O_2$ was obtained. This is 3-ethoxyflavone, from which 3-hydroxyflavone can be obtained by boiling with hydriodic acid. The intermediate products are formed as follows:—A solution of 10 grms. of resacetophenone ethyl ether and 5·9 grms. of benzaldehyde in 100 grms. of alcohol is treated with 20 grms. of a 50 per cent. soda-lye. After standing some time, the sodium salt of benzalresacetophenone ethyl ether crystallises out. It is decomposed with dilute hydrochloric acid, and separates from alcohol in yellowish needles melting at 104° C. The acetyl derivative forms similar crystals melting at 75 C. When this last product is dissolved in carbon bisulphide, it readily takes up two atoms of bromine, forming the dibromide, white needles melting at 119° C. On dissolving this substance in alcohol and adding caustic potash-lye, the solution turns reddish-yellow and then yellow, and, on the addition of water, a yellowish precipitate is obtained, which, after repeated crystallisation from benzene, forms white needles of 3-ethoxyflavone—



$$C_2H_5O.C_6H_3 < \begin{matrix} O-C.C_6H_5 \\ CO.\ddot{C}H \end{matrix} \quad (1:3:4)$$

On fusion with caustic potash the substance yields resacetophenone ethyl ether, benzoic acid, resorcinol ethyl ether, carbonic acid, and acetophenone, which is a further proof of the correctness of the above formula. When boiled with sodium alcoholate, the substance decomposes quantitatively into resacetophenone ethyl ether and benzoic acid, and continued boiling with hydriodic acid (1.7) yields 3-hydroxyflavone. This substance crystallises from alcohol in long shining needles melting at 240° C. It dissolves in dilute soda-lye with a very faint non-fluorescent yellow colour. The solution in concentrated sulphuric acid is

almost colourless and has a bluish fluorescence. The acetyl derivative forms white needles melting at 130° C. In view of the fact that the most important yellow vegetable dyestuffs contain the protocatechuic acid residue, the authors have condensed resacetophenone ethyl ether with piperonal, obtaining a product melting at 160° C., and crystallising in yellow or orange plates. The acetyl derivative (yellow needles) melts at 101° C., and the dibromide from this forms white needles melting at 130° C. On treatment with alcoholic potash, this substance gives a well-crystallised compound having the composition $C_{18}H_{14}O_5$. It is proposed to investigate these reactions in the phloroglucinol series.
—T. A. L.

### *Indigo, Artificial compared with Natural.* C. Kurz. Leipziger Färber- und Zeugdrucker-Zeit. **47**, [4], 1898, 179—182.

In an article taken from the Rev. Gén. des Mat. Colorantes, the author discusses the advantages of the pure indigo introduced by the Badische Anilin und Soda Fabrik, having dyed about 160,000 metres, for which 350 kilos. of artificial indigo were employed. Although the hydrosulphite reducing vat they recommend is too expensive for practical purposes, very good results were obtained by employing a bath made up from 22·5 kilos. of pure indigo (B.A.S.F.), 21 kilos. of zinc dust, and 42 kilos. of lime, and adding for every litre of the bath, 2 grms. of gelatin dissolved in a little water. This bath has been in use for over three months, and is still in good condition. Compared with dyeings from a natural indigo vat, it was found that the goods were dyed an equally heavy shade, the colour being much finer and brighter, less green, and just as red as with ordinary indigo, which, by comparison, appears dull and grey. The goods dyed with the artificial product do not rub so much, and, whilst being equally fast to washing and soaping, do not lose their brilliancy when exposed to light, but only become somewhat lighter. Moreover, much clearer discharges are obtained, and the deposit in the vat after three months is so small that the author reports it may be left for 5—6 months without renewing. A careful comparison of the cost of dyeing gave a result about 2·5—3 per cent. in favour of artificial indigo. It is stated that the B.A.S.F. are prepared to supply thousands of kilos. of the dyestuff, and have moreover entered into an arrangement with the Farbwerke Höchst, who will also be shortly in a position to supply indigo.

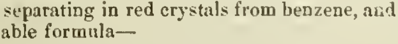
With regard to the determination of indigo, the author has again tested the various methods from that with aniline to that with acetic acid proposed by Brylinski. Although phenol gives good results, the best are obtained with naphthalene, providing that the filter in which the quantitative estimation takes place contains small garnets. The indigo then dissolves much more quickly and is less attacked. The indigo crystals obtained from aniline are thin and black; much better ones are formed from phenol; whilst naphthalene gives the best results.—T. A. L.

### *Lignone Dyestuffs and Coerulignone.* C. Liebermann and G. Cybulski. Ber. **31**, 615—621.

It has already been pointed out (Ber. **30**, 3137; this Journal, 1898, 238) that the lignone dyestuffs (Ber. **30**, 234; this Journal, 1897, 233) are too unstable for technical purposes. The alcoholic solutions only dye the fibre with great difficulty, and in presence of mineral acids the dyestuff gradually becomes decolorised. The sulphonic acids are still more unstable. Wool boiled in the dye-bath decolorises it; and this is even the case with faintly chromed or chlorinated wool. In alkaline solution there is a faint isonitrile odour. This instability is due to the quinone character of coerulignone and to the quinone-imide nature of the lignone dyestuffs, the action of acids resulting in the formation of substituted hydroquinones. Moreover, the leuco compounds of the group do not possess much tendency to regenerate the dyestuff by oxidation in the air, and in the case of the halogen-substituted hydrocoerulignones, this conversion cannot even be effected by the use of oxidising agents. Since the decomposition products of the lignone dyestuffs are usually tarry, and not readily purified (with the exception of Dimethyllignone Blue (from *p*-toluidine), which on boiling a short time with alcoholic hydrochloric acid, forms Leucochlorodimethyllignone Blue, $C_{23}H_{27}ClN_2O_4$), the authors have studied the action of reagents on coerulignone. On shaking this substance for some time with alcoholic hydrochloric acid, it yields a chlorhydro-ethylcedriret, $C_{20}H_{25}ClO_6$, or—

$$(C_2H_5O)_2(OH)C_6H_2.C_6HCl(OH)(OC_2H_5)_2$$

which crystallises from spirit in white needles melting at 130° C., and gives a diacetyl compound melting at 96° C. The lower homologue, $C_{16}H_{17}ClO_6$, is obtained by the use of methyl alcoholic hydrochloric acid, and melts at 141° C. The compound is formed by the addition of equimolecular proportions of hydrochloric acid and coerulignone, but the chlorine is so firmly attached, that its removal is not possible without otherwise changing the compound. Moreover, these chlorinated compounds can no longer be oxidised to the corresponding quinones. When the above ethyl compound, for instance, is treated with an excess of alcoholic ferric chloride, it yields a product melting at 159° C., separating in red crystals from benzene, and having the probable formula—



$$(C_2H_5O)_2(OH)C_6HCl.C_6H_2(OC_2H_5)\genfrac{}{}{0pt}{}{O}{O}> .$$

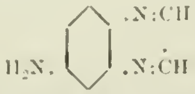
Similar products are obtained by substituting hydrobromic for hydrochloric acid, and probably the action of boiling acetic acid leads to the production of hydrocoerulignone, together with an oxidation product, although these substances have not been obtained in a sufficiently pure state for analysis. Coerulignone behaves similarly to the lignone dyestuffs with regard to reducing agents; these generally, however, give tarry products when boiled with alcoholic sulphurous acid, an exception being Leucohexamethyllignone Blue (from ψ-cumidine), which forms colourless needles gradually turning blue on the surface. In conclusion, two dyestuffs of the series are mentioned in addition to those previously described. The one formed from *o*-nitro-*p*-toluidine [78° C.], coerulignone and acetic acid has the formula $C_{28}H_{24}N_4O_8$, whilst the other is Methyllignone Blue, obtained from coerulignone and di-*p*-diamidodiphenylmethane.—T. A. L.

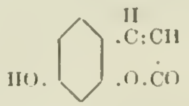
### *Monomethylaniline from Dimethylaniline, Formation of.* J. B. Cohen and H. T. Calvert. J. Chem. Soc. **73**, 163—167.

On further investigating the substance *phenylnitrocarbinol* (J. Chem. Soc. **71**, 1050), it was found that its action in most cases resembled that of nitrous acid. When added to an equivalent quantity of dimethylaniline, the mixture rapidly darkens, and nitrogen free from carbonic acid is evolved rapidly. With regard to the quantity of gas obtained, 1·0015 grm. of phenylnitrocarbinol and 2 grms. of dimethylaniline gave 13·5 c.c., whilst under similar conditions, 2·2225 grms. of the nitrocarbinol gave 27 c.c. of gas. The reaction product, which is deep red by transmitted and deep green by reflected light, was treated with hydrochloric acid and extracted with ether. The ethereal extract contains benzaldehyde, benzyl alcohol, and nitrosomethylaniline, 30 grms. of phenylnitrocarbinol giving on an average 5 grms. of benzaldehyde, 4·5 grms. of benzyl alcohol, and 5·5 grms. of nitrosomethylaniline. A very small quantity of a substance crystallising from water in brilliant golden plates and melting at 150° C. is also formed simultaneously. The nitrosomethylaniline, on reduction with stannous chloride, yields methylaniline. Since phenylnitrocarbinol on standing gives off nitrous fumes, the action of anhydrous nitrogen trioxide on dimethylaniline in dry ether was investigated. No nitrosodimethylaniline was formed, but a small quantity of the yellow compound described above, appeared to be produced. The chief product of the reaction is a substance which crystallises from hot alcohol in long steel-blue needles. It is decomposed by boiling with alcohol, and when boiled with glacial acetic acid for a short time, a rapid evolution of nitrous fumes takes place. On pouring the solution into water, a yellowish-green crystalline mass is obtained which, when crystallised from dilute alcohol, forms pale brown needles melting at 157° C.—T. A. L.

### *Fluorescence and Chemical Constitution.* R. Meyer. Ber. 31, 510—514.

The author attempts to show that the fluorescence in some groups of organic compounds, like their colour, is dependent on the presence in the molecule of certain groups of atoms for which he has proposed the name "Fluorophore." The existence, however, of these groups in a molecule is not by itself sufficient to cause fluorescence; they must occur amongst other groupings of atoms, for instance, benzene rings; moreover, substituting groups and the particular solvent also take part in the effect. Certain objections were raised by Pawlewski (Ber. 31, 310) to the author's statements, instancing as exceptions resorcinol-benzein, allo-fluoresceïn (Ber. 28, 108, 2360; this Journal, 1896, 193), benzylguaiacol, and a substance obtained by Pawlewski by the action of benzyl chloride on resorcinol. The constitution, however, of most of these substances is uncertain, and they cannot therefore really be considered. The author points out that a whole series of compounds exists to which in no sense his theory would apply, such as the fluorescent hydroxy-δ-amidonaphthalene sulphonic acids, certain quinoline derivatives and others, whilst it might be extended so as to include the thiazol ring, since dehydrothiotoluidine shows a violet-blue fluorescence in alcoholic solution. Two substances, 3-amidoquinoxalin and umbelliferone—





give fluorescent solutions, the fluorophore being in the one case the azine ring, and in the other a kind of pyrone, although in neither case does it lie between benzene rings, but is only attached to one. Neither quinoxalin nor coumarin fluoresces, although generally the effect of the introduction of a substituent is to weaken the fluorescence. It is found, however, that an alkaline coumarin solution, when allowed to stand, acquires a faint olive-green fluorescence. The author employs a U-shaped Geissler tube, containing nitrogen at 3 mm. pressure, the legs being about 10—11 cm. long. The best effects are obtained with a capillary tube, but for this an induction coil giving a 25-mm. spark is required. With a tube of 6 mm. diameter a coil giving a 6—7 mm. spark can be used, the primary current in either case being 4—8 volts. These tubes can be arranged in series, so that several observations may be made simultaneously.—T. A. L.

### *p-Dinitrodibenzyldisulphonic Acid.* C. Ris and C. Simon. Ber. 31, 354—355.

The authors, in continuation of a previous paper (Ber. 30, 2618), confirm in the main the statements of Green and Wahl (Ber. 30, 3097; this Journal, 1898, 146), but point out that the sodium salt of *p*-dinitrodibenzyldisulphonic acid is not converted by the action of hydrochloric acid into the free acid, but only into the acid salt. The free acid can be obtained by decomposing the barium salt with the theoretical amount of sulphuric acid and evaporating the filtered solution. It forms a mass of crystalline lamellæ very easily soluble in water, but not deliquescent. The pure sodium salt does not give a red coloration with caustic soda lye and phenylhydrazine, as Green and Wahl have pointed out. It is to be observed that when the reaction is performed with very strong soda lye a blue colour is produced, which, on dilution, turns red.

In a similar manner sodium dinitrostilbenedisulphonate, on decomposition with hydrochloric acid, only yields the acid salt, and the authors also find that analysis of the neutral sodium salt leads to the formula $C_{14}H_8N_2O_9S_2Na_2$; that is, it has the empirical composition of a nitrosonitro compound.—T. A. L.

### *Gallic Acid, Action of Formaldehyde on.* R. Möhlau and L. Kahl. Ber. 31, 259—266.

The authors find that by condensing formaldehyde with gallic acid, by means of hydrochloric acid, four different methylene-digallic acids are formed. Two of these (acids I. and II.) are crystalline, one being sparingly, the other easily soluble, whilst acids III. and IV. are amorphous, and also differ in their solubility. The acids can be converted into one another as follows:—The sparingly soluble crystalline acid I., and the easily soluble crystalline acid II., are convertible into the easily soluble amorphous acid III., and this into the sparingly soluble acid IV. Moreover, I., on standing in solution, is gradually converted into II. All these acids yield anhydrides; those from I. and III. have the same formula, $C_{15}H_{10}O_9$, but are different, and are formed by the elimination of one molecule of water from one molecule of acid; whilst the two anhydrides, both having the formula $C_{30}H_{22}O_{19}$, are formed from II. and IV. by the elimination of one molecule of water from two molecules of the acid. All four acids yield dyestuffs when acted on in sulphuric acid solution by nitrosulphonic acid, and the following table gives some of their reactions:—

| Solution in | Dyestuff from Acid I. | Dyestuff from Acid II. | Dyestuff from Acid III. | Dyestuff from Acid IV. |
|---|---|---|---|---|
| Conc. $H_2SO_4$ | Yellowish-red | Yellowish-red | Yellowish-red | Yellowish-red |
| $NaHCO_3$ | Violet | Violet | Violet | Violet |
| NaOH | Greenish-blue, unchanged at first, turning light yellow in 24 hours. | Blackish-blue, changing in a short time to dark green, and in 24 hours to dark yellow. | Blue<br>Unchanged at first, turning light yellow in 24 hours. | Blue |

It would thus appear that acids I., III., and IV. yield comparatively stable fluorone dyestuffs, whilst II. gives a comparatively unstable, possibly an aurin-like compound (diphenylmethane dyestuff).—T. A. L.

### *Formaldehyde-Trioxyfluorone-Dicarboxylic Acid.* R. Möhlau and L. Kahl. Ber. 31, 266—271.

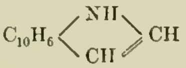
The sparingly soluble crystalline methylene digallic acid, which is one of the products of the action of formaldehyde on gallic acid (see preceding abstract), is obtained by dissolving 100 grms. of gallic acid in 1,125 c.c. of water, running in with constant agitation 375 grms. of hot concentrated hydrochloric acid and 60 grms. of 40 per cent. formaldehyde solution, and heating on the water-bath until no further precipitation of condensation product takes place. After cooling, the crude acid is filtered off, extracted with hot water, alcohol, and ether, and then forms a light crystalline powder, which is strongly electrical when rubbed. In order to convert it into the fluorone compound 10 grms. dried at 100° C. are dissolved in 300 grms. of sulphuric acid at a temperature not exceeding 70° C.; the solution is then cooled to 15° C., and the calculated quantity of nitrosulphonic acid is run in. The melt is allowed to stand 24 hours in a closed vessel, and the dyestuff is precipitated either by allowing the solution to absorb moisture, or by gradually adding dilute sulphuric acid. The product obtained by either method is well washed with water and extracted with alcohol, in which it is very sparingly soluble. The product forms a violet crystalline powder, slightly soluble in boiling water and very sparingly soluble in the usual solvents. It dissolves in concentrated sulphuric acid with a yellowish-red non-fluorescent colour, in alkaline acetates, carbonates, and borates with a violet, in ammonia with a blue, and in caustic alkalis with a greenish-blue colour. The calcium and barium salts are flocculent blue almost insoluble precipitates, whilst the uranium, cerium, nickel, cobalt, tin, aluminium, iron, and chromium lakes are insoluble and blue to greenish-blue in colour. The chromium lake, when treated with potassium bichromate and acetic acid, turns dark green. By heating the dyestuff with an equal weight of anhydrous sodium acetate and four times its weight of acetic anhydride for four hours in an oil bath, a tetra-acetyl compound is obtained, melting at 141° C.—T. A. L.

### *Naphthindole Derivatives, Synthesis of.* O. Hinsberg and A. Simcoff. Ber. 31, 250—254.

Certain amines, such as α- and β-naphthylamine, their mono-alkyl derivatives, and some alkylanilines, are converted by the action of the sodium bisulphite compound of glyoxal into indole sulphonic acids, glycocoll derivatives of the particular amine being formed simultaneously. The present paper describes the preparation of some β-naphthylamine derivatives. The principal product of the action of glyoxal sodium bisulphite is the sodium salt of β-naphthindole-sulphonic acid, whilst the compound—

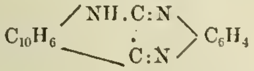
$$C_{10}H_7.NHCO.CH_2.NH.C_{10}H_7$$

is formed as a by-product. It crystallises from glacial acetic acid, and melts at 173° C. The salts of β-naphthindole sulphonic acid, when distilled with zinc dust, give β-naphthindole—



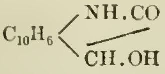
$$C_{10}H_6\left<\begin{matrix}NH\\ CH\end{matrix}\right>CH$$

which, when pure, forms colourless crystals melting at 40° C. The conversion of the sulphonic acid into β-naphthoxindole by warming with hydrochloric acid has already been described (Ber. 21, 113). By treating naphthindole potassium sulphonate in an aqueous solution acidified with acetic acid, with sodium nitrite, a brownish-red precipitate of isonitrosonaphthoxindole is obtained, which is purified by crystallisation from acetic acid. When 1 grm. of this compound is heated for 2½—3 hours with 20—30 c.c. of sulphuric acid boiling at 130° C., it yields β-naphthisatin (red needles from alcohol, melting at 252° C.), and on heating this product with *o*-phenylene diamine to 250° C., condensation takes place, forming naphthindophenazine—



$$C_{10}H_6\left<\begin{matrix}NH.C:N\\ \cdot\\ C:N\end{matrix}\right>C_6H_4$$

light yellow needles sparingly soluble in hot glacial acetic acid, and melting above 300° C. On dissolving naphthisatine in hot acetic acid and treating with zinc dust until decolorised, this compound yields naphthodioxindole—



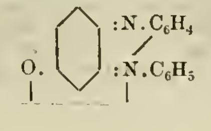
$$C_{10}H_6\left<\begin{matrix}NH.CO\\ |\\ CH.OH\end{matrix}\right.$$

which forms colourless crystals melting at 216° C. Its solutions oxidise in the air, re-forming naphthisatin.

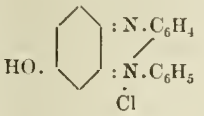
—T. A. L.

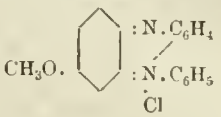
### *Safranines, Isorosindulines, and Rosindulines; Relations between the.* O. Fischer and E. Hepp. Ber. 31, 299—308.

The authors, in a previous paper (Ber. 30, 1827; this Journal, 1897, 1010), showed that rosindone or aposafranone, is converted by the action of phosphorus pentachloride into chloronaphthophenazonium chloride or chlorophenylphenazonium chloride. They now find that the reaction is a general one for all indones, and this fact, together with the behaviour of the indones and safranine bases to alkyl halogens, is evidence in support of the anhydride formula. If aposafranone has the formula—



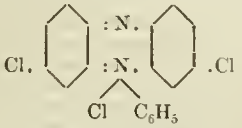
it must be possible to obtain alkyl halogen addition products possessing an analogous composition to the salts—



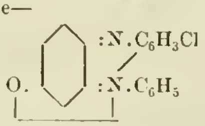


Isorosindone, phenylrosinduline, and phenylisorosinduline react in this manner, whilst rosindone behaves differently. By means of these compounds it is possible to pass from the isorosinduline to the rosinduline series, since when, for instance, the methylo-iodide of isorosindone is treated with alcoholic potash, it is converted into the methyl ether of hydroxyrosindone.

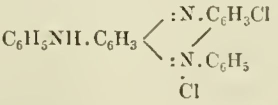
By treating safranol in 4—5 parts of phosphorus oxychloride with two molecules of phosphorus pentachloride, it is converted into dichlorophenylphenazonium chloride—



The three chlorine atoms react differently, the azonium one being easily replaced by Br, I, $NO_3$, &c., whilst of the two others, only one is in most cases easily replaceable. Thus, on boiling with sodium acetate, it is converted into chloro-aposafranone—



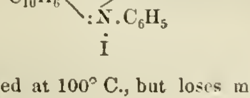
On treating the chloride with aniline, it yields the dyestuff—



$$C_6H_5NH.C_6H_3\left<\begin{matrix}:N.C_6H_3Cl\\ /\\ :N.C_6H_5\\ Cl\end{matrix}\right.$$

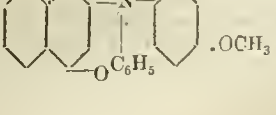
a characteristic reaction for the detection of safranolchloride.

Isorosindone (Ber. 29, 2755; this Journal, 1897, 132) gives, on treatment with one molecule of phosphorus pentachloride, chlorophenylnaphthophenazonium chloride, which reacts with aniline, forming phenylisorosinduline. This latter compound had already been obtained (*loc. cit.*). By heating phenylrosinduline with methyl bromide, an addition compound is obtained, which, on digesting in the cold with alcoholic potash, yields monomethylaniline and rosindone—a decomposition similar to that observed by Kehrmann and Schaposchnikoff (Ber. 30, 2628; this Journal, 1898, 145) in the case of dimethylrosinduline. Phenylisorosinduline also takes up one molecule of an alkyl halogen easily, but differs in its behaviour to alcoholic potash, being converted into methylphenylamidorosindone. When isorosindone is heated with methyl iodide, it yields isorosindone methyloiodide—



$$C_{10}H_6\left<\begin{matrix}:N.C_6H_3.OCH_3\\ /\\ :N.C_6H_5\\ I\end{matrix}\right.$$

which can be dried at 100° C., but loses methyl iodide at 170°—180° C., re-forming isorosindone. When this compound is treated with alcoholic potash, it is converted into methoxyrosindone—

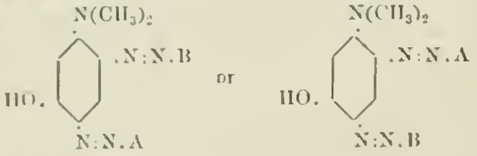


and this, when heated under pressure with hydrochloric acid to 170° C., yields hydroxyrosindone, the analogue of safranol. The methyl ether above described, is capable of again taking up methyl iodide, but the compound is very unstable, and gives up all its methyl iodide at 100° C.

It would appear from the foregoing that derivatives of the isorosinduline series are easily converted into rosindulines, but the latter show little or no disposition to undergo the inverse change. In a future paper the authors will describe further changes of this kind, as, for example, the conversion of isorosinduline into amidophenylrosinduline by means of aniline.—T. A. L.

### *Disazo Dyestuffs of the Benzene Series, New Primary.* C. Bulow and H. Wolfs. Ber. 31, [5], 488—494.

Primary disazo dyestuffs of the benzene series containing either hydroxyl or amido groups—*i.e.*, derivatives of resorcinol or of *m*-phenylene diamine—have long been known, the diazo groups most probably entering the 4- and 6-positions when the auxo-chromic groups OH or $NH_2$ are in the positions 1 and 3. Corresponding to the nature of the primary components, dyestuffs of the former class have faintly acid, those of the latter, basic characteristics. Dyestuffs possessing both acid and basic properties have not hitherto been known, and the authors describe a number of derivatives of this class obtained from *m*-hydroxydimethylaniline (dimethyl-*m*-amidophenol), when, according to the order in which the combination takes place, two isomeric dyestuffs can be formed—



where A and B are the radicles of aromatic amines. The two series of isomers do not greatly differ from one another, the shades being similar and the melting points being also close together. When the molecule only contains benzene radicles, the solution in sulphuric acid is brown to cherry-red, and, when diluted in the cold with water, turns green and then yellowish-brown. When, however, the dyestuff contains a naphthalene radicle, the undiluted sulphuric acid solution is green, and remains so until largely diluted with water, when it turns reddish-brown. The more methyl groups the dyestuff contains, the bluer the sulphuric acid solution. For instance, the colour in sulphuric acid of the dyestuff from 2 mols. of diazobenzene is brownish-red, whilst that from 1 mol. of diazobenzene and 1 of diazoxylene is decidedly bluish-red. In external appearance the isomeric substances differ in such a manner that those which are darker and more metallic have the greater number of side chains in the benzene nucleus para to the $N(CH_3)_2$ group.

The method employed consists in dissolving the monazo-compound (obtained by combining the diazo derivative with dimethyl-*m*-amidophenol in acid solution) in alcohol, adding potash and then the diazo compound. The new dyestuff, after stirring some hours, is precipitated with carbonic or acetic acid. For example, 3 grms. of benzene-azo-hydroxydimethylaniline, $C_6H_5N:N.C_6H_3(OH)N(CH_3)_2$ are dissolved in 150 c.c. of alcohol and 10 c.c. of 10 per cent. caustic potash, cooled to 0° C., and the diazo solution from 1·5 grm. of *o*-toluidine is allowed to run in. After standing 12 hours, carbonic acid is passed in, and the disazo dyestuff precipitates as a red mass. Repeated crystallisation from absolute alcohol yields reddish-brown crystals, melting at 140° C. The substance dissolves with a dark red colour in sulphuric acid, and, on the addition of a small quantity of water, turns green, becoming brownish-red on further dilution. It is insoluble in water, sparingly soluble in cold alcohol, petroleum ether, and glacial acetic acid; soluble in ether, benzene, and carbon bisulphide.

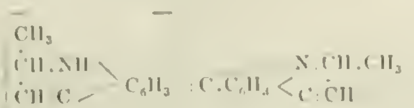
The following table gives the appearance and melting points of the various products obtained:—

| —— | 4. | 6. | Melting Point. | Crystallise from Alcohol in |
|---|---|---|---|---|
| | | | °C. | |
| $3.4.C_6H_3.OH.N(CH_3)_2$ | $.N:N.C_6H_5$ | $.N:N.C_6H_5$ | 136 | Blackish-brown crystals. |
| | $.N:N.C_6H_5$ | $.N:N.C_6H_4.CH_3(o)$ | 140 | Reddish-brown crystals. |
| | $.N:N.C_6H_4.CH_3(o)$ | $.N:N.C_6H_5$ | 124 | Brown plates. |
| | $.N:N.C_6H_5$ | $.N:N.C_6H_4.CH_3(p)$ | 149 | Greenish dark red crystals. |
| | $.N:N.C_6H_4CH_3(p)$ | $.N:N.C_6H_5$ | 144 | Blackish-brown plates. |
| | $.N:N.C_6H_5$ | $.N:N.C_6H_3(CH_3)_2(o\text{-}p)$ | 142 | Brownish-green crystals. |
| | $.N:N.C_6H_3(CH_3)_2(o\text{-}p)$ | $.N:N.C_6H_5$ | 181 | Dark bronzy plates. |

—T. A. L.

### *The Glauconic Acids: A New Group of Quinoline Dyestuffs.* O. Doebner. Ber. 31, 686—696.

By heating together aniline (1 mol.) and pyruvic acid (2 mols.) in alcoholic solution for 15 minutes, until the rapid evolution of carbonic acid ceases, and then adding formaldehyde, a condensation product—hydroglauconic acid—is obtained, which, after precipitation with ether, forms a granular crystalline yellow powder. On boiling for a short time with 10 per cent. soda lye, it yields, on simultaneous oxidation by the air, a blue salt of glauconic acid, which, on treatment with mineral acids, gives the free acid in dark blue needles having the formula $C_{31}H_{29}N_3O_6$, its constitution being probably as follows:—



Both acids, on heating alone or with soda lime, yield dihydroquinaldine. The condensation only takes place with those amines in which both positions ortho to the nitrogen are free. Thus *o*-toluidine does not give the dyestuff acid, nor does α-naphthylamine; but *p*-toluidine, *p*-anisidine, *p*-phenetidine, *m*-toluidine, and β-naphthylamine do. Hydroglauconic acid is obtained by dissolving 50 grms. of aniline in 150 grms. of absolute alcohol, and gradually adding 100 grms. of pyruvic acid. The mass turns yellow and a rapid evolution of carbonic acid takes place. After about 15 minutes, 50 grms. of a 40 per cent. formaldehyde solution are run in, when the mass turns brownish-yellow and a further evolution of carbonic acid takes place. The whole is then warmed for about an hour, the evaporated alcohol being replaced, and, after cooling for six hours, the dark brown syrupy residue is stirred up with a little alcohol and treated with an excess of acetone. The hydroglauconic acid separates in lumps, which are washed with ether and dried in a vacuum. The yield is about 90 grms. The glauconic acid is best obtained by heating 10 grms. of the hydro acid with 10 c.c. of alcohol, adding 100 c.c. of 10 per cent. soda lye, raising to the boil, then adding 15 grms. of a 40 per cent. formaldehyde solution, and boiling for another minute. An excess of alcohol is then added to the dark blue mixture, and on standing several hours the sodium salt of glauconic acid separates in blue metallic needles, which, after washing with alcohol and ether, are dried *in vacuo*, the yield from the above quantity being about 5 grms. The salts of glauconic acid have no technical value on account of their instability to acids,

soap, and light, but they are pronounced mordant dyestuffs. For instance, the dyestuff from *p*-toluidine gives light blue shades on mordanted cotton and reddish-blue on unmordanted cotton with sodium acetate or sulphate, but is not fast to soap; with a cerium mordant the blue is somewhat more stable. Wool is dyed light blue from a neutral bath, violet with an alum mordant, and bluish-green with chrome and tartaric acid, but all these shades are changed to violet-red by ammonia. The action of pyruvic acid on *p*-toluidine in presence of formaldehyde proceeds more easily than with aniline, and hydro-*p*-methyl-glauconic acid and the corresponding *p*-methyl-glaucouic acid are well-crystallised substances. On the other hand, the yield of the glauconic acids from *p*-phenetidine and β-naphthylamine is much smaller, and the conversion of the hydro acid into the dyestuff acid proceeds much less completely.
—T. A. L.

### *Yellow Colouring Matter of the Leaves of Arctostaphylos uva ursi.* A. G. Perkin. Proc. Chem. Soc. 1898, [193], 104.

Kawalier (Jahr. 1852, 685) found the leaves to contain, besides gallic acid and arbutin, a glucoside, ericolin, $C_{34}H_{56}O_{21}$, which yields, on decomposition, ericinol, $C_{10}H_{16}O$, and a sugar. More recently, B. Degraffe (Am. Jour. Pharm. 1896, **68**, 313) has identified the tannin as gallotannin. There is also present a yellow colouring matter of the composition $C_{15}H_{10}O_7$, crystallising in glistening yellow needles; this forms an acetyl compound, $C_{13}H_5O_7(C_2H_3O)_5$, melting at 188°—190°. On fusion with alkali, phloroglucinol and protocatechuic acid were formed. Though resembling quercetin in these points, it has the property of forming deep green solutions with dilute potassium hydrate. Regarding this reaction as due to impurity, the colouring matter was converted (*a*) into a sulphate, and (*b*) into an acetyl derivative, and the colouring matter regenerated from these, but in each case it was unaltered in its behaviour towards alkaline solutions. Oxidation in alkaline solution did not destroy the green coloration until complete decomposition of the colouring matter had taken place. The presence of ellagic acid has also been detected, and thus besides gallotannin, ellagitannin is also present. Broach leaves contain the same colouring matter.

### *Sicilian Sumach, Yellow Colouring Matters of Various Adulterants of. Part V.* A. G. Perkin and P. J. Wood. Proc. Chem. Soc. 1898, [193], 104—105.

This paper describes an investigation of the leaves of those plants which are employed for the adulteration of Sicilian sumach (*Rhus coriaria*). The authors were indebted for samples of these to Mr. P. Gennadius, Director of Agriculture in Cyprus.

The leaves of *Pistacia lentiscus* (shinia) contain a colouring matter of the formula $C_{15}H_{10}O_8$, forming an acetyl compound, $C_{15}H_4O_8(C_2H_3O)_5$, crystallising in colourless needles (m.p. 204°—206°). As on decomposition this yields gallic acid and phloroglucinol, it is identical with myricetin, the colouring matter of *Rhus coriaria*. Two tannins appear to be present. Shinia leaves contain 11·3 per cent. of tannin, and appear to be a useful tanning agent, though not suitable for the same purposes as sumach.

The leaves and stems of *Tamaris gallica* and *T. africana* (bruca) contain the same colouring matter. It has the formula $C_{16}H_{12}O_7$. The tannin (8·4 per cent.) is evidently a mixture of ellagitannin and gallotannin, for both ellagic and gallic acids were isolated as its decomposition products.

The leaves of *Ailantus glandulosa* contain quercetin. The tannin present (11·9 per cent.) is a mixture of ellagitannin and gallotannin, for both ellagic and gallic acids were isolated. This is a worthless tanning agent; the skin though stained a deep colour is practically untanned.

The colouring matter of the leaves of *Ficus carica*, the common fig-tree, though resembling quercetin, could not be identified, as from 2 kilos. only 0·08 grm. of the substance was obtained. It is nearly devoid of tannin (analysis gave 1·6 per cent.), skin being untanned though stained a dirty olive colour.

*Gambuzzo*, the stalks of the *Rhus coriaria*, contain a trace of myricetin and some gallotannic acid.

*Brouch leaves* are employed in South Africa in place of sumach (*R. coriaria*), and are also replacing Cape sumach (*Colpoon compressum*) (Trans. 1897, **71**, 1132). They contain 19·9 per cent. of a catechol tannin, and they form a valuable tanning agent. The colouring matter has the formula $C_{15}H_{10}O_7$, and appears to be identical with the colouring matter of *Arctostaphylos uva ursi* (see preceding abstract).

The galls of *Pistacia terebinthus* contain a trace of myricetin, whereas Mangrove cutch (*Ceriops candolleana*) is devoid of yellow colouring matter.

### *Indigo, Analysis of.* C. Kurz. Rev. Gén. des Mat. Col. 1898, **2**, 91.

*See under* XXIII., *page* 495.

## PATENTS.

### *Dyes for the Beard and the Hair, New.* H. Richez, Paris, France. Eng. Pat. 6549, March 12, 1897.

Mineral salts (silver salts) and sulphur, or vegetable colouring matters, are stirred into a mixture of wax and fat, diluted to a proper consistency with glycerin, and cast in stick form or other shapes.—T. A. L.

### *Tetrazo-azo Compounds [Reds], A Process for the Production of New.* A. M. Clark, London. From Kullmann and Rapp, Rotterdam, Holland. Eng. Pat. 8994, April 8, 1897.

One molecular proportion of a tetrazotised diamine is combined with one molecular proportion of a diazotised sulphonic acid of aniline or its homologues or naphthylamine, or with a "diazotised, nitrated, amidated, or oxylated amine." The tetrazo-azo compound so obtained is then combined with two molecular proportions of a dyestuff component, which may be similar or different. For instance, 9·2 kilos. of benzidine are converted into tetrazo diphenyl chloride, which is run into "a solution" of the diazobenzene sulphonic acid from 8·6 kilos. of *p*-sulphanilic acid. On neutralising the free mineral acid with sodium acetate, the tetrazo-azo compound separates in dark brown flakes, and when combined with two molecules of naphthionic acid in a faintly acid solution, it yields a colour which dyes unmordanted cotton a Turkey-red shade from an alkaline bath.—T. A. L.

### *Basic and Phenolic Colours for Printing, Impts. in Treating and Fixing.* C. E. Gassmann, Mulhouse, Alsace. Eng. Pat. 9392, April 13, 1897.

Colouring matters of a basic or phenolic character which are little soluble or insoluble in water are dissolved in phenols or amines, and the thickened solution printed in the usual manner. On subsequent steaming, the solvents are volatilised and the colours fixed on the fibre. In other cases an addition of phenol, &c., is made to a printing colour otherwise prepared, and containing an insoluble colouring matter of the classes mentioned above, *e.g.*, benzene-azo-salicylic acid. The invention refers to colours produced with or without the aid of mordants. The substances claimed as being suitable solvents are phenol, cresol, phenolic ether, creosote oils, or amines such as aniline and its liquid derivatives, tar-quinoline, pyridine, &c.—R. B. B.

### *Colouring Matters [Blues], Impts. in the Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. Friedr. Bayer and Co., Elberfeld, Germany. Eng. Pat. 10,605, April 28, 1897.

Dyestuffs giving fast greenish-blue shades on wool from an acid bath are obtained by combining the diazo derivatives of the so-called peri-amidonaphthol sulphonic acids with the 1.4′.3′ or 1.4′.2′-amidonaphthol sulphonic acids in an acid or neutral solution. Of the peri acids, the following are suitable for this purpose:—the 1.1′.3.3′-, 1.1′.4.3′, 1.1′.2.4-, 1.1′.3.4′-amidonaphthol disulphonic acids and the 1.1′.4-, 1.1′.3-, and the 1.1′.4′-amidonaphthol sulphonic acids. The resulting dyestuffs are dark bronzy powders.—T. A. L.

*Logwood, Improved Extract of, and Method of Obtaining the same.* J. Y. Johnson, London. From F. E. Bucher, Jamaica, West Indies. Eng. Pat. 75, Jan. 1, 1898.

Logwood extract is treated with a current of nitrogen dioxide or trioxide in order to convert the hæmatoxylin, $C_{16}H_{14}O_6$, into hæmatein, $C_{16}H_{12}O_6$, which gives superior results in dyeing. The patentee finds that for the conversion of 100 grms. (suspended in 2 litres of water at the ordinary temperature) of the former into the latter, it is sufficient to pass in 50 grms. of nitric peroxide, $NO_2$, or 80 grms. of nitrogen trioxide, $N_2O_3$. In actual practice the gas obtained by decomposing 70 lb. of nitric acid (80° Tw.) with 20 lb. of sugar at 80° C., is diluted with a current of air and passed into 1,000 lb. of logwood extract (20° Tw.) at the ordinary temperature. When the reaction is complete, 50 lb. of soda ash are added, and the product may then be used or may be concentrated. As an alternative method the acid liquor is allowed to stand 12 hours, when the hæmatein separates out, the acid liquor being neutralised and concentrated. The commercial hæmatein so obtained has a very high tinctorial value.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Wool, The Action of Caustic Soda on.* A. Buntrock. Färber Zeit. 9, (5), 69—71.

Samples of woollen yarn having a tensile strength of 610 grms. were treated with caustic soda solution of varying strength for 10 mins. They were then quickly washed with a 1 per cent. solution of sulphuric acid, followed by a washing with water, after which they were dried, and finally their tensile strength was redetermined.

When lyes of 4°—38° B. were used, the wool was found to rapidly disintegrate, and after 10 mins., so long as the yarn remained wet, the fibres could be drawn out to double their length. On drying, the wool was very much felted and had greatly decreased in strength. Caustic soda solution of 20° B. exerts the most powerful action: the tensile strength of the dried wool is reduced to 95 grms.

Solutions of caustic soda of specific gravity 38°—50° B. do not felt the wool or change its appearance, whilst the tensile strength gradually increases until a maximum of 815 grms. is reached at 42° B. It then declines, and at 50° B. is reduced to 620 grms.

If allowed to act for a longer time than 10 mins., the tensile strength is greatly reduced. Thus a lye of 42° B. in 15 mins. gives a wool breaking under 760 grms., in 30 mins. under 715 grms., and in 60 mins. under 540 grms.

The addition of glycerin to the lye hinders the weakening action. Caustic soda of 42° B., when mixed with an equal quantity of glycerin, gave in 5 mins. wool breaking under a load of 830 grms.; in 10 mins., 870 grms.; in 30 mins., 790 grms.; in 60 mins., 740 grms.

The addition of from 25—50 per cent. of glycerin to more dilute lyes was likewise effective, causing the wool to be less energetically attacked.

Wool which has been treated with concentrated alkaline solutions, shows a greater affinity for colouring matters than ordinary wool.—H. I.

*Fur Hat (Haarhut) Making: Dressing the Fur.* Deutsche Hutmacher Zeit. 29, [14] and [15].

The mercury dressing for the treatment of the fur previous to cutting and felting is composed of 1·250 kilos. of mercury and 5 kilos. of commercial (90 per cent.) nitric acid for 1,000 heavy hare skins; or 0·875 kilo. of mercury and 2·5 kilos. of nitric acid for 1,000 heavy rabbit skins, the mixture being prepared beforehand and diluted, preferably with soft water, until it indicates 12 per cent. on the acidimeter, for use. The strength of the dressing depends directly on the proportion of nitric acid and the degree of concentration. It is brushed into the fur on the skins until about one half the length of the hairs from the top downwards is moistened. The skins for white dressing are then spread out to dry slowly in the air without artificial heat, except in winter; whilst in yellow dressing they are dried in an arched oven heated by a coke fire. When dry, the skins are moistened on the back with water, spread out flat, and the matted hairs brushed out straight. After being well beaten they are then ready for cutting. —C. S.

*Hats, The Carbonising of Woollen Felt.* C. A. Otto. Färber Zeit. 9, 83.

The carbonising, especially of the lower grade wools employed in the manufacture of felt hats, is most economically carried out on the half-milled felts by treating them for one hour in sulphuric acid at from 4° to 5° B. After thorough hydro-extraction, they are dried and carbonised at a temperature beginning at 50° C. and finishing at 70° C. Carbonisation being completed, the felts are returned to the milling process, after 15 minutes washing in warm water. The milling process is followed by a bath of sodium carbonate at 2° B., washing in warm water, to which, after half an hour, from 1 to 1·5 litres of ammonia are added, and finally washing in tepid and cold water. The felts coming from the washing machine may at once be mordanted with bichromate and cream of tartar; or, if they are stiffened before dyeing, they should be boiled for 30 minutes with 300 grms. of sodium bichromate and 300 grms. of cupric sulphate. They may then be dyed with logwood or any black coal-tar dye without any danger of spotting or damage to the stiffening.—C. O. W.

*"Acetone Oil" and Ketones, Preparation of [from Wool Washings].* A. and P. Buisine. Rev. de Chim. Industr. 1898, 9, [100], 112.

*See under* XX., *page* 485.

*Weighting in Black Silks, Determination of the.* R. Gnehm and E. Blumer. Rev. Gén. des Mat. Col. 1898, 2, .

*See under* XXIII., *page* 495.

*Weighting in Silk, Determination of the.* R. Gnehm and G. Schwartz. Rev. Gén. des Mat. Col. 1898, 2, .

*See under* XXIII., *page* 495.

### PATENTS.

*Mercerising Cotton Yarns, An Improved Method of.* O. Imray, London. From The Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 10,784, April 30, 1897.

In order to prevent contraction of the fibre during mercerising, instead of performing the operation under strong tension, as is now usually done, an addition is made to the caustic soda of sodium silicate or aluminate. It is stated that this addition renders unnecessary a special tension, the contraction being very slight and the original length easily restored by means of a gentle tension after washing. The mixture recommended consists of 100 parts of soda lye of 28° B., and 10 parts of silicate of soda of 41° B.

It is claimed that yarns of cheap quality can be mercerised by this process without risk of tearing the thread. The supposition that the addition to the caustic soda acts merely by diluting the latter, is not confirmed by experiments, in which much weaker caustic soda, without addition, was found to produce a far greater contraction of the fibre than the mixture referred to above.—R. B. B.

*Silkworm Gut, Impts. in and relating to the Manufacture of.* M. Carswell, jun., Glasgow. Eng. Pat. 12,004, May 14, 1897.

This invention relates to a method of obtaining silkworm gut for fishing purposes in longer lengths than have been hitherto produced. The present length of gut usually measures about 24 ins., although certain species of silkworm produce longer gut; but in all cases a bend or curl occurs about 6 ins. from each end, which greatly reduces the serviceable length. According to this patent, the worms producing the greatest length of gut are opened at the proper stage and the silk sacs removed. The ends of these sacs are, in the worm, doubled back, and it is this fold that produces the curl in the original gut. Each sac is straightened out in a line with its centre portion, before applying the liquid which causes solidification. The result is that lengths of 30—40 ins. of gut are obtained.—R. B. B.

*Preparations* [*"Miroitine"*] *for Laundry Purposes, Impts. in or relating to.* A. J. Boult, London. From La Soc. "Meurice-Save-Simon," Brussels. Eng. Pat. 17,405, July 23, 1897.

This substance, consisting of magnesium sulphate, is stated to give a gloss to linen.—F. H. L.

*Textile Materials, Paper, Wood Substances, and the like; An Improved Process for rendering Waterproof, and for Finishing and Protecting them against Spotting and Moths.* W. P. Thompson, Liverpool, &c. From J. Rudolf, Gera-Reuss, Germany. Eng. Pat. 30,916, Dec. 31, 1897.

Substances insoluble in water, such as hydrocarbons, stearin, vegetable waxes, or resins, are either—

(*a*) made into an emulsion in hot water by violent agitation by means of centrifugal or other device, the articles being then saturated with this emulsion; or

(*b*) the substances are melted in a heated trough and conveyed on to the materials by means of a pressure roller. —R. B. B.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Chrome-tanned Leather, The Dyeing of.* F. Kast. Färber-Zeit. **9**, 82.

Chrome-tanned leather is becoming increasingly important, especially on account of its great durability. This leather, which can be produced within about 36 hours, possesses, nevertheless, a strength scarcely attainable by other tanning processes, and it resists heat far better than ordinary leather. The dyeing of chrome leather offers, however, considerable difficulty, chiefly as regards evenness of shade. To overcome this difficulty, Avellis and Kuster, of Berlin, subject this leather, first to a lime treatment, followed by washing and a subsequent treatment in a tannin bath. The leather so treated is now ready for dyeing, but clearer shades are obtained if the treatment in the tannin bath be followed up by a treatment with tartar emetic. A complicated process, consisting in successive treatments with alumina, tin, chromium, and iron mordants, was described in Kampfmeyer's Journal, producing leather especially suitable for dyeing with alizarin dyes at a temperature of 60° C. A process of preparing the leather with sodium salts, followed by a treatment with a chromium mordant, seems to be quite unknown. Leather so treated, dyes readily and evenly in solutions of the acid dyestuffs. Addition of acid to the dye-baths produces clear shades; dull shades are obtained in neutral baths. Azo Acid Yellow, Curcumein Extra, Ponceau 4 G B, Ponceau 3 R B, Fast Brown G, Guinea Green B, Fast Blue 6 B, and Wool Black 4 B all operate very well in this manner. The dyeing is carried out at 50° C., either by dipping or brushing on. After dyeing, the leather is finished with a coat of linseed oil and ironing of the half-dried leather, which finally receives once more a light dressing with grease.—C. O. W.

*Felt Hats, Dyeing of Ladies'.* C. A. Otto. Färber-Zeit. **9**, [8], 115—116.

Ladies' hats are dyed, either (1) when the hat bodies are only half-felted, or (2) when they are in the finished state.

The first method is adopted when it is desired to dye the hat bodies well through. In this case rather more dyestuff is needed, and the hat bodies must be felted with the aid of weak sulphuric acid.

The dye-bath is made up with the necessary amount of dyestuff and $\frac{1}{2}$—1 litre of sulphuric acid (168° Tw.) for every 25 kilos. of water.

Enter the felted hat bodies at 50° C., handle them well while raising the temperature to boiling point, and boil 1—1$\frac{1}{2}$ hours. Take out the felts, throw them into lukewarm water, and cool slowly by adding cold water.

Throwing the felts at once into cold water must be avoided, as the felting is thereby partially or wholly prevented.

Acid dyes are employed, which easily dye level, and are well fixed on the fibre by long boiling; otherwise, in polishing and lustreing, much dyestuff would be lost, and the goods must be again dyed.

Dark colours, black especially, are dyed in the piece. Thus less dyestuff is needed, and the felting proceeds better.

The directions for dyeing the finished hat bodies are as follows:—For 25 kilos. of goods, make up the dye-bath with 10 kilos. of Glauber's salts, 1 kilo. of cream of tartar, and the necessary amount of dyestuff.

Enter the well-wetted hat bodies at 50° C., handle $\frac{1}{4}$ hour, raise to the boil, and continue $\frac{3}{4}$—1 hour. Then take out the bodies, add 1—2 kilos. of cream of tartar, according to the depth of shade required, re-enter, and boil until the desired tone is obtained.

For very dark shades, add $\frac{1}{2}$—1 kilo. of sulphuric acid (168° Tw.) in addition.

Hat bodies which have been felted with sulphuric acid are either boiled out with $\frac{1}{2}$ litre of ammonium chloride before dyeing, or the addition of the cream of tartar is omitted in dyeing. After dyeing, the hat bodies are thrown into cold water, well hydro-extracted, and, with light shades especially, dried at not too high a temperature.

Acid dyes are used in the dyeing of the finished hat bodies, and the author finds that the colours are fresher and more striking than those obtained on dyeing in the felt.

Alizarin dyes also give good results, but the colours are not so bright.—J. E. H.

*Velvet, Dyeing of.* E. Hatlanek. Färber-Zeit. 1898, **9**, [7], 103—104.

For light mode shades, the direct cotton dyes have come more and more into favour, *e.g.*, Diamine Bronze, Diamine Green, Direct Yellow G, Pyramine Orange, several fast light browns, &c.

However, the basic dyes are preferred, because of their greater brilliancy and fulness of tone, especially when viewed overhand.

Of the single shades, green is particularly important.

Brilliant Green, Victoria Green, and Malachite Green are admirably adapted for the dyeing of light pure green shades.

Unfortunately for darker shades, the above greens are not so suitable, since they are not fast to rubbing.

In the author's experience, Diazine Green is much faster to light and rubbing than the greens mentioned above.

The following is the mode of procedure for dyeing a green on velvet:—

For two pieces of velvet, weighing about 15 kilos., dissolve 400 grms. of tannin in hot water, and add to a bath containing 100 litres of water. The dry goods are entered into the jigger, nearly at the boil.

Run the goods through the bath until they acquire the temperature favourable for the fixation of the tannin (*viz.*, 50°—60° C.).

Take out the goods, hydro-extract, and fix with 180 grms. of tartar emetic at 35° C. Thoroughly wash the goods. Enter the dyestuff, consisting of 450 grms. of Diazine Green, 90 grms. of Brilliant Green, 150 grms. of Thioflavin T., and 100 grms. of alum, into the hot bath of the jigger. Introduce the goods, and dye at about 80° C.

After dyeing, the goods are rinsed with water in the jigger, hydro-extracted, dried, and finished as usual. —J. E. H.

*Synthetical Indigo, Dyeing of Cotton with.* C. Kurz. Rev. Géo. des Mat. Col. 1898, **2**, 90—91.

The author gives the results of a comparative dye trial, extending over three months' time, which he has made with the "Indigo pure" of the Badische Anilin und Soda Fabrik (this Journal, 1898, 41; also see page 447) and natural Indigo.

A stock solution was prepared as required, with 22·5 kilos. of "Indigo pure," in powder, 21 kilos. of zinc dust, and 42 kilos. of quicklime; 2 grms. per litre of gelatin being added to the vat. With this the vat was set, and maintained during the period of the trial, no additional quantity of reducing agent being at any time required; 160,000 metres of calico were thus dyed a medium shade of Indigo with an expenditure of 350 kilos. of the synthetical product.

The vat remained clear throughout the trial. The shade of blue obtained was quite as dark as that dyed with the same quantity of refined, natural Indigo on a similar quantity of the same kind of calico. It was, moreover, brighter, and less greenish in shade, and it gave a purer white when discharged than the latter. Further, although the two colours were found to resist washing, soaping, rubbing, and exposure to light about equally, the colour dyed from the synthetical compound possessed the advantage of not altering in hue whilst fading, whereas the blue obtained from natural Indigo became greyish and dull on exposure to light.

The relative cost of dyeing was 2·5 to 3 per cent. less with the synthetical than with natural Indigo.

The author has printed 80,000 metres of calico with "Indigo pure" without experiencing any difficulty.—E. B.

### *Stoved or "Sulphured" Colours.* E. Stobbe. Färber-Zeit. **9**, [5], 71—73.

It had been customary, previous to the discovery of Rhodamine, to dye such shades as Salmon, Maize, and Chamois with mixtures of Auramine and Safranine in the soap-bath, and afterwards to stove. The latter colour, Safranine, is very fugitive to light, and Rhodamine was tried as a substitute. This dyestuff will not, however, dye with soap, but gives good results in an acid or neutral bath, even shades being obtained at temperatures below the boiling point. The soap bath was neglected, and mixtures of Rhodamine and Auramine were dyed in neutral baths. It was soon found that Auramine alone would give good results without the addition of soap, and in this way various shades of "sulphured" yellow were obtained.

Rose colours of blue shade, such as Camelia and Azalea, were difficult to dye. They were formerly obtained by topping Rhodamine with Phloxin or Rose Bengal, and stoving, but were very fugitive to light, and did not dye evenly. In 1892, several colouring matters were introduced with which a rose, fast to light and stoving, could be dyed.

*Violamine R R* and *Fast Acid Violet A 2 R* (this Journal, 1893, 38) are acid colours dyeing wool only in an acid bath, giving very brilliant and even results in the lightest shades. The former behaves like Rhodamine on stoving—light shades become bluer, and darker shades more yellow and fiery.

*Fast Acid Violet A 2 R* in dark shades is unchanged by stoving, but light shades become bluer.

*Rhodamine 3 B* is a basic colour, very similar to *Violamine R R* in shade, and is dyed in the neutral bath with the addition of Glauber's salts at the boiling temperature.

The so-called sulphured or stoved colours (Schwefelfarben) are not to be confounded with colours *fast* to stoving (Schwefelecht), such as are dyed on worsted for ladies' dress goods. Coloured yarns are woven with raw yarn, and the fabric stoved to bleach the latter.

Such colours must, of course, be fast to stoving, and must not "bleed" when subjected to moderate milling.—H. I.

### *Indigo, Process for the Economising of, in Dyeing* Wool *in the Indigo Vat.* V. H. Soxhlet. Leipziger Färber- und Zeugdr. Zeit. 1898, **47**, [11], 131—132; also Ger. Pat., July 18, 1896. Class 8. No. 91,035.

In order to obtain deeper shades of indigo, various attempts have been made to oxidise the wool fibre by means of bleaching-powder, vanadium and cerium salts, but they have all proved failures. On treating wool with permanganates, and subsequently dyeing in the indigo vat, better results were obtained; but on washing the dyed fabric repeatedly with soap, alkaline or reducing solutions, the manganese lake was removed from the fibre.

If, however, the wool be mordanted in either of the following ways—(1) by treating with a solution containing 5 per cent. of potassium permanganate, 1 per cent. of magnesium sulphite, and 2 per cent. of aluminium sulphite at 50° C.; or (2) by treating with a solution of 5 per cent. of potassium permanganate, and then, for 15 minutes, in a separate bath containing 10 per cent. of chromium sulphite at 25° C., on dyeing in the indigo vat, shades are obtained varying in intensity with the concentration of the mordant solution, and fast even to concentrated alkaline solutions.

The inventor claims that the above method economises the indigo.—H. I.

### *Aniline Black, Dyeing Vegetable Fibres with.* V. H. Soxhlet. Leipziger Färber- und Zeugdr. Zeit. 1898, **47**, [9], 97—99.

After reviewing the various attempts made to avoid the use of copper sulphate, or to prevent its objectionable action on the fibre in dyeing with aniline black by the oxidation method, the author states that they have been for the most part failures. If copper sulphate were not employed, the colours obtained were weak, and of bad tone; if used, the fibre was tendered.

Quite recently a method has been discovered whereby it it possible to retain the copper sulphate and still protect the fibre. To the ordinary mixture for aniline black—consisting of aniline oil, hydrochloric acid, copper sulphate, sodium chlorate, and ammonium chloride, the last three being dissolved in water before mixing with the aniline hydrochloride—when cold, a solution of aluminium acetate is added, in proportions depending on the strength of the preparation used. It is very important that the acetate should be free from iron, and fairly rich in alumina (3½—4 per cent. of $Al_2O_3$).

The whole mixture for the black, when ready, should show a specific gravity of 7° B. The dyeing of the cotton is then proceeded with as usual. In the warm oxidation room, the acetic acid of the alumina compound volatilises; the alumina set free combines with the sulphuric acid of the copper sulphate, and is removed during the usual chroming and soaping processes as aluminium sulphate by the water. Thus, on drying, its action on the fibre is prevented.

In this way a good black is obtained, free from the objectionable features so often met with in aniline black dyed cotton.—H. I.

### *Paranitraniline Red, Discharging.* [*Calico Printing.*] E. Lauber. Leipziger Farber- u. Zeugdr. Zeit. 1898, **47**, [2], 55—56 and 85.

H. Schmid's white discharge colour-mixture (this Journal, 1897, 534) is prepared with 90 kilos. of Discharge White P N (Hoechst Farbwerke; Eng. Pat. 8860 of 1896; this Journal, 1897, 438), 5 kilos. of finely-ground wheat starch, and 3 kilos. of British gum. The starch and British gum are mixed with part of the Discharge White, the rest of this is added, and the whole is heated in an enamelled or earthenware vessel.

Black is produced along with white by printing a vanadium Aniline Black mixture or a steam Aniline Black mixture containing potassium ferrocyanide. With the first mixture the printed tissues are hung over night at a temperature of 40° C. (35° C. by the wet-bulb hygrometer), and are then steamed for 7—8 minutes in a steam Aniline Black ageing machine. In this way, the Paranitraniline Red becomes partially discharged before steaming, and on steaming is easily discharged without the fibre being injured.

Blue is obtained with 20 kilos. of White Discharge printing mixture, 3 kilos. of gum tragacanth paste (1:10), and 3 kilos. of Phenocyanin R paste. The last two are mixed and gradually added to the White Discharge mixture. Gallamine Blue gives a shade more nearly resembling one of Indigo, especially in the presence of a chromium salt.

To produce yellow, a mixture of 20 kilos. of White Discharge colour and 2 kilos. of Persian berries' extract at 30° B., or 600 grms. of Xanthaurin, is employed. The latter dyestuff gives a brighter and purer colour than the former, and this may be rendered faster to light than it otherwise would be, by the addition of a chromium salt to the printing mixture.

A mixture of 20 kilos. of the White Discharge printing mixture, 2 kilos. of gum tragacanth paste, 20—40 grms. of Brilliant Green, 500 grms. of Phenocyanin R, and 1 kilo. of Persian berries extract at 30° B., gives a green colour.

The blue dyestuffs used for the blue and green discharges become reduced to leuco-compounds on steaming. They are re-oxidised on washing (in running water). Probably hydrogen dioxide, if not too costly, could be usefully employed to effect this oxidation, the employment of alkali bichromates not being practicable on account of their injurious action on the lakes of the natural yellow dyestuffs. —E. B.

*Lustre-Cotton, Dyeing of.* C. Zinn. Leipziger Färber- u. Zeugdr. Zeit. 1898, **47**, [2], 47—53.

Cotton-substantive dyestuffs are very suitable for dyeing lustre-cotton, as they do not, like other dyestuffs, affect the gloss of the fibre.

In applying them, care must be taken that dyeing does not take place rapidly, and, consequently, unevenly. It should be performed at as low a temperature as possible, and some time (half an hour) should be allowed to elapse before any addition of sodium sulphate is made to the baths.

The latter are prepared with 1 per cent. of sodium carbonate, 1—2 per cent. of Turkey-red oil and the requisite amounts of dyestuffs; further additions of 2—5 per cent. of sodium phosphate, for pale colours, 3—10 per cent. of sodium sulphate (Glauber's salt), for medium and dark colours other than blacks, and of 15—20 per cent. of the last-named salt, for blacks, being made as the dyeing proceeds. The colours obtained may be modified by dyeing in the cold with basic dyestuffs from baths acidified with acetic acid.

Basic dyestuffs are applied in the usual manner, about two-thirds of the amounts of tannic acid and antimony salt commonly employed, sufficing for mordanting.

Chlorine enhances the brilliancy of the fibre. Therefore, the treatment (under tension) with concentrated caustic soda solution, of yarns which are to be left white or are to be dyed after bleaching, should be effected before bleaching.

Strong twisting and pressure also, increase the lustre of the fibre, and a scroopy touch may be imparted to it by precipitating upon it a fatty acid or lime soap (this Journal, 1897, 437).—E. B.

## PATENTS.

*Fabrics in Imitation of Fabrics Woven in Colours, Improved Process of Producing.* P. Dosné, Aglié, Italy. Eng. Pat. 9804, April 17, 1897.

This process consists in preparing the warp, either during sizing or as a separate operation, with one of the constituents of an insoluble colouring matter—*e.g.*, β-naphthol—and, after weaving, developing the colour by a passage through the other constituent of the colour—*e.g.*, diazotised paranitraniline. The weft may then be dyed to any desired shade with substantive colouring matters.

Various modifications of the above process are described, one of which is to prepare the warp during sizing with a substance, such as acetate of soda, capable of resisting a colour—*e.g.*, Aniline Black—to be afterwards printed on the woven fabric.—R. B. B.

*Ink Stains, An Improved Composition for Use in Removing, from Substances.* J. N. Lyle, Liverpool. Eng. Pat. 12,281, May 18, 1897.

A solution of 4 oz. of citric acid in 1 pint of water, and the cleared liquor obtained after mixing 1 lb. of bleaching powder with 1 gallon of water, are applied successively and in the order named for the removal of ink stains. Claim is for a composition for, &c., consisting of a solution of citric acid and a solution of chlorine.—E. S.

*Bleaching, Dyeing, Washing, or Treating Fabrics and other Materials with Liquid; Improved Method of, and Apparatus therefor.* F. Erban, Hoechst-a/Main and L. Pick, Nachod, Prussia. Eng. Pat. 1634, Jan. 20, 1898.

In this apparatus the material is contained in a circular vessel, and the liquid is supplied from above through a perforated cover divided into sections. The end of the supply pipe is caused to rotate above this cover, so that the liquid passes in turn through each section. The vessel is provided with a false bottom, and the space below this is also divided into several compartments, and each of these is connected with the pump or injector which causes circulation of the liquid. This connection is formed by means of a perforated cock-plug, and by rotation of this cock-plug, the several compartments are successively placed in communication with the injector. By rotating the supply pipe and the cock-plug at varying speeds, the direction of the liquid through the material is continually changed, and it is claimed that a thorough distribution of the liquid thus takes place, and consequently the material is equally treated in every part.—R. B. B.

*Bleaching Liquids, Apparatus for the Electrolytic Treatment of.* [*Sediment Chambers.*] M. Haas, Aue, Germany. Eng. Pat. 1697, Jan. 21, 1898.

*See under* XI. A., *page* 466.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Ammonia-Soda Waste Liquors.* H. Schreib. Zeits. angew. Chem. 1898, [12], 274.

A criticism of a paper by Jurisch (this Journal, 1898, 349), in which the author states that certain values given for the waste liquors from the La Madeleine and a certain Austrian works, are not concordant. He draws special attention to the values given for the specific gravity, and compares these figures with the salt content of the solution, and further points out that the amount of calcium chloride said to be running away is only about $\frac{1}{3}$ or $\frac{1}{2}$ of that theoretically necessary in the cases of the two works given above.—J. T. C.

*Ammonia-Soda Process: Influence of the Addition of Salt while Carbonating.* K. W. Jurisch. Zeits. f. angew. Chem. 1897, [22], 714—717.

A continuation of the author's former paper (this Journal, 1898, 46).

*Influence on the Salt Consumption.*—The following are some figures from practice, showing the amount of salt used per 100 kilos. of finished soda :—

| — | Solvay. 1874. | Solvay. 1881. | Austrian. 1885. | Austrian. 1887. | Average with Carbonators 20 m. high. with—without Salt Addition. | |
|---|---|---|---|---|---|---|
| Salt in brine | 194 | 150 | 200 | 200 | 210 | 180 |
| Salt added. | .. | 60 | 30 | .. | | |
| Total.... | 194 | 210 | 230 | 200 | | |

Taking the average figures just quoted, we see that in the one case 52, in the other 60·7 per cent. of the salt consumed is utilised (for 100 kilos. of soda need, theoretically, 110·4 kilos. of salt). The rest is lost in the waste liquors. Nowadays, however, these waste liquors are worked up, often to give precipitated calcium sulphate, calcium carbonate, and pure calcium chloride solution. At Syracuse, New York, this is done; and the small salt consumption shown in the following figures seems to indicate that the salt in the waste liquors is partially recovered by evaporation (which with some of the modern evaporators can be done very cheaply). The process is carried on without salt addition, and with very high towers. Taking the average salt consumption at 131 kilos., we see that 83·3 per cent. is utilised.

| — | 1892. | 1893. | 1894. | 1895. | 1896. |
|---|---|---|---|---|---|
| Total output of soda in tons (1 000 kilos.). | 116,600 | 118,560 | 147,900 | 170,700 | 161,570 |
| Consumed, per 100 kilos. finished soda, of— | | | | | |
| Salt, as brine ...... | 129·0 | 135·0 | 133·0 | 126·0 | 131·0 |
| Limestone......... | 130·0 | 168·0 | 162·0 | 158·0 | 161·0 |
| Coal.............. | 119·0 | 137·0 | 115·0 | 117·0 | 116·0 |
| Coke ............. | 16·1 | 14·5 | 13·5 | 14·7 | 15·2 |
| Ammonia as sulphate | 1·43 | 1·35 | 1·21 | 1·06 | 1·46 |

*Influence on the Utilisation of Carbon Dioxide.*—The carbon dioxide comes from the lime-kilns and the calciners. The gases from the kilns in an Austrian works (1885—1887) contained 26—30 per cent. of $CO_2$, those from the calciners 35—40 per cent. As the kilns work at a steady rate, the mixed gases will be richer or poorer in $CO_2$ as more or less bicarbonate is calcined per 24 hours; hence the average content of $CO_2$ may be taken as 32 per cent. with salt addition, 30 per cent. without. This is in favour of salt addition; in fact, in 1885, working with addition of salt, 89·8 per cent. of the carbon dioxide was utilised, whilst in the same works in 1886 without salt addition, only 66·3 per cent. was utilised. Two English works, adding salt, utilised 92 to 97 per cent. of their $CO_2$. Similarly good results, however, seem now to be attained without salt addition when the towers are as much as 20 m. high. The low figures for limestone in the table given above, seem to point to this.

*Influence on the Limestone Consumption.*—The kilns work at a steady rate, so that the amount of limestone per 24 hours is independent of the working of the towers. The amount per 100 kilos. of soda consequently varies in the inverse direction to the amount of bicarbonate produced per 24 hours, and is hence less with than without salt addition (a saving, however, which is effected at the cost of additional salt). The modern heightening of the towers, however, has effected a much more notable economy in limestone: against the average of 156 kilos., in the above table, we have 180 kilos. as the average of five Solvay works from 1881 to 1887.

*Influence on the Lime Consumption.*—Inasmuch as the content of ammonium chloride in carbonated liquors is the same whether salt is added or not, the same amount of lime is used for a given quantity in either case; but as with salt addition more bicarbonate is precipitated and less remains in the mother-liquors, the amount of lime per 100 kilos. of soda is less when salt is added than when it is not (though it is to be remembered that the bicarbonate in the mother-liquors does the same work as lime in the distillation). These differences are negligeable, however, in comparison with those that may arise from better or worse supervision of the distillation columns.

*Influence on the Distillation.*—As the liquors contain, when not adding salt, more bicarbonate, the amount of decomposition taking place during the preliminary heating is then greater than with salt addition, whilst, of course, there is a correspondingly smaller amount of work thrown on the main column where the lime is added.

*Influence on the Coal Consumption.*—Exactly the same remarks apply here as in the case of the consumption of limestone, above.

*Influence on the Output of Soda.*—From 1876 to 1886 the output per 24 hours of an "element" of five 18-m. towers was taken as 20,000 kilos.; and the figures in successive years of the same works show that the difference between the two methods of working is quite negligeable in comparison with the yearly increase which practice and experience in the conduct of the work bring about. In 1886 the placing of the feed-tube for the brine higher in the tower at once brought up the capacity of an element to 30,000 kilos. per 24 hours, while an element of the larger and higher towers in use since 1887 has a capacity of 40,000 kilos.

The main reason why the addition of salt has been practically discontinued lies in the attempt to manufacture (and in the demand for, created by this attempt) a purer soda. The following analysis of "best soda" from an English work in 1880 (I.) and of "good soda" from an Austrian work in 1887 (II.) shows this difference:—

| — | I. | II. |
|---|---|---|
| $Na_2CO_3$ | 98·5 | 99·0 |
| NaCl | 0·61 | 0·35 |
| Insoluble | 0·546 | 0·05 |
| (Containing $Fe_2O_3$) | 0·007 | 0·005 to 0·01 |
| | 99·656 | 99·40 |

For special purposes (where, for example, most of the product is converted into caustic soda) it may still be profitable to work with addition of salt.—J. T. D.

*Zinc Hydrate and Cadmium Hydrate, Action of, on Ammonium Sulphate.* J. Tröger and E. Ewers. Arch. der Pharm. **235**, 644—646; Chem. Centr. 1898, **1**, [11], 658.

The author has succeeded in preparing the double sulphates of ammonium with zinc and cadmium, the formation of the latter of which is regarded as the cause of the non-precipitation of cadmium hydrate from an acid solution by means of ammonia.

*Zinc ammonium sulphate*, $Zn(NH_4)_2(SO_4)_2 + 6H_2O$, is prepared by introducing well-washed moist zinc hydrate into an aqueous solution of ammonium sulphate, until no more dissolves. Ammonia is evolved, and after filtering the solution, and evaporating the filtrate to incipient crystallisation, the double salt gradually separates out.

*Cadmium ammonium sulphate*, $Cd(NH_4)_2(SO_4)_2 + 6H_2O$, is prepared in a similar manner, except that the ammonia evolved must be expelled, else little cadmium hydrate will dissolve. The crystallisation of the double salt is expedited by the addition of some sulphuric acid.—A. S.

*Sodium Hyposulphite (Hydrosulphite), Preparation of, by Electrolysis.* A. Dubosc. Rev. Gén. des Mat. Col. 1898, **2**, 110—112.

Villon's apparatus is well adapted for the generation by electrolysis of sodium hyposulphite (hydrosulphite). It consists of a series of cells, composed half of zinc and half of cast iron, with porous asbestos-ware partitions, the cell-compartments being severally connected together by pipes. A solution of sodium bisulphite is passed successively through each of the negative compartments, and one of ferrous chloride through the positive compartments, in a direction opposite to that in which the current is flowing. The sodium hyposulphite which is formed is received under a layer of oil, to preserve it from contact with air.

Renard's voltameter may also be used, as also a simple apparatus consisting of a wooden tank of about 600 litres capacity, divided into two by a partition of unglazed porcelain or asbestos cardboard. 300 litres of sodium bisulphite solution at 35 B. are put into one compartment and dilute sulphuric acid (1 : 10) into the other. A cathode of zinc or carbon is fixed in the former, and a carbon anode in the latter. A lid serves to cover the tank for the purpose of excluding air, and the current is passed through the solutions until the sodium bisulphite has been completely transformed into sodium hyposulphite, about 350 h.p.-hours of electricity being required for the quantity mentioned, at a cost of about two francs per 100 kilos.—E. B.

*Pyruvic Acid, Two Colour Reactions for.* L. Simon. Bull. Soc. Chim. 1898, **19**, [7], 294.

*See under* XXIII., *page* 493.

*Ammonia Soda; Analysis of Waste Liquors from Distillation.* K. W. Jurisch. Zeits. angew. Chem. 1898, [12], 273.

*See under* XXIII., *page* 492.

*Ammonium Nitrate and Ammonia, Balanced Ratio between.* B. Kuriloff. Zeits. Physik. Chem. **25**, 107; Chem. Centr. 1898, **1**, [11], 655.

*See under* XXIV., *page* 501.

*Alkali Carbonates in Presence of Excess of Bicarbonates or Borax, Qualitative Examination for Traces of.* A. Leys. Bull. de l'Assoc. des Chim. 1898, **15**, [7], 723.

*See under* XXIII., *page* 490.

*Chlorates, Bromates, Iodates, and Hypochlorites; A New Method for the Electrolytic Production of.* W. Vaubel. Chem. Zeit. **22**, [34], 331.

*See under* XI. A., *page* 464.

## PATENTS.

*Ammonium Nitrate and other Salts, Impts. in the Manufacture of.* W. Garroway, Glasgow. Eng. Pat. 7066, March 18, 1897.

Sodium nitrate or potassium nitrate, mixed with silica in the form of sand or ground flints, is passed through a valved hopper into a muffle furnace, heated externally, to which superheated steam is admitted. The mixture is carried forward by arms and discs attached to a revolving shaft for delivery at the opposite end of the furnace, so as to make the process continuous. The nitric acid vapours evolved are taken, first to a condenser, in which a portion of the acid collects, and then through a series of towers, fitted with baffle plates, to the first of which ammonia gas is admitted. The residual vapours are passed through a tower, where any excess of ammonia is neutralised by a sulphuric acid spray. The ammonium nitrate solution formed in the condensing towers is drawn off, and concentrated in enamelled cast-iron pans, to obtain the solid salt.

The alkali silicate formed may be either utilised as such, or it may be treated in solution with lime to obtain caustic soda or potash, and a residue of calcium silicate, available in the manufacture of cement, &c. Reference is made to the inventor's Eng. Pat. 2489, 1896 (this Journal, 1897, 239).—E. S.

*Nitrites of the Alkali or Alkaline Earth Metals, Improved Process for the Production of.* J. McGougan and A. McGougan, Glasgow. Eng. Pat. 7715, March 25, 1897.

A mixture of sodium nitrate and caustic soda is melted in a suitable vessel, and finely powdered galena is added by small portions. The main reaction is represented by the equation—

$$PbS + 4NaNO_3 + 2NaOH = PbO + 4NaNO_2 + Na_2SO_4 + H_2O,$$

but some sodium plumbite is also liable to be formed. The sodium nitrite and sulphate may be dissolved from the litharge by water, and separated by crystallisation. If a plumbite be formed, this may be decomposed by sulphuric acid to obtain lead hydroxide and sodium sulphate. Or the plumbite may be decomposed in the original melt by further additions of sodium nitrate and of galena, which react on the plumbite to produce litharge and sodium nitrite and sulphate. Sodium plumbate may be obtained by adjusting the proportions of the materials taken, according to the following equation :—

$$PbS + 5NaNO_3 + 4NaOH = Na_2PbO_3 + Na_2SO_4 + 5NaNO_3 + 2H_2O.$$

The sodium plumbate is obtained as a yellow precipitate on treating the mass with water, and lead peroxide may be produced by boiling with water or treatment with an acid. The other salts may be separated, as before, by crystallisation. Red lead may be obtained from the peroxide by any ordinary method. Or, red lead may be obtained as a by-product in the melt reaction "by taking such quantities of sodium nitrate, caustic soda, and lead sulphide as will represent the quantities necessary for the production of lead oxide and lead peroxide in the proportion of two equivalents of lead oxide to one equivalent of lead peroxide."—E. S.

*A Salt or Compound containing Bromine, A new Manufacture of.* B. C. Hinman, New York, U.S.A. Eng. Pat. 8671, April 5, 1897.

A salt chiefly intended for the extraction of gold from its ores is prepared by adding bromine, saturated with chlorine, to a solution of an alkaline hydrate or carbonate, whereby an alkali bromate and chloride are formed, according to the equation—

$$Br_2 + 5Cl_2 + 12NaOH = 10NaCl + 2NaBrO_3 + 6H_2O.$$

By using a smaller proportion of chlorine, the compound may be modified so as to contain a bromide as well as a bromate. On adding hydrochloric acid, for instance, to such a compound, bromine is set free, and, on using it in the treatment of ores, "will, by virtue of its possessing an excess of available oxygen, decompose the bromides which may have formed during the treatment, so that after the leaching, all the bromine will be found in the free state. Consequently the bromine can be saved without any further chemical treatment."—E. S.

*Borax, Impts. in the Preparation or Manufacture of.* Ch. Masson, Gembloux, and C. Tillière, Brussels. Eng. Pat. 10,361, April 26, 1897.

An ammonium salt is treated with lime in a distilling column, and the ammonia set free, is passed into water contained in a digester, and the solution is then charged with carbonic acid. The digester is heated gently, and *pandermite* (the calcium borate of Asia Minor) is introduced. The digester is then closed and more strongly heated for several hours, whilst the contents are mechanically agitated, and are transferred, when the reaction is complete, to filter presses, whence the liquor passes to a "reaction chamber," where it is agitated with sodium chloride, and after addition of a little ammonia, is discharged into crystallisers. After removal of the borax that crystallises out, the mother-liquor is used instead of water in repeating the operation. After several operations, the mother-liquor requires a special treatment, which, as well as the treatment of the solid residues, is described.

In the preceding process, an ammonium biborate is first obtained by the ammonium carbonate treatment of calcium borate, and from this ammonium salt, borax is formed by the reaction with sodium chloride. But sulphurous acid may be passed into the ammonia used in the process, instead of carbonic acid, the succession of operations remaining the same. A modification of the process used in treating boronatrocalcites instead of calcium borate, is also described. —E. S.

*Red Oxide, Impts. in the Manufacture of, from the Waste Liquors from Galvanising Works, Tin Plate Works, Wet Copper Process, or other Residual Liquors containing Iron in the Form of Chloride.* T. T. Sill, Runcorn, Chester. Eng. Pat. 10,509, April 27, 1897.

The ferrous chloride liquor is kept in contact with an excess of scrap iron until the free acid is neutralised, and it is then precipitated by milk of lime, or sodium carbonate or the like, added in such proportion as to leave some iron in solution, the proportion so left being modified according to the desired tint of the ferric oxide which is to be produced. The ferrous hydrate precipitated, is collected and washed, and is heated with full exposure to air on suitable drying beds. The washing may, if desired, be effected after the drying. The oxide is then roasted until the required tint is obtained.—E. S.

*Salt from Brine, Impts. in Apparatus for obtaining, and for similar Purposes.* W. Shedlock, London. Eng. Pat. 11,240, May 5, 1897.

This invention consists of improvements in the apparatus described in Eng. Pat. 7796, 1896 (this Journal, 1897, 441), in which the salt, as it deposits from brine, is automatically and continuously elevated from the pan into moulds. The circular pan is heated by a steam coil and steam chambers, and revolving scrapers remove the crystals as they deposit into an external trough, whence they are lifted by an elevator, which deposits the salt in moulds or boxes brought successively under its outlet. These moulds are arranged on a circular frame or carrier over or on the pan, which frame revolves intermittently. "After each mould is filled, it will remain on the carrier during the filling of several other moulds, so that the liquid may drain from it into the pan. As each mould, after being filled, arrives at a certain point in the revolution of the carrier, it is to be lifted from the latter, and the block of salt formed in the mould can then be immediately removed, since the liquid will have been sufficiently drained from it to ensure its retaining its shape." —E. S.

*Hydrochloric Acid, Impts. in the Manufacture of, Free from Arsenic.* J. R. Wylde, J. W. Kynaston, and The United Alkali Company, Limited, Liverpool. Eng. Pat. 11,353, May 6, 1897.

The impure hydrochloric acid gas evolved in the manufacture of sodium sulphate, or when nitre-cake is heated with sodium chloride, is passed through cooling pipes, and then through a "dry tower," packed with coke or other suitable porous substance, in which some steam condenses, carrying with it, besides hydrochloric acid, sulphurous and sulphuric acid, and arsenic. To ensure that the gas leaving the dry tower is free from arsenic, its issuing temperature should not exceed 55° C. The purified acid gas is then condensed in the usual way.—E. S.

*Chromates or Bichromates, Impts. in the Manufacture or Regeneration of, from Waste Battery or other Liquors, or from Chrome Alum or Sesqui Salts of Chromium.* J. Y. Johnson, London. From H. Dereum, Philadelphia, U.S.A. Eng. Pat. 3801, Feb. 15, 1898.

Spent bichromate battery liquors, containing sulphuric acid, are neutralised, or nearly so, with milk of lime, and to the neutral or faintly acid heated solution, bleaching powder is added. The solution is then filtered from the precipitated calcium sulphate, and the regenerated potassium or sodium bichromate is separated by concentration and crystallisation. Chrome alum and the "green salts known as sesqui salts of chromium" are similarly treated, omitting the preliminary neutralisation with milk of lime.
—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

### PATENTS.

*Alabaster, Natural; Impts. in Treating, for enabling it to take a Fine Polish and to resist Atmospheric Influences.* H. F. Lamock, Aspelt, Luxemburg. Eng. Pat. 679, Jan. 10, 1898.

*See under* IX., *adjoining column.*

*Pottery, Bricks, and the like; An Improved Process for the Manufacture of a Compound for use in Making.* H. H. Lake. From V. Micheli, Florence, Italy. Eng. Pat. 830, Jan. 11, 1898.

Clay, marl, sandstone, kaolin, puzzolana, or similar substances, are baked, powdered, kneaded into a paste with suitable cements or adhesive materials, and moulded. In this way, pottery ware can be made in larger sizes than is possible when the articles have to be baked after moulding.
—H. H. B. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Portland Cement Makers' Association at Berlin, General Meeting of the German.* Feb. 23 and 21, 1898. Chem. Zeit. 22, [20], 184.

A paper was read by Schiffner on the influence of carbonic acid upon Portland cement and trass mortar, in which the opinion was expressed that carbonic acid in running water would in time cause the destruction of all calcareous binding materials by the production of the soluble calcium bicarbonate, but that the action is hindered by the gelatinous silica and alumina becoming covered with a protecting coat of vegetable organisms.

Attention was called by Schuliatschenko to the discovery by a Russian engineer of a method of determining free lime in Portland cement by the aid of a mixture of methylic iodide and benzene. From the experiments cited, it appears that the quantity of free calcium hydrate in cement mortar increases rapidly during the first month up to 30 per cent., but that after that time its formation proceeds only very slowly. A mass of concrete, which had been immersed in sea-water for 31 years, showed at a distance of 3 mm. from the surface, 20 per cent. of carbonic acid, but no free lime; at 6 mm. from the surface, 6 per cent. of carbonic acid; and in the middle of the block, 33 per cent. of free calcium hydrate.—H. H. B. S.

### PATENTS.

*Kiln to Economise Waste Heat by Means of "Assisted Draught," An Improved "Continuous."* A. Tracy, Retford, Notts. Eng. Pat. 6928, March 17, 1897.

The kiln comprises a number of chambers, either arranged as one, with partitions, or as a number of partly detached kilns, each connected with the next by flues. The draught is controlled by one or more fans, which are used for the purpose of forcing air through a kiln or chamber that is full of hot bricks, into the next, to utilise the heat from the bricks for "steaming." The same system of pipes or flues is also used to force the fires, the gases being conveyed through flues to drying sheds. To prevent waste of heat from the chimney or flues, air is forced by the fan or fans through spaces surrounding the flues, and is utilised to force the draught through the fires, &c.—R. A.

*Cement, Impts. in, and Apparatus for the Manufacture of.* T. Holden and C. Major, both of Bridgwater. Eng. Pat. 18,779, Aug. 13, 1897.

By this invention the drying of the "slurry" or mixture of cement-making materials before burning, is obviated. The damp materials are placed in layers alternately with layers of fuel in a kiln provided with one or more passages to allow of the escape of moisture. The passages are formed of perforated tubes or hollow pieces built up upon each other.—H. H. B. S.

*Cement, Impts. in the Manufacture of.* T. Holden and C. Major, both of Bridgwater. Eng. Pat. 20,670, Sept. 8, 1897.

The cement-making materials are burnt in the form of dry powder in layers alternating with layers of fuel, the latter being connected by columns of fuel passing through the layers of cement-making materials.—H. H. B. S.

*Artificial Stone, Impts. in the Production of.* L. Brockmann, Berlin, Germany. Eng. Pat. 24,609, Oct. 23, 1897.

Artificial stone having a smooth surface is produced by working up the ingredients with a solution of magnesium chloride, alum, and water-glass. The following are suitable proportions:—500 parts of gravel, 150 parts of magnesite, 3 parts of barytes, and 5 parts of colouring matter, the whole to be worked up with a solution of 65 parts of magnesium chloride, 7 parts of alum, and 3 parts of water-glass.
—H. H. B. S.

*Alabaster, Natural; Impts. in Treating, for enabling it to take a Fine Polish and to resist Atmospheric Influences.* H. F. Damock, Aspelt, Luxemburg. Eng. Pat. 679, Jan. 10, 1898.

The object of the patent is to enable alabaster to take a good polish, and to render it non-absorbent of water, and therefore more resistant on exposure to low temperatures. The pieces of alabaster as they come from the saw, or the finished articles, are dried in an oven, which is heated to 80° C. by a steam coil at the bottom, and provided with ventilators to facilitate the drying of the pieces. They are then placed on cradles or open trays, and at once plunged into a cold solution of fluosilicate of magnesium at 40° B. They are rapidly removed and kept in the air until the liquid clinging to the surface is absorbed, and are then again immersed and removed, alternately, until the alabaster remains permanently moist. The operation may be repeated once, or more often if desired, the saturated pieces being placed at once in the drying oven. Alabaster that has become air-dried should be thoroughly moistened before placing in the oven in the first place.—W. G. M.

*Substitute for Wood or Stone, An Improved Process for the Manufacture of a.* H. H. Lake. From V. Micheli, Florence, Italy. Eng. Pat. 822, Jan. 11, 1898.

Powdered carbonaceous materials, such as coal, lignite, peat, coke, charcoal, &c., either with or without the addition of terra-cotta, puzzolana, ashes, fossil farina, and the like, or of vegetable fibre or metals, are mixed into a paste with any mastic substance or cement and pressed into moulds.

The name "carbolignite" is given to the material, which is intended to be used as a substitute for wood, stone, cardboard, papier-maché, &c.—H. H. B. S.

*Building Stones and like Articles, Process for the Production of.* P. Kleber, Malstatt-Burbach, Germany. Eng. Pat. 1319, Jan. 17, 1898.

A mixture of pulverised quick-lime and sand, or the like, is tempered with water, shaped as required, and exposed to a mixture of superheated steam and hydrochloric acid vapour; or it is tempered with dilute hydrochloric acid, shaped as required, and exposed to superheated steam only. A few hours' exposure renders the stones sufficiently hard for building purposes.—H. H. B. S.

*Cements, Impts. in the Process of Manufacturing.* P. M. Justice. From The Lithosite Manufacturing Co., Saint Louis, Missouri, U.S.A. Eng. Pat. 1378, Jan. 18, 1898.

The mother-liquor from sea-water, being a solution of magnesium chloride with a little magnesium sulphate, is evaporated to a density of about 36° B. A certain proportion of magnesium oxide is then added and heat again applied. The resulting product, consisting of soft porous lumps, is mixed with calcined magnesite and ground to a fine powder. A solution of commercial magnesium chloride may be substituted for the mother-liquor, but in this case sufficient oxalic acid or ammonium oxalate must be used to convert the calcium chloride, which is present, into oxalate. It is preferable to add a little burnt clay to assist the drying of the magnesium chloride and prevent its crystallisation. Boric acid may also be used in the production of the cement, by adding it to the mother-liquor of sea-water, and grinding the resulting double salt with calcined magnesite. For the production of a cement that can be used for mortar, magnesium sulphate and aluminium sulphate, in equal proportions, are reduced to powder and mixed with calcined magnesite.—H. H. B. S.

*Wood, Preserving, Colouring, and Hardening; An Improved Process for.* F. Grumbacher, Berlin. Eng. Pat. 3900, Feb. 16, 1898.

The wood is treated *in vacuo* at 40°—60° C., until the free (and most of the hygroscopic) moisture is removed, and is then heated to 130°—170° C., so that the intercellular material is converted into preservative substances. By raising the temperature to 200° C., the colour of the wood is intensified, and an artificial ageing is effected. By cooling under pressure—down to a temperature below 0° C., if desired—the wood becomes permanently hardened. The low temperature is obtained by means of cooling coils. Before undergoing this treatment, the wood may be saturated with one of the well-known materials for rendering it incombustible.—W. G. M.

*Cement and Building Blocks, Impts. in the Manufacture of.* F. J. Bergmann, Neuheim-on-the-Ruhr, Germany. Eng. Pat. 4594, Feb. 24, 1898.

This invention consists in the production of cement clinker from ungranulated blast-furnace slag and lime ground together, with or without the addition of a mixture of coke dust and slag rich in silicates, such as cupola slag, copper slag, &c. For use in the manufacture of building blocks, &c., the ground ungranulated slag and lime are allowed to harden in the air without burning.—H. H. B. S.

## X.—METALLURGY.

*Gold, Recovery of, from Cyanide Solutions; Some Notes on the.* S. Cowper-Coles. Trans. Inst. Mining and Metallurgy, April 20, 1898.

The cathode, in the electrolytic method, should be of such a character that the gold is adherent during the process of deposition, but is capable of being readily stripped subsequently, whilst it should be electro-positive to the gold in the solution, so that it becomes coated with the metal on immersion. Aluminium fulfils all these conditions, as it is capable of receiving the deposit of gold in the form of a continuous sheet, which is non-adherent, by reason of the film of oxide which forms instantaneously on any exposed surface of aluminium. Hence the gold may be stripped off at intervals in the form of sheet or foil, or it may be rubbed off continuously by attaching the cathode to a revolving drum. In this way the gold is recovered at once, and without admixture with zinc or lead, which would have to be separated subsequently. Gold has thus been extracted from solutions containing 0·01 per cent. of KCy and $2\frac{1}{2}$ dwt. of gold per ton, preferably at 100° F., to the extent of 95 per cent. of the total amount present in 10 hours, the rate of flow of the solution being 15 galls. per 100 hours for every cubic foot of electrolysing cell or 3 sq. ft. of cathode surface. The current density should be higher when the aluminium plate is introduced than it is afterwards, in order to guard against the tendency to form aluminium hydroxide. Gold has been deposited from solutions containing only 0·0075 per cent. of KCy with a current density of 0·03 ampère per sq. ft., and an E.M.F. at the poles of the generator of 6 volts.

Three experiments are described. In the first a 0·5 per cent. solution of KCy containing 2 dwt. of Au per gallon, was electrolysed. Iron anodes were used, placed $1\frac{1}{4}$ in. from the cathode, and the cathodes were renewed at successive intervals of 15, 15, 30, 35, 60, and 60 minutes, the current density increasing in the first period from 1·5 to 3·1, and then decreasing to 2 ampères per sq. ft., while the voltage increased from 1·4 to 4·4, and then gradually to 4·9 volts at the terminals of the electrolytic cell. In this way 2·71 grms., or 84·7 per cent., of the gold was recovered in 3 hrs. 35 mins. In the second experiment a $\frac{1}{4}$ per cent. solution of KCy containing 3·8 dwt. of Au per gallon, yielded 50·6 per cent. of its gold in 1 hr. 50 mins., with a current density of from 3 to 2 ampères per sq. ft., and a P.D. of 4·75 to 5·1 volts; whilst a further quantity equal to 14·73 per cent. was recovered in 2 hrs. 20 mins., with a C.D. of 2·1 to 2 ampères per sq. ft., and a P.D. of 4·9 to 4·8 volts. In the third trial 32·2 per cent. of the gold in a $\frac{1}{2}$ per cent. KCy solution, containing 6 dwt. of Au per gallon, was recovered in 30 mins., with a C.D. of 6·25—5·2 ampères per sq. ft., and a P.D. of 4·3—4·1 volts; a further amount (14·68 per cent.) in the next 33 mins., with a C.D. 4·8—4·2 ampères, and a P.D. of 4·1—4·3 volts; and lastly, 22·46 per cent. of the gold (making 69·13 per cent. in 3 hrs. 33 mins.) in 1 hr. 50 mins., with a C.D. of 2·2—2·1 ampères, and P.D. of 2·3 volts. In an experiment with tailings containing 1 dwt. of Au per ton, a $\frac{1}{2}$ per cent. KCy solution was used, and this, with washings, was electrolysed, a pressure of 6 to 7 volts being required to drive a current of 0·3 ampère per sq. ft. through the solution. The gold formed a thin metallic detachable film.
—W. G. M.

*Gold, Absorption of, by Wooden Leaching Vats.* F. L. Bosqui. Eng. and Mining J. 1898, **65**, [9], 248.

The author made a number of experiments with regard to the absorption by wood of cyanide solutions containing gold, with a view of testing the accuracy of the supposition, that this is the cause of the discrepancy frequently noticed in new cyanide mills between the actual bullion returns and the yield indicated by assays. Two series of experiments were made, in order to ascertain whether the amount of gold absorbed by the leaching vats is greater in the case of (1) Continuous or (2) Intermittent contact with the solution.

*Continuous Contact.*—(A.) A piece of dressed American "redwood," thoroughly seasoned, weighing 2 lb. 2 oz., and exposing 180 sq. in. of surface, was immersed for three weeks in a solution containing on an average 5 dols. per ton of gold. The wood was then taken out, dried, reduced to charcoal in a clean solder stove, and further reduced to ash in a new assay muffle. The ash was carefully fluxed, cupelled, and the button weighed. The following results were obtained, silver being valued at 50 c. per oz.:—Actual absorption, 1·65 mgrms. of gold and 2·75 mgrms. of silver; absorption per ton of wood, 1·552 grms. of gold, value 1·03 dols., and 2·588 grms. of silver, value 0·04 dol. (B.) A piece of seasoned, dressed pine, weighing 3 lb. and exposing 180 sq. in. of surface, was submerged at the same time as the redwood, and subjected to the same conditions:—Actual absorption, 1·7 mgrms. of gold and 2·6 mgrms. of silver; absorption per ton of wood, 1·133 grms. of gold, value 0·75 dol., and 1·733 grms. of silver, value 0·02 dols. (C.) A piece of rough pine weighing 2 lb. 7 oz., and exposing 144 sq. in. of surface, was taken from the bottom of a discarded screen frame, which had been constantly and directly exposed to a strong solution (averaging about 5 dols. per ton in gold) for a period of 8 months, and again for a period of 4 months:—Actual absorption, 9·5 mgrms. of gold and 10·8 mgrms. of silver; absorption per ton of wood, 7·794 grms. of gold, value 5·18 dols., and 8·861 grms. of silver, value 0·11 dol.

*Intermittent Contact.*—(A.) A piece of dressed "redwood," weighing 2 lb. 7 oz., and exposing 180 sq. in. of surface, was subjected to intermittent submersion—immersed for 16 hours out of 24, then removed and dried in the sun for the other 8 hours—in a strong solution for a period of 3 weeks:—Actual absorption, 4·8 mgrms. of gold, and 2·1 mgrms. of silver; absorption per ton of wood, 3·657 grms. of gold, value 2·43 dols., and 1·6 grms. of silver, value 0·02 dol. (B.) Two pieces of rough pine, weighing together 3 lb. 4 oz., and each exposing 160 sq. in. of surface, were taken from near the top of a discarded screen-frame. This portion of the frame had been alternately exposed to the solution and dried for a period of 12 months, probably being under the solution about one-half of the time:—Actual absorption, 6·7 mgrms. of gold, and 13·7 mgrms. of silver; absorption per ton of wood, 4·122 grms. of gold, value 2·74 dols., and 8·429 grms. of silver, value 0·13 dol.

It is stated that with leaching vats thoroughly coated with asphaltum or paraffin paint, the losses by absorption would probably be quite insignificant. At the Standard Consolidated Mining Company's Works at Bodie, U.S., a considerable loss has been observed in the first clean-up of each new season, but the loss has always been made up during the remaining 7 or 8 months of operation. This loss is attributed to the residue of metal left behind in the zinc boxes, in the form of a firmly adherent film on the shavings; the latter averaging between 30 and 75 c. of gold per oz. The author considers that real losses may be due to (1) Imperfect sampling of vat charges and residues; (2) The presence of lumps of high-grade slimes among the coarser tailings, making it almost impossible to procure a fair average sample; (3) Losses from awkward methods of cleaning-up, or from careless management; and (4) Losses due to running to waste a high-grade effluent from the weak-solution boxes, or to discharging vats saturated with a weak solution containing a considerable gold value.

—A. S.

### *Slimes, Economic Treatment of.* C. Butters. J. Chem. and Metallurg. Soc. of Sth. Africa, **1**, 5—10.

In his presidential address the author deals with the difficulties attending the economic treatment of slimes, and with the methods and appliances which have been instrumental in making that treatment a practical success in S. Africa. Owing to the slimes retaining 40 to 50 per cent. of cyanide solution after treatment and settling, it is necessary that the solution be very dilute to prevent waste. To remove it from the slimes, suction has been employed with satisfactory results, but the wear and tear on the filter was excessive, and the cost of attending to and keeping the pumps in order was too great. Large, deep vats are now employed with a capacity of 1,000 tons, so that the slime, settling to a greater depth, becomes closer, and squeezes out some of the solution by its own weight. By this means the moisture is reduced to an average of 40 per cent., and sometimes to as low as 34 per cent. The efficiency of these vats has been further enhanced by pouring the pulp into a central tube of large diameter, and open at both ends, placed vertically with one end dipping half-way down into the solution in the vat, thus introducing fresh pulp without disturbing that already settled. The operation may be continuous, as the solution rising in the annular space between the central tube and the wall of the vat is clear and overflows as the fresh pulp is run into the centre. Attempts to concentrate the gold solution by leaching fresh slimes with it have shown the "double wash" to be the most practical. This consists of washing two fresh charges of slime with the one solution before precipitating the gold, and using the same solution after precipitation for the second washings.

The difficulty of dissolving old slimes as compared with new, owing to insufficiency of oxygen, is then dealt with. Agitation to absorb oxygen, which would be effectual with new slimes in six hours, fails to act with old material in 24 hours. Then again, it is found that a thick pulp will not rapidly absorb oxygen, nor even dissolve cyanide, so that a dilute pulp (4 tons of liquid to 1 ton of slime) is necessary for the quick and complete solution of the gold. The oxidation of the reducing substances by aeration is accomplished by agitation, by the injection of air into the bottom of the pulp, and, most successful of all, by the introduction of an air jet into the suction of the centrifugal pump used for pumping the slimes from one vat to another.

The only practical means of precipitating gold from these dilute solutions is electrolysis, and it is necessary that the solution be clear and that no deposits take place on the anode and cathode. The maintenance of clearness depends largely upon the anode used. The Siemens and Halske iron plate anode, with a current density of about 0·035 ampère per sq. ft., will last for years, but with an increased density, oxidation and corrosion rapidly take place. A future is anticipated for the peroxide of lead anode, which shows no corrosion on a small scale. The lime, used for coagulating the slimes, forms salts in the solution, which interfere in this direction, and it is a problem of the future to ascertain how to prevent their deposition in the electrolytic boxes, or how to satisfactorily reduce the amount present. In regard to a substitute for lime for settling purposes, the opinion was generally expressed, in the discussion which followed this paper, that none would be found. Many substances have been successfully used for the cathode.

Finally, the author anticipates that in any future treatment of slimes, the main features of the present South African treatment will be retained, *viz.*, the use of lime for settlement, the use of "spitzkasten" (settlers for deposit collection) for collecting-tanks for natural settlement, dissolving the gold by means of agitators or centrifugal pumps, the decantation of the liquid, and precipitation by an electrolytic method; and, furthermore, that vacuum presses, filter presses, or pressure filters will never be successfully employed.—A. W.

### *Black-Etard Permanganate Process, The.* [*Gold Extraction.*] Eng. and Mining J. 1898, **65**, [12], 345.

The original process of Etard consisted in subjecting the roasted ore to the action of a dilute solution of permanganate of potash in the presence of hydrochloric acid. The improvement claimed by Black, and patented by him in all the Australasian colonies, consists in the use of sulphuric acid and salt instead of hydrochloric acid, on account of the cheapness of the former; and it is stated that the modified process is cheap, simple, gives a high extraction, and does not give rise to the formation of obnoxious fumes. The precipitation is effected by ferrous sulphate.

It is stated that at the Mount Morgan mine, Queensland, ore containing so much copper as to render the application of cyanide impossible, was treated by the above process, and an extraction of 92 per cent. of the gold content obtained after 15 hours' percolation and in 5 hours by agitation. Concentrates from Barewood, Otago, containing antimony and manganese, as well as iron and arsenical pyrites, gave an extraction of 95 per cent.—A. S.

### *Metallic Sodium from Sodium Peroxide.* H. Bamberger. Ber. **31**, 151.

When sodium peroxide is mixed with carbon—wood charcoal, coke, or graphite—and heated in a covered crucible to 300°—400° C., the mass becomes incandescent and sodium distils off, condensing on the upper sides and cover of the crucible: $3Na_2O_2 + 2C = 2Na_2CO_3 + Na_2$.

By substituting carbides of the alkaline earths for the carbon, a similar reaction takes place: $7Na_2O_2 + 2CaC_2 = 2CaO + 4Na_2CO_3 + 3Na_2$.

If an excess of the carbide be employed, amorphous carbon, with or without carbide, is obtained in addition, according to the amount of carbide employed.—T. A. L.

*Lead-Plating Iron and other Metals.* E. v. Münstermann. Ackermann's Gew. Zeit.; Der Metallarbeiter, **24**, [3], 21.

The articles are cleaned by brushing with petroleum, rubbed dry and immersed, for some time, in a lead-lined box containing nitric acid and water, after which they are dried and transferred to a mixture of sulphuric acid, urine, and water, and there left until rust and dirt have disappeared. The third stage of preparation consists in immersion in a mixture of hydrochloric acid, water, and potassium-mercury iodide in order to facilitate the adhesion of the metal to the lead, into a bath of which (in the molten state) the articles are finally dipped. For wrought iron and other metals, the lead bath should have a temperature of at least 470° C., but greater heat is required for cast-iron articles.—C. S.

*Tinning Iron, Brass, &c.* Claudius. Der Metallarbeiter, **24**, [4], 28—29.

Polished brass or German silver can be tinned, without diminishing its lustre, by immersion in a boiling bath of 2 parts of alum, 2 parts of common salt, and 1 part of potassium bitartrate in 300 parts of water, the solution containing also a little tin, either from boiling it in a tin vessel or from the addition of stannous chloride. The articles rest directly on racks of metallic tin, supported by a perforated iron plate, which is contained in a bowl-shaped sieve of copper. The bath liquor dissolves a trace of the metal composing the objects under treatment, and replaces the same by a very thin coating of tin, which exhibits the initial lustre of the article; this reaction is assisted by the electrical current developed in the bath.

Another bath, for "contact" tinning, consists of a solution of 30 parts of potassium bitartrate and 3 parts of stannous chloride in 3,000 parts of water; and a third, of sodium pyrophosphate, 60 parts; crystallised stannous chloride, 6 parts; fused stannous chloride, 23 parts; and water, 3,000 parts.—C. S.

*Copper Bullion, Assaying of* Eng. and Mining J. 1898, **65**, [8], 223.

*See under* XXIII., *page* 492.

## PATENTS.

*Sulphide Ores containing Zinc or otherwise, Impts. in the Treatment and Reduction of.* H. R. Angel, London. Eng. Pat. 9409, April 13, 1897.

Each ton of partially roasted ore is mixed with $\frac{1}{3}$ ton of sulphate of soda and $\frac{1}{6}$ ton of coal, and zinc oxide about equal in weight to the amount of sulphur left in the ore. The finely crushed mixture is heated sharply in a V-shaped furnace with rabbling. Lead and precious metals are to be recovered in one operation. With moderate heating, the bulk of these metals is recovered at once, the residue by concentration, caustic soda then being a by-product. When raw ores are smelted, the slag is cleaned by re-smelting with galena. Lime (5 to 20 per cent.) may be used, with or without zinc oxide, when caustic soda is not required. The zinc from the ore is recovered as oxide together with that introduced at first; and when good zinc-collecting chambers are added to the furnace, all the metals present may be recovered in one operation. Calcined sodium sulphate may be substituted for the mixture of sulphate and coal. The claim is for "the use of sulphate of soda or the like, mixed with coals, &c.; also the use of calcined sulphate of soda; both to be used as a flux for the reduction of sulphide ores containing zinc, and for other ores."—W. G. M.

*Lead and Zinc, Impts. in or relating to the Smelting of Mixed Minerals, Ores, and the like containing.* G. H. Blenkinsop, Swansea. Eng. Pat. 8990, April 8, 1897.

The ore is smelted in blast furnaces, with or without flux and using a hot blast, taking care that the gases drawn off from the throat of the furnace are always at a temperature above that at which zinc vapour condenses. Lead alloys, matte, and slag remain in the furnace, while the zinc distils off and is to be collected from the waste gases in condensers. A typical charge is as follows:—1 ton of (Broken Hill) calcined sulphides, 8 cwt. of burnt ore (cupriferous pyrites with 3 per cent. of Cu, and a little gold and silver), and 10 cwt. of slag.—W. G. M.

*Cupels and Crucibles to be Employed in the Refining of Metals, Impts. in the Manufacture of.* J. L. Dobell, London. Eng. Pat. 10,715, April 30, 1897.

Calcined magnesite is crushed to a fine powder, moistened with a saturated solution of boric acid in water, or in spirit and water, or with boric acid and spirit alone, kneaded into a stiff paste, and shaped into the article required, which is then dried, and may subsequently be fired if desired. To increase the porosity of the finished pot, sawdust, bran, or flour may be added to the magnesite, in which case firing must afterwards be resorted to, in order to burn out the organic matter.—W. G. M.

*Iron and Nickel, Impts. in the Manufacture of Alloys of or Containing.* J. C. Mewburn, London. From La Soc. Anon. de Commentry-Fourchambault, Paris. Eng. Pat. 11,695, May 11, 1897.

The claim is for the production in crucibles or by any of the ordinary processes, of an alloy of iron and nickel, containing approximately 37 per cent. of nickel, which does not expand when heated.—J. H. C.

*Gold, Extracting, from its Ores and Compounds; An Improved Process of and Apparatus for.* H. R. Cassel and B. C. Hinman, New York. Eng. Pat. 7555, March 23, 1897.

The pulverised ore, if not already acid, is treated in a tank, having a perforated bottom, with a dilute acid, which is made to percolate through the ore. It is then similarly but more slowly treated with an alkaline bromide solution, which dissolves the gold.

The bromine is afterwards liberated from the solution by means of chlorine or other reagents, driven off from the solution by air under pressure, then re-absorbed and used over again. After the bromine has been removed, the gold is precipitated by any suitable method.—J. H. C.

*Gold or Silver, Improved Means applicable for Use in the Treatment of Ores or Compounds containing, and in Means applicable for Use in the Treatment of such Materials with the Aid of Solvents.* J. C. Montgomerie, Stair, Ayrshire, and H. Parkes, Dulwich. Eng. Pat. 9964, April 21, 1897.

The ores to be treated are finely crushed and mixed with water enough to form a thin mud. The water is placed in a barrel, to which is then added cyanide of potassium or sodium, or a mixture of the same, together with ammonia or other alkali or alkaline earth in the form of oxide or hydrate, or an alkaline or earthy nitroprusside, bromide, or fluoride, or barium peroxide, or peroxide of lead, or generally a substance capable of yielding oxygen, or an alkaline phosphide, carbide, or sulphide, or ammonium sulphate, or bromine or fluorine water. The cyanide, &c. having been dissolved, the pulverised ore is added and the barrel is charged with air under a pressure of 50 to 100 lb. per square inch, and is then rotated for several hours, after which it is emptied, and the contents are filtered and washed. When the ore is refractory, or when the gold or silver exist in combination with sulphides of copper, zinc, lead, or other base metal—*e.g.*, the "Broken Hill" ore—it is roasted dead in a reverberatory furnace. A mixture of various specified solid chlorides, sulphates, nitrates, oxides, &c. is then intimately mixed with the ore, and a dull red heat is maintained for an hour or more. After cooling, the mass is lixiviated, first with water to extract the gold, then with dilute sulphuric acid to extract the copper, and finally with "hyposulphite of soda" to extract the silver. Method are specified for the recovery of the copper, zinc, silver, &c., from the several solutions. To recover the gold, the solutions are passed through a charcoal filter before separating the base metals.—J. H. C.

*Gold, Impts. in Plates for Collecting or Separating.* C. E. Greene, Providence, Rhode Island, U.S.A. Eng. Pat. 976, Jan. 13, 1898.

The plates are composed essentially of zinc, quicksilver, and sodium. The quicksilver and sodium are first combined in the ordinary way, and then thoroughly mixed with the molten zinc. The whole is poured into a heated mould containing a piece of metal adapted to form a metallic strengthening or base plate. Collecting plates so formed are placed in the sluices or riffles used for collecting gold in placer mining and gold milling.—J. H. C.

[*Pyritic Smelting.*] *Impts. relating to the Smelting and Converting of Metal Ores, and to Apparatus therefor.* O. S. Garretson, Buffalo, U.S.A. Eng. Pat. 1432, Jan. 18, 1898.

In pyritic smelting the molten matte is Bessemerised continuously in the furnace in which it has been produced or in an adjacent furnace, the slag produced during the conversion being made to flow through the matte on its way to the slag outlet.—J. H. C.

# XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## (*A.*)—ELECTRO-CHEMISTRY.

*Aqueous Solutions, Electrolytic Decomposition of.* L. Glaser. Zeits. f. Elektrochem. **4**, (1898), 355—359, 373—379, 397—402, 424—428.

The author has made an extended series of experiments to obtain more precise information with regard to the electromotive force necessary for the electrolysis of aqueous solutions, and the results are described in three parts.

*I. The Formation of Water in the Gas Battery* (*H, O Cell*).—In these experiments platinised platinum electrodes were used, and the gases, oxygen and hydrogen, were led over them in a continuous stream. Different electrolytes, comprising acids, bases, and salts, were employed, and it was found that the mean value of the electro-motive force of the battery was 1·08 volts. Certain salts, however (with the exception of those—*e.g.*, aluminium sulphate, &c.—which react acid in solution), occasionally gave slightly lower values, as has already been observed by Smale (Zeits. f. physical Chem. 1895), but these low values disappeared on making the electrolyte slightly acid or alkaline. Also, certain solutions which, on electrolysis, do not evolve pure oxygen and hydrogen, gave in general lower results. The normal E.M.F. of the battery is only obtained after the lapse of four or five hours; and it was proved, by measuring the potential of each electrode against a normal electrode, that this sluggishness in attaining the normal value is due almost entirely to the oxygen electrode. This is further ascribed to the low absorptive power of platinum black for oxygen. A minute quantity of hydrogen peroxide added to the electrolyte causes the battery to assume its normal E.M.F. in a shorter time; larger quantities, on the other hand, produce a lowering of the E.M.F., due probably to secondary reactions. It is also noteworthy that the final value is in general reached much quicker with bases than with acids.

As regards the capacity of the cell, it was found, on taking a current of 1·8 × $10^{-4}$ ampères from an ordinary gas battery filled with a normal solution of caustic soda, that the voltage fell in 300 minutes from 1·080 V. to 0·914 V., where it remained fairly constant.

On breaking the circuit, the voltage gradually rose, and attained its original value after two hours' regeneration. When heavier currents were taken from the cell, a greater fall in voltage occurred, and each current strength corresponded to a lower limiting value of the E.M.F. For example the following values were obtained:—

Current taken, 2·3 × $10^{-4}$ ampères: voltage fell to 0·760 V. in 300 minutes.

Current taken, 4·2 × $10^{-4}$ ampères: voltage fell to 0·463 V. in 80 minutes.

From these results it follows that the gases can only diffuse very slowly into the electrodes, and that for each current strength a point of equilibrium is reached when just as much gas diffuses into the electrodes as is removed as water during the reaction which takes place. The E.M.F. of the gas battery diminishes with rise of temperature, and it was found from measurements with six different solutions that the mean value of the temperature coefficient was 0·00143, whereas Smale obtained the value 0·00146. In accordance with the osmotic theory, it was found that an increase in the concentration of the electrolyte produced a slight but perfectly definite increase in the electro-motive force of the gas battery. The substitution of organic liquids for the ordinary electrolytes generally gave low and very inconstant results. Thus far the above experiments confirm the law enunciated by Smale, *viz.*, that the formation of water in the gas battery is a reversible phenomenon.

*II.—The Decomposition of Water.*—Hitherto practically all observers agree in assigning about 1·67 volt as the lowest E.M.F. necessary for the electrolysis of acidified water, but between this value and the E.M.F. of the gas battery there exists the large difference of 0·59 volt. In order to throw some light on this apparent contradiction, the next series of experiments was undertaken.

The P.D. between platinum electrodes dipped into acidified water, was gradually increased from zero upwards, and the intensity of the current which passed through the cell at each stage, measured by means of a very sensitive galvanometer. It was found that a greater increase in the deflection of the galvanometer took place about 1·08 V., and it was also noticed that there were certain irregularities at about 0·5 V. —0·6 V. At 1·08 V., therefore, a continuous but perfectly distinct electrolysis goes on, which, on account of its relative insignificance has hitherto escaped the notice of observers. It is thus shown directly that the formation and decomposition of water is a reversible process. The further discussion of the curve obtained by plotting the strength of the current against the E.M.F. is to be found in Part III. In the first experiments two bright platinum electrodes, which did not differ very much in size, were used, and the interpretation of the curve obtained, was rendered more difficult owing to the summation of the effects of polarisation. To overcome this difficulty, first the anode and then the cathode was made non-polarisable by making it relatively very large. The electrodes actually used consisted of a sheet of platinised platinum foil with a surface of 20 sq. cm., which was always exposed to the air before being introduced into the cell, and a small piece of platinum wire sealed into a glass tube and having a superficial area of 4·7 sq. mm.

In order to make sure that the potential of the large electrode with respect to the solution remained constant throughout the experiments, a large working lead electrode coated with peroxide was used in some cases, and the values so found referred to the platinum electrode. The following table will serve to illustrate the results obtained when the small electrode was the cathode. The deflections of the galvanometer $n_1$, $n_2$, and $n_3$, are recorded immediately after making connection, then after the lapse of half a minute, and finally after one minute. Each millimetre deflection corresponds to a current of 1·14 × $10^{-9}$ ampères. Although the amplitude of the deflection varies with the time, analogous curves are described, and the point at which decomposition begins does not change.

It will be seen from the table that a small current, probably a convection current, begins to pass at 0·200 V. and remains of the same order of magnitude up to the point of decomposition, when there is a sudden and unmistakable increase in the intensity of the current, showing that the electrolysis has set in. The same irregularity is noticeable about 0·5—0·6 V., when the differential quotient $\frac{dn}{d}$ actually becomes negative. On repeating the experiment after 0·7 V. had been reached, this irregularity disappeared, and the curve remained practically linear and parallel to the abscissæ axis up to 0·6 V. The point at which electrolysis begins, *viz.*, 1·08 V., is nearly independent of the concentrations of the acid.

| Volt. | $n_1$ | $n_2$ | $n_3$ | $\frac{di_2}{de}$ |
|---|---|---|---|---|
| 0·050 | 0·00 | 0·00 | 0·00 | |
| 0·200 | 0·10 | 0·00 | 0·00 | 0·0 |
| 0·265 | 0·20 | 0·10 | 0·10 | 1·5 |
| 0·350 | 0·70 | 0·60 | 0·60 | 5·9 |
| 0·420 | 1·30 | 1·25 | 1·25 | 9·3 |
| 0·515 | 1·70 | 1·45 | 1·25 | 2·1 |
| 0·550 | 1·50 | 1·20 | 1·10 | – 7·1 |
| 0·655 | 1·70 | 1·40 | 1·35 | 1·9 |
| 0·750 | 2·00 | 1·70 | 1·55 | 3·2 |
| 0·800 | 2·15 | 1·82 | 1·65 | 2·4 |
| 0·848 | 2·30 | 1·95 | 1·75 | 2·7 |
| 0·900 | 2·40 | 2·02 | 1·95 | 1·4 |
| 0·982 | 2·50 | 2·10 | 2·00 | 1·0 |
| 1·020 | 2·90 | 2·50 | 2·30 | 1·1 |
| 1·100 | 27·00 | 17·00 | 13·20 | 181·0 |
| 1·170 | 56·40 | 44·00 | 38·50 | 385·0 |
| 1·285 | 92·80 | 81·80 | 77·20 | 330·0 |

In a similar way it was found that the same E.M.F. was necessary and sufficient for the electrolysis of solutions of the bases, although the point of decomposition is not nearly so well marked. The irregularity at 0·6 V., on the other hand, was much more pronounced. Whilst the evolution of hydrogen at the small electrode (cathode) can easily be seen at about 1·11 V. when acid solutions are electrolysed, this could only be detected at 1·38 V. in N-NaOH solution.

The author ascribes the reason for this to the fact that in acid solutions there are a large number of H-ions, whilst in solutions of bases there are scarcely any; and he further embodies this in the following fundamental rule, which has been of great assistance in the study of electrolysis.

*In order that the point of decomposition of an ion may be well marked, it is necessary that it should be present in the solution in relatively large quantity.*

Increase in concentration in solutions of the bases, requires a slightly increased E.M.F. to produce electrolysis, in accordance with what was found respecting the influence of concentration on the E.M.F. of the gas battery. In the case of 10N-NaOH the E.M.F. necessary was 1·095 V.

When the electrolyte consisted of salt solutions, curves exhibiting several breaks were obtained, so that one could speak of a regular series of points of decomposition. On electrolysis, $K_2SO_4$ furnishes acid at one pole and base at the other, so that the variable opposing E.M.F. which results, does not permit of such a clear insight into the phenomenon. If, however, the solutions are acidified or made alkaline, the same results are obtained as before.

Hitherto the small platinum point has always been employed as cathode, but an equally important set of results was obtained by making it the anode. When acid solutions were placed in the cell, the first indication of electrolysis was observed at about 0·6 V., but when bases were investigated, a sudden break occurred at exactly 0·590 V., which was even more sharply defined than the break at 1·08 V. when the experimental electrode was cathode. This explains the apparently abnormal behaviour already referred to. The break at 0·590 V. occurs both in the case of acids and of bases, and hence it must be a point at which a particular ion separates out; and, further, by applying the rule already laid down, it must be an anion. Now the only possible anions in (*a*) $H_2SO_4$ are $\overset{-}{HSO_4}$, $\overset{--}{SO_4}$, $\overset{-}{OH}$, and $\overset{--}{O}$, whilst in the case of (*b*) KOH they are $\overset{-}{OH}$ and $\overset{--}{O}$, provided we consider water as a weak dibasic acid which splits off its first H ion much more easily than the second, thus:—

$$H_2O = \overset{+}{H} + \overset{-}{OH}$$

$$\overset{-}{OH} = \overset{+}{H} + \overset{--}{O}$$

Up to the present, however, oxygen has always been supposed to take part in electro-chemical reactions as if it were hydroxyl. The anions common to the acid and alkaline solutions are the anions of water, and since the $\overset{-}{OH}$ ions are present in enormous quantity in the solution of alkali, we must conclude that the point of decomposition, 0·59 V., is the point at which the $\overset{-}{OH}$ ions give up their charge when they are at liberty to react on each other, thus: $4OH = 2H_2O + O_2$, producing water and oxygen.

If two similar platinum electrodes are used, the E.M.F. necessary to produce an appreciable electrolysis will thus be the sum of 1·08 + 0·59 = 1·67 V., *i.e.*, the value previously observed by Helmholtz, Le Blanc, and others.

On the other hand, a visible evolution of hydrogen at the cathode at 1·08 V. has been confirmed, and consequently a corresponding quantity of some other ion must have separated out at the anode. Since the conditions, as determined above, do not admit of the discharge of hydroxyl ions, the author concludes that doubly-charged O ions must have separated out. There are thus two points at which water is decomposed, *viz.*, at 1·08 V. and at 1·67 V. In each case hydrogen is discharged at the cathode, whilst $\overset{--}{O}$ is discharged at the anode at the lower value and $\overset{-}{OH}$ at the higher. The process of $H_2O$ formation in the gas battery must therefore be represented thus:—

$$2\overset{+}{H} + \overset{--}{O} = H_2O,$$

where the oxygen, by means of the platinum black, goes into the solution as doubly-charged O ions.

III. *The Decomposition of Dissolved Substances.*—In the third part of the investigation, the author examined the behaviour of solutions when the E.M.F. employed, exceeded 1·08 V. From a consideration of the theory, it follows that if only one anion or one cathion is present in solution, then only one point of decomposition can exist.

In water, for example, the only possible cathions which can be present are the H ions, and, since this also applies to dilute $H_2SO_4$, it follows that, with respect to the cathions, there can only be one point of decomposition, *viz.*, 1·08 V., and that if the E.M.F. is increased above this amount, the curve must remain linear and equally inclined to the abscissæ axis without exhibiting any other break. This was found to be the case on increasing the E.M.F. up to 2·29 V. The differential quotient $\frac{di}{de}$, representing the change in the intensity of the current with the alteration of the E.M.F., remained constant, above 1·08 V., within the limits of experimental error.

There are, however, several anions in sulphuric acid, so that, with respect to the oxygen polarisation, a different behaviour is to be expected. The curve begins to rise gently at 0·59 V., and at about 0·9 V. a sudden break occurs and it becomes very steep. At the same time a continuous evolution of minute bubbles of oxygen takes place at the platinum point. The anions $\overset{--}{O}$ and $\overset{-}{OH}$ have already been discussed, so that we have only to consider the anions $\overset{-}{HSO_4}$ and $\overset{--}{SO_4}$. One of these must have occasioned the break at about 0·9 V. Now, dilute (0·1N-$H_2SO_4$) $H_2SO_4$ contains relatively more $\overset{--}{SO_4}$ ions than $\overset{-}{HSO_4}$ ions, so that the break in all probability is due to the $\overset{--}{SO_4}$ ions. This assumption was confirmed by employing a solution of magnesium sulphate acidified with dilute $H_2SO_4$. Such a solution contains practically only $\overset{--}{SO_4}$ ions, and the break was found to take place at 0·87 V. The conditions necessary for the formation of persulphuric acid, $H_2S_2O_8$ (*viz.*, a concentrated solution of the acid and a high current density) also favour this view, but it is still uncertain whether the doubly-charged ion gives up only one of its charges, or whether it gives up both and then reacts with water, in the following way:—

$$2SO_4 + 2H_2O = 2H_2SO_4 + O_2.$$

If it be granted that 0·87 V. is the point at which $\overset{--}{SO_4}$ ions give up their charge in normal solution, there must still be another break at the point where the $\overset{-}{HSO_4}$ ions are discharged. By applying the rule already laid down, we must so arrange matters that a relatively large number of $\overset{-}{HSO_4}$ ions are present. This is best attained by saturating 8N-$H_2SO_4$ with $KHSO_4$, since, in this way, we obtain a solution which contains scarcely any $\overset{--}{O}$ ions, whilst the quantity of $\overset{-}{OH}$ and $\overset{--}{SO_4}$ ions is reduced to such an extent that it becomes relatively small when compared with the number of $\overset{-}{HSO_4}$ ions present. On electrolysis it was found that a copious evolution of oxygen only took place above 1·6 V.

Careful experiments showed that the exact point at which the break occurred was 1·57 V., so that this may be accepted as the point at which the $\overset{-}{HSO_4}$ ions give up their charges. Besides the ions already considered, there are no others possible, either in a more concentrated or in a more dilute solution, so that we can assign to each ion its own place. Thus, putting the E.M.F. necessary for the discharge of $\overset{--}{O}$ ions, equal to 1·08 V. ($\overset{+}{H} = 0$), we have the following points of decomposition :—

$$\overset{--}{O} = 1{\cdot}08\text{ V.}$$
$$\overset{-}{OH} = 1{\cdot}67\text{ ,,}$$
$$\overset{--}{SO_4} = 1{\cdot}95\text{ ,,}$$
$$\overset{-}{HSO_4} = 2{\cdot}65\text{ ,,}$$

all of which apply to a normal solution, excepting the last, which refers to a more concentrated solution (8 – normal).

Above 2·7 V., therefore, there is no further break in the continuity of the curve, *and we now have an explanation of the fact that the maximum polarisation of dilute $H_2SO_4$ is about 2·7 V.*

The author points out that similar results are to be expected on investigating all other acids and bases, and he also refers to the possibility of rapidly performing analysis by simply measuring the points of decomposition.

When the respective values for all the anions and cathions are determined, it will only be necessary to add together any particular pair, allowing for the concentration, in order to find the E.M.F. required for the decomposition of any solution, which is an important matter in technical electro-chemistry.

The following table shows the position of the break in the curve, on electrolysing solutions of different bases :—

| | | |
|---|---|---|
| KOH | 1·00 Normal | 1·40 volt. |
| NaOH | 1·00 ,, | 1·315 ,, |
| $Mg(OH)_2$ | 0·0002 ,, | 1·395 ,, |
| $Ca(OH)_2$ | 0·02 ,, | 1·33 ,, |
| $Ba(OH)_2$ | 0·22 ,, | 1·185 ,, |
| $Sr(OH)_2$ | 0·063 ,, | 1·26 ,, |

whilst the variation of the E.M.F. for different strengths of KOH was found to be—

| | | |
|---|---|---|
| KOH | 10 Normal | 1·32 volt. |
| ,, | 5 ,, | 1·38 ,, |
| ,, | 1 ,, | 1·40 ,, |
| ,, | ½ ,, | 1·45 ,, |
| ,, | [illegible] ,, | 1·47 ,, |

This variation is not as great as it ought to be according to the theory, and the author points out that it may be due to the fact that what is measured may not be the point at which the potassium ions give up their charge, or, at any rate, that this point may be influenced if a potassium hydride separates out, or if an alloy of platinum and potassium is formed, which immediately decomposes into hydrogen, caustic potash, and platinum.

In conclusion, it is shown that, although the electrolysis of water can take place in a primary fashion, yet, as a matter of fact, and especially if appreciable currents are employed, the decomposition is a secondary phenomenon.

—J. S.

*Chlorates, Bromates, Iodates, and Hypochlorites, A New Method for the Electrolytic Production of.* W. Vaubel. Chem. Zeit. **22**, [34], 331.

The addition of from 1 to 5 per cent. of alkali bicarbonate to the anode chamber of a chloride decomposing cell, in order to increase the yield of chlorate, is proposed in Ger. Pat. No. 83,536, granted to Schuckert and Co. The author, in the course of other investigations, has observed that when the anode chamber contains bicarbonate of an alkali, and the cathode chamber a saturated solution of the corresponding chloride, and a diaphragm separates the two, a complete conversion of the chlorine into chlorate occurs in the anode compartment of the cell. He gives the following equation to explain this change :—

$$NaCl + 6NaHCO_3 = NaClO_3 + 6CO_2 + 6Na + 3H_2O.$$

The carbon dioxide is liberated as gas at the anode, and may be used to form fresh bicarbonate with the hydrate produced at the cathode.

In the author's experiments, a temperature of between 60° and 70° C. was used ; the current density varied between 5 and 10 ampères per sq. dm. (45 and 90 ampères per sq. ft.), the yield of chlorate being greater at the higher current density. The E.M.F. was from 4 to 6 volts, and 5 grms. of sodium bicarbonate were added to each 100 c.c. of anode liquid at intervals of four hours. This liquid was found to contain 6·5 grms. of sodium chlorate and 7·5 grms. of sodium bicarbonate at the end of the experiment ; and continuance of the electrolysis beyond this point was not found to be advantageous. The cathode liquid contained 10 per cent. of sodium hydrate. The yield of the latter represented a current efficiency of 90 per cent. ; that of the chlorate one of about 50 per cent. The substitution of caustic alkalis for the bicarbonate salts, caused formation of chlorides in the anode compartment of the cell. As regards energy efficiency, 736 watt hours yielded about 50 grms. of sodium chlorate. Oettel, in his experiments upon this same subject, obtained 88·14 grms. of potassium chlorate from 736 watt hours ; but the E.M.F. used by him was only 3·3 volts, and he worked without any diaphragm with slightly alkaline chloride solutions. The Gall and Montlaur chlorate process is worked on a manufacturing scale. Diaphragms are used, and a solution of an alkali hydrate is supplied at intervals to the anode compartments of the cells. By this process 1,003 watt hours are required to yield 88·14 grms. of potassium chlorate as against 951 watt hours in the author's experiments. Though theoretically the author's method would appear to be the more profitable, experiments upon a large scale have shown that this is not actually the case.

Bromates and iodates of exceptional purity can be produced by the same method as that described for chlorates.

If a lower temperature than that named be employed, hypochlorites are formed without any chlorides ; but the solution so obtained is very unstable.

Experiments made by the author showed that the oxidising properties of the bicarbonate solution contained in the anode compartment of the cell could also be utilised in the production of Prussian Blue. Attempts to oxidise barium hydrate in the same manner failed ; but chromium hydrate was raised to the higher stage of oxidations. The yield in the latter case was, however, only 3 per cent. as against 47 per cent. by the Haussermann method.

The attempt to produce fluorates in a similar manner failed.

The experiments were carried out in the laboratory of Prof. Dieffenbach at Darmstadt.—J. B. C. K.

*Electric Spark, Chemical Action of, on Alcohols, Ethers, and Phenols, in Presence of Nitrogen.* Berthelot. Comptes Rend. 1898, **126**, 616—627.

The author has investigated the reactions which occur when various alcohols, &c., are submitted for a lengthened period to the influence of elecric sparks, in an atmosphere of nitrogen. The following are the conclusions :—

1. All the alcohols examined, fix nitrogen, yielding bodies of an amido or basic nature.

2. At the same time, hydrogen is evolved in the case of the $C_nH_{2n+2}O$ alcohols, methyl alcohol giving off 1 atom and ethyl and propyl alcohols, 2 atoms of hydrogen.

3. This loss of hydrogen is comparable with the decomposition suffered, under similar conditions (also in absence of N) by $CH_4$ and $C_2H_6$, which split off 2 atoms of carbon. Allyl alcohol, however, yields up comparatively little hydrogen, resembling in this respect, acetylene and allylene.

4. The phenols fix nitrogen in proportions varying greatly in individual cases.

5. The phenols evolve only traces of hydrogen—a circumstance noteworthy in respect to quinol, which so readily parts with 2H in many reactions.

6. Alcohols of the fatty series fix nitrogen at the rate of 1 atom for every 2H evolved, *i.e.* (see 2) 2 mols. of methyl, or 1 mol. of ethyl or propyl alcohol, fix 1 atom of nitrogen. As regards allyl alcohol, 3 mols. fix $N_2$.

7. Comparing the ratio of C to N in the compounds formed from the alcohols and corresponding hydrocarbons respectively, this ratio is the same for methane and methyl alcohol; whilst ethane (or ethylene) and propylene fix only half as much N as the respective alcohols. Allyl alcohol fixes more N than allylene, the ratios being in the proportion of 3:5.

8. In the case of isomeric bodies, the C:N ratio is the same for propyl and isopropyl alcohols, as also for propylene and trimethylene. The three dihydric phenols differ so widely in their behaviour that no conclusions can be drawn.

9. As regards structure, it is probable that these nitrogen compounds are aldehydic condensation products, possessing a cyclic constitution, resembling that of the pyridine and quinoline bases.—H. T. P.

*Sodium Hyposulphite (Hydrosulphite), Preparation of, by Electrolysis.* A. Dubosc. Rev. Gén. des Mat. Col. 1898, **2**, 110.

*See under* VII., *page* 456.

## PATENTS.

*Chromates or Bichromates, Impts. in the Manufacture or Preparation of, from Waste Battery or other Liquors, or from Chrome Alum or Sesqui Salts of Chromium.* J. Y. Johnson, London. From H. Dercum, Philadelphia, U.S.A. Eng. Pat. 3801, Feb. 15, 1898.

*See under* VII., *page* 458.

*Electrical Resistances, An Improved Manufacture of.* J. F. Bachmann, A. Vogt, J. Kirchner, A. König, C. C. Weiner, and A. Jörg, all of Vienna. Eng. Pat. 15,238, June 25, 1897.

A bad conductor is mixed, in a finely divided condition, with a good conductor, in suitable proportions, and the mixture is fashioned into the desired shape. Thus, clay, cement, or gypsum may be mixed with pulverulent graphite or metals, with or without water-glass, zinc chloride, gum, or sugar solution, and may then be moulded into shape, and either fired or treated with a hardening agent. Or the non-conductor is mixed with "asphalte," sugar, or the like, or with certain metallic compounds which, on heating, become converted into conductors. Or a stone compound is mixed with easily reducible metallic oxides, and the mixture is heated in a carbon envelope (or the carbon may be mixed in the mass), so that metal becomes reduced. The proportions of the ingredients are varied according to the degree of resistance required. Thus, 95 per cent. of graphite with 5 per cent. of kaolin forms a relatively good conductor, the conductivity of which may be further increased by soaking the fired mass in syrup and burning it again. The resistance may be increased by using a larger proportion of kaolin. By placing a layer of the resistance-mixture between two metallic surfaces connected with the opposite poles of an electric generator, a convenient electrical heating or cooking apparatus may be produced, and by varying the mixture (and therefore the conducting capacity) at different points, the temperature may be controlled locally.—W. G. M.

*Primary Batteries, Impts. in and relating to.* W. Rowbotham, Birmingham. Eng. Pat. 2378, Jan. 29, 1897.

Claims: I. "A primary battery having two compartments, one a closed compartment, made pressure-tight, which is open to the interior of porous tubes containing carbon rods, the compartment being so arranged that the pressure produced within it by the generation of gas [nitrous fumes], due to the action of the battery, forces acid [sulphuric and nitric], through the walls of the porous tubes into the water contained in the second compartment containing the anodes," &c. II. "In a primary battery of the kind specified in the first claim, the arrangement of the cells in steps, with water or acid flowing from one cell to the other in series through air gaps," &c.—J. C. R.

*Electric Batteries, Impts. in [Electrolytes] or relating to.* A. R. Adams, London. Eng. Pat. 3831, Feb. 12, 1897.

The author states:—

"According to my invention, instead of employing chemicals in the form of finished products for this purpose," an exciting fluid, "I make use of the crude salts." . . . . . "I mainly utilise a chromium salt in solution, treated by sulphuric acid, and added to a solution of nitric acid or a nitrate in conjunction with mercury or a salt of mercury." Combinations of such are given.—J. C. R.

*A Voltaic Battery having its Depolarisation effected by Heat.* H. E. de R. de Lavison, Neuilly-sur-Seine, France. Eng. Pat. 9803, April 17, 1897.

Claim: "An electric battery wherein the positive electrodes, made of suitable oxidisable metal, have successive portions," metal discs made to revolve, "alternately immersed in the electrolyte ['acidulated water' with sulphate of copper] and raised out of the same, while the non-immersed portions are depolarised by the application of heat thereto, such heat being supplied either by arranging the battery within a chamber, the air in which is heated by a lamp or other source of heat, or by directly heating the non-immersed portions of the electrodes by means of flames from a gaseous or liquid combustible or by heat derived from the electric current itself, or by any other means." . . . . "An element weighing about 4 kilos. has been found capable of sustaining an intensity of 30 ampères under a pressure of 0·8 volt."—J. C. R.

*Batteries, Primary [Manganese]; Impts. in and relating to.* C. Koenig, Berlin. Eng. Pat. 2024, Jan. 25, 1898.

Claim: "In primary batteries the provision of alternate concentric elements with insulating pots between each pair, and suitable exciting paste between each set of elements, all as a means for obtaining a fairly constant high voltage current from a cell of small size," &c.—J. C. R.

*Ozone, The Manufacture or Production of, and in Apparatus therefor; Impts. in.* M. Otto, Paris. Eng. Pat. 11,010, May 3, 1897.

Dielectrics, as glass, mica, &c., between the electrodes of high-tension ozonisers, are dispensed with. "Unless currents of very high potential and very low quantity—supplied, for example, by an electrostatic machine—are employed, it is not long before arcs are produced between the electrodes of opposite signs, whereby the said electrodes are rendered useless. The formation of such arcs is due principally to the reduction of the resistance of the gaseous body by the heating thereof."

In the present case, the electrodes (at least one-half of them) are movable, and "the electric discharge takes place directly into air, or into the oxygen between metal parts, which are raised to different potentials, without any dielectric whatever being interposed between the said metal parts. The movability of the electrodes (or at least one-half of the same) has for its object the permission of the discharge to take place only during a very short period, in such a manner as to avoid all heating of the electrified mass, and consequently all subsequent short-circuiting." . . . . . "One of the most simple arrangements consists in employing two parallel conducting circular discs, in which are cut out a series of parallel or slightly inclined sector-shaped holes similar to the blades of a screw-propeller, the centres of the two discs being situated on the same axis. The two discs are connected respectively to the two poles of a high-tension transformer. One of the discs being fixed, then, if the other disc have imparted to it rotary motion, a discharge will take place each time the two full sectors come opposite each other, and the discharge will cease when the discs move away from each other, or turn relatively to each other sufficiently to bring the full sectors in one disc opposite the holes in the adjacent disc." It is stated that with 18,000 volts, the distance of electrodes apart should be about three centimetres. The claims embrace, (1) the use of electrodes that are movable relatively to one another for effecting polymerisation, combination, or decomposition of gases, &c.; and, (2) the apparatus shown and described for the manufacture of ozone and other substances [hydrocyanic acid, from nitrogen and acetylene; nitrated products, from nitrogen and oxygen; and ammonia, from nitrogen and hydrogen].—J. C. R.

*Bleaching Liquids, Apparatus for the Electrolytic Treatment of.* M. Haas, Aue, Germany. Eng. Pat. 1697, Jan. 21, 1898.

The apparatus consists of an electrolytic vessel containing upright electrodes, preferably bi-polar, and so arranged that the electrolyte flows alternately under and over them. Sediment chambers, which are removed from the influence of the electric current and the flow of the liquid, are provided at the bottom of the trough between each pair of bi-polar electrodes, and are formed by alternate perforated and unperforated cross ridges of insulating material under each electrode.—G. H. R.

## (*B.*)—ELECTRO-METALLURGY.

*Silver, Electrolytic Deposition of, and Separation from Copper.* F. W. Küster and H. von Steinwehr. Zeits. f. Elekrochem. **4**, 1898, 451—455.

Analyses of silver coins carried out at Göttingen by the authors, in accordance with the methods published in well-known text-books upon electro-chemical analysis, yielded incorrect results owing to the formation of spongy silver at the cathode. A thorough investigation of the conditions of silver deposition from solutions of the nitrate was therefore undertaken. The authors found that the maintenance of a constant E.M.F. during the *whole period* of the deposition was the *sine quâ non* for obtaining reliable deposits of silver. The cause of the greater number of failures to obtain correct results is, in their opinion, the variation in the E.M.F. used during the electrolytic deposition of the silver. If a current of higher E.M.F. than 1·10 volts be used, a *continual increase* in the external resistance of the circuit is necessary during the course of the electrolysis, in order to balance the increased resistance of the electrolyte due to the removal of the silver ions from the solution. This fact is generally overlooked, and when the required E.M.F. has been obtained at the commencement of the deposition, no further attention is paid to it. In order to obviate the necessity for continual additions to the resistance of the external circuit, the authors used in their experiments a Gulcher thermopile. The E.M.F. of the current produced by this can be increased or diminished at will. Details of 40 experiments bearing upon the deposition of silver from solutions of silver nitrate are given in five tables. The E.M.F. used in these experiments varied from 1·1 to 1·4 volts. Varying amounts of free nitric acid and of alcohol were added to the electrolyte. Some of the depositions were carried out at the normal temperature and the remainder at 55° C. As a result of these experiments, the authors found that silver up to 2 grms. in weight could be deposited in compact adherent form, suitable for quantitative determination, from solutions of the nitrate containing from 1 to 2 c.c. of free nitric acid (1·10 sp. gr.) and 5 c.c. of alcohol, using an E.M.F. of between 1·35 and 1·38 volts. The temperature of the electrolyte should be 55°—60° C. With an E.M.F. above or below the limits named, unsatisfactory results will be obtained. Six determinations of the silver in mint silver were also made by the authors by the electrolytic method, under the conditions named above. The results were perfectly satisfactory, and agreed closely with the figures obtained by check analyses carried out by purely chemical methods.

Prof. Küster, in conclusion, adds some remarks concerning the ionic reactions which lead to the formation under certain conditions of silver peroxide at the anode, and gives reasons for differing with the views of Revay upon this point.—J. B. C. K.

*Argentiferous Copper, Notes on the Electrolysis of.* W. Terrill. Trans. Inst. Mining and Metallurgy, April, 1898.

The impurities occurring in anode copper are the following:—Bismuth, antimony, arsenic, iron, nickel, lead, silver, and gold. Of these, bismuth, iron, and silver are always present. The impurities caused by the above eight metals in the cathode deposit may be due either to mechanical or electrolytic transference, but only the metals bismuth, antimony, arsenic, and silver are capable of the latter.

In time the electrolyte becomes surcharged with impurities, and purification or renewal, becomes a necessity. Many methods have been proposed to effect the former. Oxidation by means of injected air, or by aid of chlorine, is often used, but as this leads to formation of peroxide salts of iron, in presence of which silver is readily transferred to the cathode by the action of the current, and the electrolyte loses its acidity, these methods cannot be recommended. The author considers that renewal of the electrolyte is the best plan to adopt, and he recommends that the impure electrolyte should be worked up to yield commercial copper sulphate. This can be used chiefly in making up the new electrolyte; the surplus which is necessarily produced must be sold.

In order to determine when the impurity of the electrolyte has reached a point dangerous to the purity of the cathode deposit, he advises the daily testing of a sample taken from the vats with a drop of bromine water. The instantaneous production of a white cloud of bismuth hydrate signifies that this point has been reached; but warning of its approach is given by the slow formation of the cloudiness in samples taken 48 hours earlier.

—J. B. C. K.

### PATENTS.

*Metals, Certain Impts. in Apparatus for the Electro-Deposition of.* W. Evans, Walsall; and J. W. Smith, Aston. Eng. Pat. 10,822, April 30, 1897.

The apparatus is of the kind in which the anodes, cathodes, electrolyte, and the articles to be plated are contained in a revolving water-tight barrel. The anode plates are fixed round the inner walls of the barrel by perforated boards, and are made with their end strips projecting through and turned down outside and against the barrel end, and are connected to a plate, and the metal bush carrying the axle end and forming the positive pole of the apparatus. The bush at the other end of the barrel forms the negative pole and is connected with a central cathode rod carrying metal discs and distance blocks, or, in lieu of this, two metal rings fixed inside the barrel and connected with the bush by insulated wires, may be employed as cathodes.

—G. H. R.

*Zinc Chloride, Impts. in the Electrolysis of Fused.* [*Dehydration.*] F. M. Lyte, London. Eng. Pat. 11,190, May 5, 1897.

The improvement consists in the complete dehydration of the fused zinc chloride before it is electrolysed between a carbon anode and a zinc cathode as described in Eng. Pat. 15,813, 1895 (this Journal, 595, 1896). The dehydration is effected by digesting the fused zinc chloride in the presence of metallic zinc, preferably fused, and this may be carried out either in a separate vessel or in the electrolytic cell described in Eng. Pat. 7594, 1893 (this Journal, 958, 1894), and the process may be assisted by passing a current through the electrolytic cell, using the zinc as anode and the carbon as cathode. When the bath is completely dehydrated, the current is reversed.—G. H. R.

*Aluminium or other Metals, Improved Processes for Coating with Nickel or other Metals.* G. Weil, E. Quintaine, and C. Lepsch, all of Paris. Eng. Pat. 12,691, May 22, 1897.

The aluminium surface is prepared as is usual with metals which are to be electroplated, and are then made the cathodes in an electrolyte of nickel, cobalt, gold, silver, or copper, containing a carbohydrate. The following solutions are recommended for the several metals:—(1.) Double sulphate of nickel (or cobalt) and ammonia, 80 lb.; ammonium sulphate, 25 lb.; lactose, 3 lb.; water, 60 galls., the bath to be used preferably at 140° F. (2.) Gold chloride, sodium bisulphite, ammonia, of each 1 oz.; sodium phosphate, $\frac{1}{2}$ lb.; pure potassium cyanide, $\frac{1}{2}$ oz.; lactose, 6 oz.; and water, 12 galls., to be used cold or warm. (3.) Silver nitrate and potassium cyanide, of each $1\frac{1}{2}$ lb.; ammonia, 2 lb.; lactose, 1 oz.; and water, 12 galls., to be used cold. (4.) Copper sulphate, $3\frac{1}{2}$ lb.; ammonium sulphate, 1 lb.; lactose, 4 oz.; and water, 20 galls., to be used cold. Maltose, raffinose, or melezitose may be substituted for lactose.—W. G. M.

*Aluminium or other Metals, Coating [Electrolytically], with Silver or Other Metals; Improved Processes for.* G. Weil and A. Levy, Paris. Eng. Pat. 22,961, Oct. 6, 1897.

"Although specially adapted for the coating of aluminium [electrolytically with silver or copper], the processes are applicable to the coating of other metals." . . . "Baths composed of metallic and alkali salts to which has been added one of certain definite substances belonging to the class of so-called *photographic developers*, viz., hydroquinone, pyrocatechin, or pyrogallate or gallate of ammonia." . . . . "For coating with *silver* we prepare a bath of: Water, 12 galls.; nitrate of silver, $1\frac{1}{2}$ lb.; cyanide of potassium, $1\frac{1}{2}$ lb.; and liquid ammonia, 2 lb. To which is added crystallised hydroquinone, 5 oz., or an equal quantity of pyrocatechin. This bath is preferably employed cold." . . . . "For coating with copper: Water, 40 galls; crystallised chloride of copper, 10 lb.; liquid ammonia, 12 lb.; and cyanide of potassium, 11 lb. To which is added pyrogallate of ammonia, $1\frac{1}{2}$ lb.; or this may be replaced by an equal amount of gallate of ammonia. This bath may be used hot or cold; preferably a temperature of 100° F. is employed. The best results are obtained with alkaline baths . . . but may be rendered neutral or acid by means of organic acids—gallic, tartaric," &c. The claims are: "(1) In the electro-deposition of metals, the employment in an electrolytic bath of hydroquinone or of pyrocatechin; and, (2) in the electro-deposition of silver or copper on aluminium or other metals the employment of baths composed substantially of the ingredients and in the proportions specified."—J. C. R.

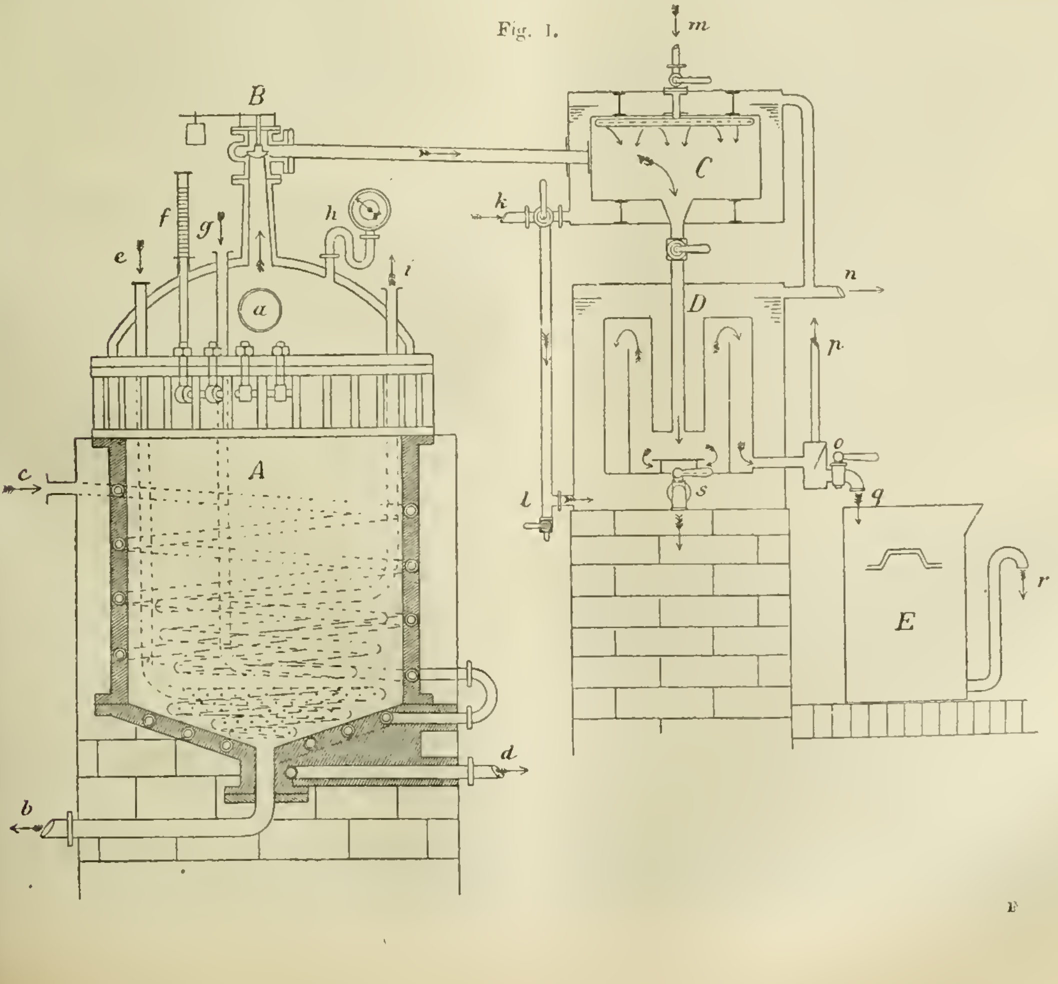
*Metals, Impts. in Appliances used in Connection with the Electro-Deposition of.* F. Greenfield, Birmingham. Eng. Pat. 21,976, Sept. 25, 1897.

In this apparatus instead of the ordinary cathode wires, suspending staffs are employed which are preferably enamelled at non-conducting parts, and have in electrical contact with them detachable branches or work-holding brackets; or the staff may be formed of wood or other insulating material, and be provided with metallic conducting strips making contact with the detachable brackets. These brackets or carriers are given various forms to suit the shape of the articles to be plated, and may consist of bars, plates, rods, or the like, either with or without plain or screwed holes, gaps, hooks, notches, or of pierced or openwork bottomed trays.—G. H. R.

## XII.—FATS, OILS, AND SOAP.

*Hydrocarbons, Oils, and Fats; Distillation under High Pressure of.* Fr. Boleg. Chem. Rev. Fett- u. Harz-Ind. 5, [1], 9—12; [2], 24—27.

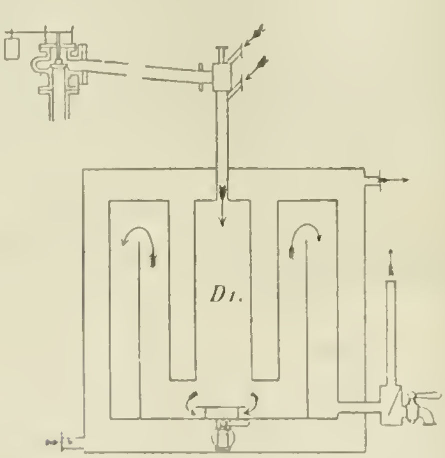
Fig. 1.

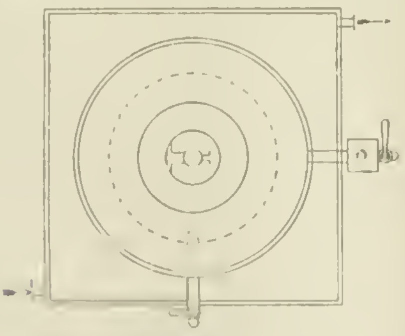


THE general scheme of the apparatus is shown in the annexed figures, though the details may be varied according to the requirements of each case. For moderate charges and pressures a simple, strong, cast-iron still with thick rounded bottom will suffice, but where high pressure is needed and direct fire dispensed with, the (wrought-iron) still should be jacketed with cast-iron; or the Frederking stills (A, Fig. 1), the cast-iron walls of which enclose a hollow spiral, may be employed.

B (Fig. 1) is an adjustable pressure-valve through which the vapours in A escape to a preliminary cooler C, where their temperature and pressure are reduced by a cold-water douche, *m*. Thence the partly condensed distillate flows into the actual condenser D (or $D_1$, $D_2$, Figs. 2 and 3, the two first being specially designed for heavy hydrocarbons, &c., and the last for lighter products), and from this, *viâ q*, into the separating vessel E, where the oil and water are separated on the Florentine bottle principle. Provision is made in the still for direct (coil *e*) and indirect (coils c *d* and *g*) steam heat.

Fig. 2.





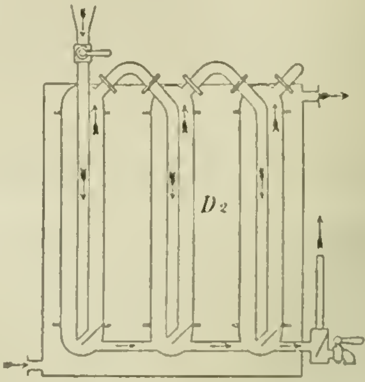
PLAN OF $D_1$.

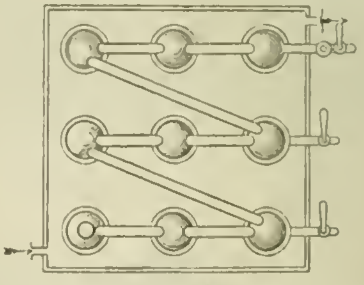
The still is charged to about two-thirds its capacity, and heated at first by indirect steam under 5—6 atmos. pressure, the steam being superheated after a while. When direct steam also is employed, it should be first raised to the temperature of the still, and should preferably be taken from the same feed pipe as the superheated indirect steam. The operation will then progress rapidly, and free from danger of explosion.

Treated in this apparatus, with direct superheated steam (4—12 atmos.), crude coal tar benzene will yield "10—14 per cent. of benzene and toluene, rectifiable below 150° C." Creosote oil, when distilled under 3—4 atmos. pressure, gives, on refining, a light, fluid, pale-orange, and almost odourless tar-oil suitable for indoor work (carbolineum); the naphthalene can also be recovered pure. A superior quality of anthracene oil is also obtained from the corresponding crude oil, treated under a pressure of 1—2 atmos. Anthracene, chrysene, and pyrene may likewise be recovered, and, by the use of a pressure of 10—12 atmos., a burning oil containing 6—8 per cent. of benzene is prepared. The residues, some hard and some soft, are suitable for working up into black varnish for ironwork, pitch for ships, &c.

The process also gives good results when employed for crude turpentine, oil of turpentine, and resin oils and spirit, an increased yield of 5—7 per cent. of normal turpentine oil (sp. gr. 0·860—0·862) being obtained, either by direct steam or by indirect steam at 200° C. In rectifying oil of turpentine, sundry essences are obtained possessing odours resembling various fruit ethers, and these are worthy of investigation.

Fig. 3.





PLAN OF $D_2$.

Whereas in the ordinary rectification of crude resin spirit, only 50 per cent. of spirit suitable for working up into oil of turpentine is obtained, the yield is increased to 80 per cent. by distillation under pressure (3—4 atmos.). Moreover, under 4—6 atmos. pressure, up to 20 per cent of utilisable rosin spirit can be recovered from "pale" (rosin) oil, 5 to 6 per cent. from blue rosin oil, and 10 per cent. from the green oil; the quality of these oils being at the same time improved, and the green oil yielding, direct, a thick, quick-drying oil suitable for the manufacture of carbolineum.

Experiments made with wood-tar oils from (fir) cellulose works, showed that by distillation under a pressure of 4—6 atmos., from 26 to 32 per cent. of refined turpentine spirit of fair quality can be recovered.

In the treatment of mineral oils, 10 per cent. more petroleum can be obtained from crude oil by (3—4 atmos.) distillation under pressure than by the usual methods, the thick residuum yielding an excellent cylinder oil. The method is also valuable in the preparation of viscous lubricating oils and vaseline from refined mineral oils.

Under the same conditions, brown-coal-tar oils yield 10—12 per cent. more solar (burning) oil, heavier and more viscous paraffin oils, and 2—3 per cent. more paraffin than usual.—C. S.

*Black Beeswax, Examination of.* G. H. Blits. Nederl. Tijdschr. Pharm. **10**, 35—43; Chem. Centr. 1898, **1**, [12], 755.

Black beeswax is found in the Dutch Indies. It is soft and sticky at the ordinary temperature, and is soluble in chloroform, with the exception of a dark-brown pollen dust, which is probably the cause of the black colour of the wax. Its melting point is 54° C. (yellow beeswax melts at 62°—62·5° C.), sp. gr. 0·978 (yellow beeswax having the sp. gr. 0·959—0·967); "iodine number, 69·06"; acid number, 34·3; saponification number, 78·05; ether number, 43·25. Eighty per cent. of the wax is soluble in petroleum spirit, and remains behind after evaporation of the solvent, as a soft sticky mass, having the acid number 35·4. The portion insoluble in petroleum spirit dissolves in ether, with the exception of a residue, amounting to 3·8 per cent. of the wax. The ether residue forms a hard, black mass, with the acid number, 26·5.—A. S.

*Soaps, Estimation of Fat and Alkali in, and Examination of the Fatty Acids by Means of the Refractometer.* C. Huggenberg. Zeits. f. öffentl. Chem. **4**, 163; Chem. Centr. 1898, **1**, [12], 756.

*See under* XXIII., *page* 496.

### PATENTS.

[*Glycerin Recovery.*] *Purification of Liquids, Impts. in the.* H. McPhail, Wakefield. Eng. Pat. 13,429, May 31, 1897.

The object of this patent is the purification of soap-makers' spent lyes, weak distillates containing glycerin, &c., by the removal of organic matter and arsenic. When the liquors are alkaline, they are nearly neutralised by the addition of sulphurous acid, or the lyes are mixed with some of the glycerin distillates, which are generally acid, owing to the presence of organic acids; then a solution of ferric sulphate is introduced till the whole is faintly acid, and the precipitated matter is removed by filtration. If preferred, the iron salt may be used before the sulphurous acid; and if the solutions contain much sodium sulphate, hydrochloric acid and ferric chloride may be employed instead of the sulphate. The operations are repeated, if necessary, until the filtrates are rendered sufficiently pure; while a still further purification can be effected by making the acidified liquors alkaline again with sodium bicarbonate.—F. H. L.

*Antiseptic Soaps containing Mercury Albuminate Impts. in the Manufacture of.* R. Ehrhardt, Augsburg, Bavaria. Eng. Pat. 2407, Jan. 29, 1898.

Antiseptic mercurial soaps are commonly prepared by mixing with the soap liquor, mercuric chloride; and, as this salt is decomposed by the alkali, the soap so prepared contains the mercury in an insoluble and not sufficiently effective state. The inventor, therefore, proposes to use mercury caseïn, which he obtains by boiling free caseïn suspended in alcohol with one-fifth of its weight of mercuric chloride, or any other mercury albuminate soluble in alkali.

In making the soap (by the cold saponification process), the mercury albuminate is first dissolved in the caustic alkali solution, which is then incorporated with the oil or fat and stirred until saponification is complete.—L. A.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (*B.*)—RESINS, VARNISHES.

*Copal-Resinates as Driers for Linseed Oil.* M. Bottler. Dingler's Polyt. J. 1898, **307**, [3], 70.

In order to test the value of the lead, manganese, and zinc salts of the organic acids contained in copal (to which reference has already been made: this Journal, 1898, 163), as driers for linseed oil, specimens of white and red Angola and yellow Benguela copal were dissolved in alkali, mixed with a solution of the desired metal, the precipitates washed thoroughly, dried at 30° or 40° C. (the manganese precipitates in partial absence of air, to prevent them darkening in colour)—and finely powdered. For the sake of comparison, similar resinates were prepared from colophony, and examined with the former. The linseed oil was heated to 150° C., and 2 per cent. of the drier added by degrees; but, as solution was not complete in the case of the copal-resinates at that temperature, the oil was afterwards raised to 165° or 180° C., and maintained thereat for a longer time. Further tests were also made by dissolving the colophony-resinates in rectified turpentine at 100° C., the copal compounds in the same solvent at about 120°, and then mixing the resulting solutions with linseed oil at about 80° to 90°.

*Colophony-Resinates.*—The manganese salt gave a clear and pale reddish-brown varnish, yielding on wood a brilliant and transparent film. At the beginning of October it dried hard on wood in 28 hours and on glass in 30 hours; in a warm room (18° to 20° C.), in 20 and 24 hours respectively. The lead salt produced a brownish-yellow varnish drying superficially in 30 hours, hard in 40 hours; in the warm, on wood in 24 hours, on glass in 30 hours. The zinc varnish dried hard in the warm, on wood in 28 hours, on glass in 32 hours. After a previous solution in turpentine, the behaviour of the manganese salt was the same as before; the lead resinate dried rather more quickly; the zinc salt was useless.

*Copal-Resinates.*—The manganese salt of white Angola copal was not completely soluble at 165° C.; even at 180° some insoluble matter remained. The varnish was yellow, and, after standing a short time, was bright and clear. It gave a very brilliant, transparent, and strong film; drying in the warm, on wood in 18 hours, becoming hard in 22 hours, hardening on glass in 24 hours. The lead salt was totally soluble at 165° C., and the varnish resembled the preceding one, but the film was even more brilliant. Its speed of drying, however, was lower. The lead and manganese salts of the other copals all behaved in an analogous manner, as did also all the copal-resinates when they had first been dissolved in turpentine. The latter method of procedure is nevertheless tedious and wasteful (for solution is not complete); and there seems nothing to be gained by resorting to it; although probably the use of old ozonised turpentine might give better results.

Experiments were tried with hot and cold pressed linseed oil; the latter yielded paler varnishes, but the drying was not affected. Altering the proportion of drier from 3 to 1·5 per cent. made no appreciable difference in the speeds. Mixtures of equal parts of linseed and hemp oil produced exceptionally brilliant films, but they dried much more slowly. After the copal-resinate varnishes had been kept for about four weeks they dried more rapidly.

Speaking generally, the lead and manganese salts of these copal acids may be said to form very efficient driers for linseed oil. They are readily soluble, and the varnishes are therefore easy to prepare. The films are all peculiarly brilliant, transparent, and permanent; and the copal-resinates should accordingly find a large sphere of usefulness in the arts. The annexed table also shows that resinate varnishes appear to dry more quickly than such as are manufactured in other ways.

*Drying of Oil-Varnishes.*

| —— | Time of Drying (Hours). | |
|---|---|---|
| | Superficially. | Hard through. |
| Manganese borate varnish (on wood)..... | 24 | 48 |
| Ozonised linseed oil (on wood)........... | 22 | 46 |
| Lead oxide varnish (on wood)............ | 36 | 72 |
| Double-boiled manganese varnish (on glass). | .. | 30 |
| Colourless photographers' copal varnish (on glass). | .. | 28 |

—F. H. L.

### PATENTS.

*Waterproof Coating and Ornamenting Paper and other Surfaces, A Composition for.* H. Bittner and C. Villedieu, Paris. Eng. Pat. 7975, March 27, 1897.

This is composed of nitrocellulose, 3 to 5 parts: acetone, or other solvent, 100 parts; toluene, 20 parts; mononitronaphthalene, 5 to 10 parts; and rosin or castor oil, 5 parts. The product may be coloured with any dye soluble in alcohol, rendered opaque with any white base, and made non-inflammable by the addition of 10 per cent. of any suitable metallic chloride (tin or magnesium, &c.). It is capable of being printed on by lithographic or chromo-lithographic processes.—F. H. L.

*Linoleum or Linoleum-like Compositions, Impts. in.* L. Knoche, Hamm, Westphalia. Eng. Pat. 12,051, May 15, 1897.

The oil of elæococca vernicia (aleurites cordata) is heated to 60° C., an equal weight of ground cork stirred in, the mixture raised to 250° for two hours, and, if desired, some resin, collodion, or castor oil added. The product becomes so plastic and doughy that it can be shaped or moulded in any manner, or it may be spread on canvas in the ordinary fashion. The process is claimed as quicker and cheaper than when linseed oil is employed for linoleum manufacture. —F. H. L.

## (C.)—INDIA-RUBBER, &c.

*Gutta-Percha.* E. F. A. Obach. "Cantor Lectures." J. Soc. Arts, 1897 and 1898, **46**, 97, 117, 137, and 169.

The crude gutta-percha found on the market at the present time is derived from a large number of different trees, which yield products of very unequal value, and some of which are still imperfectly known to botanists. The true gutta-percha tree, the Taban merah of the Malays, called by Hooker *Isonandra gutta* (erroneously, *I. percha*), is now generally spoken of in England as the *Dichopsis gutta*; while abroad it is referred to as the *Palaquium gutta*, Burck. Two of the more important sources of slightly inferior material are the Taban sutra of Perak, *Dichopsis oblongifolia*, or *Palaq. oblongifolium*, Burck; and the Sundek of Perak, or Soendi of Sumatra, *Payena seerii*, Benth. et Hook., or *Keratophorus leerii*, Hasskarl. The taban merah usually grows to a height of 60 or 80 ft., with a diameter of 2 or 3 ft. reaching maturity in 30 years. The yield of coagulated latex, as obtained by the ordinary native process of felling and ringing the bark, has been variously stated at from 13 lb. to only 11 oz. per tree Taking Wray's figures of 2¼ to 2¾ lb. at 3s. 6d. per lb., the monetary value of each tree is about 8s 9d. The latex of caoutchouc trees and balata is thinner, and these materials are therefore generally procured by tapping; but in the case of gutta-percha, if the tree be not felled, the yield is smaller, although the juice coagulates more quickly. The leaves of the *D. gutta* are characterised by a projection or beak at the end, a prominent reddish-brown tinge, and a silky lustre. They have at least 22 lateral veins on each side, branching from the midrib at an angle of 70° or 80°. The leaves of the inferior *D. polyantha* and *D. pustulata* have only 12 or 15 veins on either side, and the angle with the midrib is less than 70°. If a leaf or leaf-stalk of the genuine tree be torn asunder, a number of fine white threads stretch across the gap, a large stem requiring considerable force to break them. Leaves from inferior trees yield very few, if any, such threads; and, accordingly, but little gutta-percha is found in them. A tree 10 years old produces about 15 lb. of dry leaves, and the crop increases 1 lb. for every two years it advances in age. If a tree were plucked annually from its tenth year to its maturity at 30, it would give, theoretically, 420 lb. of leaves; and as they contain 9 or 10 per cent. of gutta-percha, this would correspond to 40 lb. at least 20 times as much as the quantity obtained by felling and bleeding an adult specimen in the ordinary way. Regarded as raw materials for the manufacture of a definite weight of pure gutta, leaves are 15 times more bulky than the raw gutta-percha.

Crude gutta-percha contains much woody fibre, bark, sand, and colouring matter (phlobaphenes), which may be classed together as "dirt," and water. After mechanical cleaning, the "gutta-percha proper" consists essentially of gutta and two or three resins, albane, fluavile, and guttane; although a certain amount of dirt still remains, the proportion depending chiefly on the nature of the materials, owing to the fact that some kinds part with their impurities much more readily than others. Of the formulæ given by Montpellier (this Journal, 1897, 925) and Oesterlé (1893, 848) for albane, the more recent figures, $C_{10}H_{16}O$, appear to be correct; fluavile is represented by $C_{40}H_{64}O_3$, which lies between the results found by the previous observers. According to Oesterlé, guttane contains 86·4 per cent. of C and 12·0 per cent. of H; but both fluavile and guttane vary in different sorts of gutta-percha. As pure gutta becomes plastic in hot water, it is evidently a mixture (alloy) of several hydrocarbons, differing probably only in their molecular constitution. It is the ingredient to which gutta-percha owes its peculiar properties; and the material must therefore be valued mainly according to the ratio existing between the gutta and the resins. In Table I. are recorded the extreme variations in the composition of raw and cleaned gutta-percha and soondie; column 6 shows the percentage of gutta-percha proper commonly found; column 9 the amount of gutta divided by that of the resins. Referring for a moment to Table II. (which, however, exhibits only the composition of cleaned samples), the same ratio, $\frac{G}{R}$, is given in column 7; but since the author finds this factor somewhat too sensitive for industrial work, he has devised the expression Q (quality), which is the percentage of resin divided by 10, letters *a*, *b*, and *c*, corresponding to ¼, ½, and ¾ respectively. If a sample is described as of 3*a* quality, it means that 10 lb. of gutta-percha proper are made up approximately of 6¾ lb. of gutta and 3¼ lb. of resins. It is, however, necessary to remember that the resins vary in composition, and also that gutta itself is by no means uniform in its physical and mechanical characteristics, more especially in its durability; so that it is indispensable to make a careful examination of the latter, as well as a mere analysis of the whole sample. Nevertheless, materials in which the percentage of resin reaches or exceeds that of gutta are always of a decidedly inferior description.

*Analysis of Raw or Clean Gutta-Percha.*—The sample, preferably in thin sheet, is cut into small pieces and divided into two portions. In one, the water is determined by heating it in a current of rarefied air or hydrogen and absorbing the moisture in a sulphuric acid tube. The second is extracted with cold ether to dissolve the resins, the solution is distilled, and the solid matter dried and weighed. The residue of gutta and dirt unattacked by the ether, is dried *in vacuo* to check the other result and then treated with carbon bisulphide or chloroform to recover the gutta. The liquid is submitted to the action of a centrifugal machine to assist in the removal of the dirt, or else it is mixed with alcohol if $CS_2$ has been employed as the solvent, or water in the case of chloroform, for the same purpose; and, after separation, the gutta solution is evaporated, dried *in vacuo*, and weighed. The process is not strictly accurate, because (1) the gutta still retains some resin and colouring matter, (2) in crude samples a little resinous matter is extracted from the wood and bark present, and (3) the resin is more completely removed from the raw than from the clean material, owing to the greater porosity of the former: still the results are close enough for practical purposes, and it is more convenient than the older method of precipitating the gutta by means of alcohol In some instances it is sufficient to estimate approximately the percentage of resin in clean gutta-percha. This may be effected by noting the increase in specific gravity of a solvent (ether) caused by the dissolution of the resins. A suitable apparatus consists of two test tubes on a stand connected together by an inverted U-tube passing to the bottom of each through caoutchouc corks. A weighed quantity of gutta-percha is put into the first tube, a measured volume of ether into the second, which also contains a hydrometer and a thermometer; the solvent is driven over to the former by air pressure, allowed to act for a certain time, then drawn back again, and the specific gravity read off. The whole apparatus is enclosed in a case to insure uniformity of temperature; and special precautions are taken to guard against loss by evaporation. By the aid of tables, the increase in density is converted directly into percentage of resin.

*Commercial gutta-percha* of the present day may be classified into four groups, which can be further subdivided into 12 typical brands. Group I.* comprises the best kinds, probably all derived from some species of *Palaquium*. Its chief brands are (1) Pahang, from the Malay peninsula (or elsewhere); (2) Bulongan red, from Borneo; (3) Banjer red, also from Borneo. Group II. contains three kinds of gutta-percha of the second order, probably obtained from some species of *Payena*: (4) Bagan goolie and (gutta) soondie; (5) goolie red soondie, both from Borneo; (6) Serapong goolie soondie, from Sumatra. Group III., comprising the "white guttas," is produced from unknown trees: (7) Bulongan white; (8) Mixed white; (9) Banjer white, all from Borneo. Group IV. consists of "mixed" or "reboiled" materials: (10) Sarawak mixed, from Borneo, a useful second-class gutta-percha; (11) Padang reboiled, from Sumatra; and (12) Banca reboiled, both of which are distinctly inferior. In its crude state, genuine gutta-percha is not distinguished by any specially large proportion of gutta; indeed, some members of the other groups contain more, but it is sharply separated from the inferior materials by the size of the $\frac{G}{R}$ ratio, or the smallness of its Q.* The cleanest raw material is (6), which contains 3·3 per cent. of dirt and about 25 per cent. of water; (9) is one of the least favourable, containing 33·3 per cent. of water and about 15 per cent. of dirt, or nearly 50 per cent. of waste in all.

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* These are the numbers used in the first and second columns of Table II., and elsewhere throughout this abstract.

*Mechanical Cleaning Process.*—The blocks of crude gutta-percha are first cut into slices by being driven mechanically against a revolving disc fitted with fluted and serrated knives. The slices are then washed in a cylindrical iron vessel provided with one or two projecting ribs on its inside, and containing a star-shaped roller. The cylinder has a number of small holes in its lower part to allow the dirt to pass through; and it is completely immersed in a case filled with water heated by direct steam. The gutta-percha is next squeezed by hydraulic power (14 to 18 cwt. per square inch) through fine gauze, then "masticated" hot in an apparatus much resembling the washer; mixed, if necessary to blend different qualities, in a "Universal mixer," and finally rolled into sheets of convenient thickness ($\frac{1}{8}$ to $\frac{1}{4}$ inch). The amount of gutta-percha recovered on the large scale is generally about 91 per cent. of that found by analysis, the net loss (not including dirt and water) being about 6 per cent. of the original raw material. The ratio $\frac{G}{R}$ is, on the whole, improved by cleaning, this being accounted for by the errors of analysis mentioned above. The percentage of waste has to some extent increased with the quantity of raw gutta-percha imported into the United Kingdom; roughly speaking, it is twice as great as it was 20 years ago. Accidents sometimes happen to the chopping machine, owing to the presence of stones, &c. concealed in the body of the material. Experiments with the Röntgen rays have shown that gutta-percha is exceedingly transparent, so that heavy foreign substances can easily be detected by their help.

*The chemical washing process* was devised by Hancock in 1846, and consists in cutting up the raw gutta-percha and treating it with caustic alkali or chloride of lime. It removes any unpleasant odour and greatly reduces the quantity of dirt; but, unless care be exercised in the subsequent washing, the action of any chemical is very liable to be injurious, causing the gutta-percha to perish after a comparatively short time. Instead of separating the impurities in raw gutta-percha by washing it with water or an alkaline solution, this may be done by dissolving it in a suitable liquid, filtering the solution and evaporating the solvent. It should seem, however, that most products obtained by the evaporation of a solution are unstable, probably because the molecular structure of the gutta is thereby affected. Nevertheless, it is perfectly safe to precipitate a solution of gutta-percha (see below); and this modification has the additional advantage of yielding practically pure gutta in place of the mixture of gutta and resins commonly known as gutta-percha.

*The chemical hardening process* is the name given to a method of improving the quality of gutta-percha by removing the resins by means of petroleum spirit (sp. gr. 0·65 to 0·67). It was introduced by Obach about 16 years ago. The crude material is sliced, dried by warm air in a revolving drum, sifted into two degrees of fineness, placed in alternate layers of fine and coarse stuff in three tanks worked in series, and systematically extracted with light petroleum till all soluble matter is withdrawn. The gutta is then masticated in presence of steam in a vessel which also serves as a still to recover the solvent; and it is finally washed in the usual way with water, or, if necessary, with an alkaline liquor. The resins are freed from petroleum in another still, and run into barrels for sale or use.

*Extraction from Leaves.*—These processes are of three kinds, according as the gutta is recovered from its solution by evaporation, precipitation, or cooling. To the first belong Rigole's method with carbon bisulphide (this Journal, 1892, 829), and Sérullas' first process with hot toluene (Eng. Pat. 11,166, 1892; this Journal, 1893, 700). In the second class are found Sérullas' improved process of solution in toluene, followed by precipitation with acetone (Eng. Pat. 654, 1896; this Journal, 1897, 52), and Ramsay's suggestion for using rosin oil as the solvent, followed by acetone as before (Eng. Pat. 17,936, 1897; this Journal, 1897, 816). The third class comprises Obach's process of dissolving the gutta in hot petroleum spirit, precipitating it by cooling the whole below 60° F. (Eng. Pat. 19,046, 1896: this Journal, 1897, 816). This method therefore requires only one liquid, and so avoids the necessity for any subsequent separation of the reagents.

Each of these processes for purifying raw gutta-percha seems to possess certain advantages. The older methods are wasteful and extravagant, inasmuch as they involve the destruction of the trees. The raw material also is liable to adulteration with worthless or detrimental substances. Nevertheless, when gutta-percha is obtained in a conscientious manner from the proper trees it is peculiarly durable, and, moreover, it can be cleaned mechanically. At first sight it would appear that the chemical hardening process should be specially suitable for the treatment of the inferior kinds of crude gutta-percha, because as a rule they contain larger proportions of resins; however, the gutta itself is of lower quality as well, so that in spite of the small percentage of resin in the hardened products, they cannot be considered fully equal to the best varieties of gutta-percha obtained directly from the trees. Extraction from detachable parts of the plant is far more economical; and, as already pointed out, the leaves are easy to identify. But all the processes hitherto devised for this purpose have been of a chemical nature, and this the author holds to be disadvantageous, since the durability of the gutta-percha when exposed to light and air is imperilled by chemical treatment.

*The physical and mechanical properties* of cleaned gutta-percha are mostly shown in Table II. The specific gravity of the material obtained from leaves by the author's process is 0·9625, the lightest of all. Para-caoutchouc has a specific gravity of 0·9275. To test the absorption of water, strips weighing about 10 grms. with an area of 1 sq. dm. were immersed for 18 weeks. The members of each group behave very similarly: I. absorbs about 1 per cent.; II., 0·7 per cent.; III., 0·45 per cent.; IV., gutta-percha from leaves, and balata, all about 0·6 per cent.—the "white" sorts thus being least absorbent. But if the specimens are reduced to pure gutta, on the assumption that it is the hydrocarbon which chiefly takes up the water, the best kinds of material are the most pervious, gutta-percha from leaves the least. Pure Para-caoutchouc is far more porous: it absorbs over 1 per cent. in one week. The softening temperature of gutta-percha rises with the $\frac{G}{R}$ ratio. It is conveniently determined by holding strips of the material under spring tension in a jacketed water-bath in contact with an electric bell and indicator, so arranged that when the spring stretches the samples, the bell rings. As recorded in Table II., column 10, however, the softening point represents the temperature at which a 2-mm. plate of gutta-percha heated in a shallow water-bath is permanently indented by the pressure of a vertical stud 3 mm. in diameter, carried at the centre of a long lever pivoted at one end and weighted with 1 lb. at the other. The "time hardening" (col. 12) is the time taken by a sample, after heating to the "pliable" temperature, to become sufficiently rigid to resist the said stud while it is kept in water at 75° F. Pliability (col. 11) is estimated by heating a strip 70 × 25 × 2 mm. in a water-bath while it is subjected to the pull of a half-oz. weight attached to its upper end by means of a cord and pulley. When gutta-percha is stretched cold, it gives way easily at first, but afterwards becomes much stronger. The tensile strengths shown in the table refer in the usual way to the original area of the strip; if they are recalculated to the final area, the figures become 12,000 lb. per sq. in. and upwards.

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* The numerous analyses of raw gutta-percha quoted in the original are not reproduced in this abstract, except as in Table I.; but the above fact is evident from the figures given for the cleaned specimens in Table II., each of which is the mean of a large number of determinations.

*The electrical properties* are shown partly in Table II., and partly, together with those of several rival substances, in Table III. Unlike the physical and mechanical properties, they are very little affected by the extraction of the resin. A low inductive capacity, which, of course, is desirable, is mostly associated with other good qualities; but this is not always the case with a high insulation. The figures in cols. 3 and 6 of Table III. refer to a cylindrical layer or tube the outside diameter (D) of which, is 10 times its inside diameter (d), so that log. $\frac{D}{d} = 1$, which is a convenient way of expressing the relationship for the cable manufacturer. As the material contains more water, its insulating power falls unmistakably; the inductive capacity also rises in a very marked manner, especialy in brands 9, 7, 1, and 4. As an example: brand 1, containing 1·5 per cent. of water, gave an insulation of 6,173 megohms and an induction of 0·0523 microfarad per cube knot; when the moisture rose to 6·3 per cent., the figures became 5,480 and 0·0675 respectively. No. 7 showed 57,910 megohms and 0·0575 microfarad at 0·9 per cent. of water; 39,030 and 0·0809 at 11·2 per cent. The dieletric strength of a layer of gutta-percha 0·127 inch thick is 40,000 volts; 0·109 inch, 28,000 volts.

The various properties of hardened gutta-percha are shown in Table IV. It is chiefly used (500 tons per annum) for the manufacture of golf balls, which are required not to become brittle at low atmospheric temperatures, and also to float in water. The material may be tested for this purpose by being made into balls of the proper size; allowing them to drop from a height of 10 ft. on to a stone slab, and noting the rebound, which should be 50 per cent. or more of the distance fallen.

*The Absorption of Oxygen* was tested by exposing two spheres of each brand of gutta-percha 8 sq. cm. in superficial area and 2 c.c. cubical contents over mercury in glass tubes filled with 30 c.c. of 91 per cent. O (the gas being renewed when it approached the composition of the atmosphere) in the open air for 24 weeks (April to October); the total sunshine being 680 hours, and the mean day temperature, 19·8 C. The members of each group behaved very similarly, II. absorbing most—40 c.c.; III. least—20 c.c.; I.—30 c.c.; gutta-percha from leaves, which is practically a hardened product, and balata — 37 c.c., as averaged results. Calculated for pure gutta, II. gave 70 c.c.; balata, 60; IV., 50; gutta-percha from leaves and I., 40; III., 37 c.c. It is curious to notice that balata and gutta-percha from leaves take up the gas at first more quickly than soondie; while there is a certain analogy between the curves for the absorption of oxygen and those representing the absorption of water. Ozone, which attacks caoutchouc very energetically, has no special action upon gutta-percha. These experiments show that for industrial purposes the material should be protected from light, and from air if possible; and they explain why submarine cores are more safely placed than subterranean wires. Specimens of casts, such as the gun impressions used by

TABLE I.

*Extreme Composition of Raw and Cleaned Gutta-Percha.*

| State of Material. | Percentage Composition. | | | | Totals. | | Ratios. | |
|---|---|---|---|---|---|---|---|---|
| | Gutta. G. | Resin. R. | Dirt. D. | Water. W. | G.P. (G.+R.) | Waste. (D.+W.) | G.P. / Waste. | G. / R. |
| | | | | | Per Cent. | Per Cent. | | |
| Raw.................... | 23·1—84·3 | 10·1—65·2 | 1·8—22·3 | 1·0—26·7 | 56·4—96·8 | 3·2—43·6 | 1·3—30·2 | 0·3—7·9 |
| Cleaned .................. | 21·4—88·8 | 11·2—67·4 | 0·9—2·0 | 0·5—2·3 | .. | .. | .. | 0·5—7·9 |

TABLE II.

*Chemical and Physical Properties of Cleaned Gutta-Perchas.*

| Description of Material. | | Percentage Composition. | | | | | Quality. | Physical Properties. | | | | Mechanical Properties. | | Electrical Properties (per Cub. Knot). | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Group. | No. of Brand. | Gutta. G. | Resin. R. | Dirt. D. | Water. W. | Ratio. G. / R. | r 10 Q. | Specific Gravity at 15°/15° C. | Temperature in C. Commencing to soften. | Temperature in C. Pliable. | Time hardening. | Tensile Strength. | Elongation. | Insulation 75° F. 2nd Min. | Inductive Capacity. |
| | | | | | | | | 2·2 mm. sheet. | | | Mins. | Lb. per sq. in. | Per Cent. | Megohms. | Microfarads. |
| I. | 1 | 78·1 | 19·2 | 1·5 | 1·2 | 4·08 | 2 | 0·9858 | 47·7 | 65·5 | 3½ | 1,577 | 427 | 2,108 | ·0535 |
| | 2 | 68·6 | 29·0 | 1·4 | 1·0 | 2·37 | 3 | 0·9911 | 45·5 | 63·3 | 8 | 3,557 | 419 | 7,643 | ·0552 |
| | 3 | 67·0 | 30·2 | 1·5 | 1·3 | 2·22 | 3 | 0·9868 | 43·8 | 66·1 | 6¾ | 3,581 | 394 | 8,400 | ·0559 |
| II. | 4 | 57·5 | 40·9 | 1·0 | 0·6 | 1·40 | 4a | 0·9709 | 40·0 | 61·6 | 9½ | 2,431 | 379 | 5,728 | ·0521 |
| | 5 | 55·2 | 42·9 | 1·2 | 0·7 | 1·29 | 4a | 0·9729 | 39·4 | 60·5 | 20 | 2,094 | 372 | 3,739 | ·0563 |
| | 6 | 56·2 | 42·4 | 0·9 | 0·5 | 1·33 | 4a | 0·9767 | 40·5 | 60·5 | 15¾ | 2,371 | 391 | 33,590 | ·0537 |
| III. | 7 | 52·2 | 45·4 | 1·5 | 0·9 | 1·15 | 4c | 1·0093 | 41·1 | 67·7 | 23¾ | 2,474 | 426 | 44,220 | ·0574 |
| | 8 | 49·8 | 47·4 | 1·1 | 1·7 | 1·05 | 5 | 1·0186 | 42·7 | 76·1 | 21½ | 2,548 | 364 | 67,800 | ·0606 |
| | 9 | 51·8 | 44·1 | 1·8 | 2·3 | 1·17 | 4b | .. | 42·2 | 73·3 | 28½ | 2,891 | 409 | 50,000 | ·0621 |
| IV. | 10 | 55·6 | 40·9 | 1·8 | 1·7 | 1·36 | 4a | 0·9912 | 40·0 | 69·7 | 27 | 2,169 | 384 | 17,060 | ·0618 |
| | 11 | 50·3 | 45·8 | 2·0 | 1·9 | 1·10 | 4c | 0·9880 | 38·3 | 62·7 | 78 | 1,892 | 446 | 62,520 | ·0613 |
| | 12 | 46·8 | 51·1 | 1·1 | 1·0 | 0·92 | 5a | .. | 39·4 | 62·7 | 63 | 1,638 | 390 | 46,750 | ·0571 |

### TABLE III.

*Electrical Properties of Gutta-Percha and some other Materials.*

| Description of Material. | Insulation Resistance. Megohms. | | Inductive Capacity. Microfarads. | | |
|---|---|---|---|---|---|
| | Per Cube Knot. | Per Knot. (Log. $\frac{D}{d}$ =1.) | Air = 1. | Per Cube Knot. | Per Knot. (Log. $\frac{D}{d}$ =1.) |
| **GUTTA-PERCHA:** | | | | | |
| Cleaned, commercial, highest | 139,300 | 51,050 | 4·196 | 0·0801 | 0·2184 |
| " " lowest | 382 | 140 | 2·619 | 0·0466 | 0·1272 |
| From leaves, Sérullas' (Kelvin's tests) | 57,980 | 21,260 | 2·950 | 0·0525 | 0·1433 |
| " " " (Hopkinson's tests) | 120,700 | 44,260 | 3·120 | 0·0555 | 0·1515 |
| " " Obach's | 48,630 | 17,830 | 2·707 | 0·0482 | 0·1315 |
| Willoughby Smith's | 955 | 350 | 3·122 | 0·0556 | 0·1515 |
| **BALATA** | 2,145 | 786 | 2·724 | 0·0485 | 0·1323 |
| **CAOUTCHOUC:** | | | | | |
| Pure vulcanised | 130,000 | 47,660 | 2·662 | 0·0474 | 0·1293 |
| Mixed " | 81,700 | 29,950 | 3·405 | 0·0606 | 0·1654 |
| Pure unvulcanised | 15,410 | 5,659 | 2·505 | 0·0446 | 0·1217 |
| Submarine cable | .. | .. | 3·561 | 0·0634 | 0·1730 |
| Overland " | .. | .. | 3·405 | 0·0606 | 0·1654 |
| **EBONITE** | 16,540 | 6,061 | 3·160 | 0·0562 | 0·1534 |
| **PARAFFIN WAX** | 34,230 | 12,550 | 2·310 | 0·0411 | 0·1122 |
| **SULPHUR** | 21,180 | 7,764 | 3·825 | 0·0681 | 0·1858 |
| **RESIN:** | | | | | |
| Colophony | 21,700 | 7,952 | 2·550 | 0·0454 | 0·1239 |
| From gutta-percha | 14,360 | 5,264 | 3·270 | 0·0582 | 0·1587 |
| **WATER** | .. | .. | 75·700 | 1·3480 | 3·6770 |

### TABLE IV.

*Properties of Hardened Gutta-Percha.*

| Description of Material. | Percentage Composition. | | | | | Physical Properties. | | | Mechanical Properties. | | Electrical Properties (per Cube Knot). | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Temperature in ° C. | | | | | | |
| | Gutta. G. | Resin. R. | Dirt. D. | Water. W. | Ratio. $\frac{G.}{R.}$ | Commencing to soften. | Pliable. | Time hardening. | Tensile Strength. | Elongation. | Insulation 75° F. 2nd Min. | Inductive Capacity |
| | | | | | | | | Mins. | Lb. per sq. in. | Per Cent. | Megohms. | Microfarads. |
| Medium Q., cleaned only. | 54·7 | 39·4 | 2·7 | 3·2 | 1·4 | 37·7 | 58·8 | 17 | 1,592 | 360 | 34,970 | 0·0613 |
| Same after hardening | 93·0 | 2·8 | 2·5 | 1·7 | 33·2 | 57·2 | 91·1 | $\frac{3}{4}$ | 5,662 | 285 | 27,410 | 0·0575 |
| Various hardened samples | 97·3 | 1·2 | 0·3 | 1·2 | 78·5 | 58·3 | 94·4 | $1\frac{1}{2}$ | 6,757 | 380 | 6,640 | 0·0471 |
| | 94·1 | 1·2 | 2·0 | 2·7 | 78·4 | 60·5 | 90·0 | 1 | 5,937 | 425 | 10,030 | 0·0608 |
| | 94·9 | 0·8 | 2·2 | 2·1 | 120·0 | 61·6 | 93·3 | 1 | 5,008 | 265 | 8,350 | 0·0588 |

### TABLE V.

*Composition of Raw and Cleaned Balata.* 1886—1897.

| State of Material. | Percentage Composition. | | | | Totals. | | Ratios. | |
|---|---|---|---|---|---|---|---|---|
| | Gutta. G. | Resin. R. | Dirt. D. | Water. W. | Balata. (G.+R.) | Waste. (D+W.) | $\frac{\text{Balata}}{\text{Waste.}}$ | $\frac{G.}{R.}$ |
| | | | | | Per Cent. | Per Cent. | | |
| Raw, maximum | 52·4 | 48·0 | 14·5 | 37·6 | 92·2 | 41·9 | 10·1 | 1·3 |
| " minimum | 31·1 | 27·0 | 3·7 | 0·7 | 58·1 | 7·8 | 1·2 | 0·9 |
| " average | 43·0 | 39·6 | 10·0 | 7 4 | 82·7 | 17·3 | 5·2 | 1·1 |
| Cleaned, maximum* | 56·8 | 53·0 | .. | .. | .. | .. | .. | 1·3 |
| " minimum | 47·0 | 43·2 | .. | .. | .. | .. | .. | 0·9 |
| " average | 52·1 | 47·9 | .. | .. | .. | .. | .. | 1·16 |

* The "quality" (Q.) of balata proper varies between 4*a* and 5*a*. Its specific gravity at 15° C. is 0·9731.

the War Office, which have to be kept for reference, may be varnished with advantage.

Among the *Chemical Properties* of gutta-percha, the following may be mentioned. It is not attacked by dilute (1 : 20) hydrochloric, nitric, or sulphuric acid; strong sulphuric acid chars it, strong nitric acid completely oxidises it. Strong hydrochloric and acetic acid, ammonia of 0·880, and caustic potash of sp. gr. 1·28, have no appreciable action upon gutta-percha. Ether, alcohol, acetone, petroleum spirit, turpentine, and benzene dissolve it more or less, extracting principally the resins; carbon bisulphide, chloroform, and carbon tetrachloride dissolve it entirely.

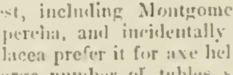
*Substitutes.*—With the possible exception of "gutta-shea," from the shea butter tree or Karité, which, however, needs further study, the only natural substitute worthy of the name is balata. Bullet trees are found in Jamaica, Trinidad, Venezuela, British, Dutch, and French Guiana. The latex is obtained by bleeding, and each gallon yields about 4 lb. of dry balata. The composition of this substance is shown in Table V., its electrical properties in Table III. The gutta is very strong and tough, but the percentage of resin is high; and balata can only be regarded as a substitute for second- or third-class gutta-percha. The resins are softer, and consist of about 2 parts of albane and three of fluavile. The artificial substances include "gutta Française," made by distilling birch bark, which is said to be more durable than gutta-percha, but renders it brittle and interferes with its electrical properties; "Velvril" (Eng. Pat. 21,995, 1895; this Journal, 1896, 910), which has not yet been tested electrically; and "Chatterton's compound," a mixture of gutta-percha, rosin, and Stockholm tar.

*Uses.*—The most important use for gutta-percha is to insulate submarine telegraph wires; and it may be calculated that up to the end of 1896 some 184,000 knots of cable had been laid in different parts of the world, absorbing 16,000 tons of gutta-percha altogether. Its property of giving an accurate and permanent image of any surface against which it may be pressed while soft is also very convenient; and it can be vulcanised and employed in a great many directions that are too well known to need recapitulation. After extraction, the resins can be mixed with small coal and pitch, and used as artificial fuel: but their low melting point and tendency to smoke deprive them of much of their value.

In an appendix the author gives some documents of historic interest, including Montgomerie's letter first describing gutta-percha, and incidentally mentioning that the natives of Malacca prefer it for axe helves even to horn. He also prints a large number of tables, some of physical and chemical valuations of various kinds of gutta-percha, extending over several years, and supplementary to the tables in this abstract. Others deal with imports, exports, and other commercial details. The tables of import show a growth in quantity with rather violent fluctuations. Since 1891 the annual figures for the amount of raw gutta-percha imported into the United Kingdom have been:—61, 45·5, 40·5, 16·7, 48, and 13·8 thousands of cwts. Prices have fluctuated slightly, but tend to rise; so that on the whole it may be taken that pure gutta-percha is of greater monetary value now than in former years.—F. H. L.

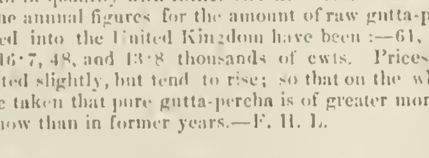
*Isoprene, On a Synthesis and the Constitution of.* W. Euler. J. Prakt. Chem. **57**, [3 and 4], 131—158.

IPATIEW and Wittorf (this Journal, 1897, 248) showed that the constitution of isoprene is identical with that of β-methyl-divinyl. Ciamician and Magnaghi, in 1885 (Ber. **18**, 2080), observed the reaction between dimethyl-pyrrolidyl ammonium iodide and caustic potash, resulting in the formation of divinyl. It might, therefore, be presumed that the application of this reaction to the already known β methylpyrrolidine—



would lead to the formation of β methyl-divinyl. The reactions by which this formation was accomplished are as follows:—

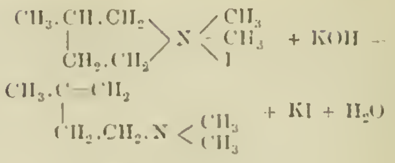
I.



β-methylpyrrolidine.

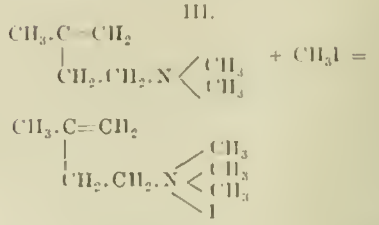
β-methyl-v-dimethylpyrrolidylammonium iodide.

II.



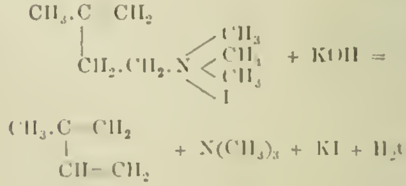
β-methyl-v-dimethylpyrrolidine.

III.



β-methyl-v-trimethylpyrrolidylammonium iodide.

IV.



β-methyldivinyl.

(Identical with isoprene from caoutchouc and turpentine.)

This identity of isoprene with β-methyl-divinyl was proved by the identity of the addition-products obtained by their treatment with hypochlorous acid. The addition-product in question contained two molecules of hypochlorous acid, and had the composition expressed by the formula $C_5H_{10}O_2Cl_2$. Being an addition-product, the original hydrocarbon must be $C_5H_8$, *viz.*, β-methyl divinyl or isoprene.—C. O. W.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Chrome-Tanned Leather, The Dyeing of.* F. Kast. Färber Zeit. **9**, 82.

*See under* VI., *page* 453.

*Sicilian Sumach, The Yellow Colouring Matters of various Adulterants of. Part V.* A. G. Perkin and P. J. Wood. Proc. Chem. Soc. 1898, [193], 101—105.

*See under* IV., *page* 451.

### PATENTS.

*Leather Articles coated with India-Rubber, Improved Process for Manufacturing.* R. Schwarzwald, Grosse Bleichen 33, Hamburg, Germany. Eng. Pat. 17,343, July 23, 1897.

BARK-TANNED leather is first cut into shape for the article, such as shoe, satchel, portmanteau, and the like, then placed upon a last or mould, and covered with india-rubber sheeting, which is then vulcanised in an air-bath, unconfined in wrappers or enclosures. This method of vulcanisation, that is, in a chamber filled with heated dry air, the articles being unenclosed in moulds, is absolutely necessary. The leather and rubber are thus intimately united, as if they consisted of one piece. The formation of wrinkles or folds is avoided, and the leather is in no wise injured, as would be the case if steam were used in vulcanising.—J. G. P.

*Removing Lime from Skins and Hides preparatory to Tanning, An Improved Process for.* J. A. Bragg, Church Street, Irthlingborough, Northamptonshire. Eng. Pat. 30,698, Dec. 27, 1897.

In this process for "bating," the natural decomposition of raw skin is used to form a decomposing solution with water, gelatin (common glue or size), or other suitable material; or a solution of gelatin decomposed or fermented by mixing with it brewers' yeast, German yeast, gluten made from flour, or any other suitable material which will decompose the gelatin. The limed skins are placed in a bath of either solution and allowed to remain until they are "down," and the lime is neutralised; they are then washed in water, and are ready to be "bran-drenched," after which they are tanned.—J. G. P.

*Glue, An Improved Manufacture of.* R. Brauer, Lüneburg, Brunswick. Eng. Pat. 2400, Jan. 29, 1898.

This invention relates to the manufacture of glue in the form of powder. The "broth of glue," obtained in the usual manner in a boiling or vacuum pan, is filled into flat castings or moulds of metal, burnt clay, porcelain, or other material, and drying is effected *in vacuo*, the glue being now in lumps or cakes. These, owing to the drying under vacuum, are extremely porous, and readily disintegrated into a dry powder, in which form it is a new article of manufacture.—J. G. P.

## XV.—MANURES, Etc.

*Alinit. (A Communication from the Agricultural Physiological Department of the Experimental Station at Prague.)* J. Stoklasa. Chem. Zeit. **22**, [20], 181.

Under the name of "Alinit," a new bacteria soil fertiliser for grain crops has been introduced in Germany. The discoverer, Caron, experimenting on various soils, succeeded in isolating certain rod-shaped and other bacteria, with which successful pot experiments were carried out. Subsequent field experiments extending over several years proving equally successful, the culture and sale have become a commercial reality.

The author has succeeded in identifying the bacterium to which the observed effects are due as the already well-known *Bacillus megatherium* of de Bary. The results are due partly to the assimilation of atmospheric nitrogen by the microbes, but much more to their power of decomposing the nitrogenous organic compounds of the soil.—H. H. B. S.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Pentosans, Rôle of, in the Manufacture of Beetroot Sugar.* Stift and Komers (from the Wochenschrift des Centralvereins). Bull. de l'Assoc. des Chim. 1898, **15**, [7], 727.

The further study of this question has completely confirmed the former conclusions of the authors that the pentosans disappear almost entirely during carbonating. These substances do not seem to undergo visible decomposition on evaporation. The authors found only very small quantities of pentosans in the first scums, and mere traces in the second, whence it follows, contrary to their former supposition, that pentosans are decomposed by the various operations of carbonating. They were surprised to find that the exhausted pulp contained notable quantities of pentosans, indicating that these substances possess a diffusive power much weaker than that of saccharose or non-sugar constituents. This explains why so small a quantity passes into the juice, in which it suffers a partial decomposition by the action of the lime. They have not been able to determine the proportion decomposed by carbonating; still they have noticed that pentosans form the greater part of the organic non-sugar of the first runnings raw sugar.

—L. J. de W.

*Beetroot Sugar Manufacture, Pentosans in the.* J. Weisberg. Bull. de l'Assoc. des Chim. 1898, **15**, [8], 802—804.

The author states that pentosan (the mother substance of the sugar pentose) and pectin (the substance which on hydrolysis, gives arabinose) are chemically one and the same body, and therefore claims to have reached, in 1888, the same conclusions in his paper on "The Rôle of Pectic Substances in Manufacture and in the Analysis of Juice" (this Journal, 1889, 206), as are given in the paper of Stift and Komers in 1897 on "The Rôle of Pentosans in the Manufacture of Sugar" (Oest. Ung. Zeits. 1897 [4 and 6], although he acknowledges that the work of these authors is richer in details and analytical results.

Stift and Komers state that the pentosans (*i.e.*, pectin) of the juice disappear in the course of manufacture, and they suppose that the lime has precipitated them in the scums. They conclude, however, that the greater part of the pentosans remains in the exhausted pulp, these bodies having thus a relatively low power of diffusion. The author showed that pectic substances pass in relatively small quantity into the juice, but are precipitated during defecation and carbonating, and are found in an insoluble condition in the scums.—L. J. de W.

*Diffusion Juice [Sugar], Existence of Ferments in.* A. M. Julhiard. Bull. de l'Assoc. des Chim. 1898, **15**, [8], 804—806.

The opinion of Jesser (this Journal, 1897, 451), that the heat of diffusion juice precludes the existence or vitality of micro-organisms is not shared by the author, who considers that the temperature of 75° C. is insufficient to sterilise the juice. No doubt at this temperature the germs do not develop, but they are not killed, and may continue their evolution when the temperature falls, as is actually the case, since the juice leaves the diffusers at 28° to 35° C. When work is proceeding normally the action of these ferments has generally not time to make itself felt to an appreciable extent, but as soon as diffusion drags slightly and the juice has to stand some time before being carbonated, reducing sugars are produced in consequence of the development of germs. From observations made during last season on beetroots containing a certain proportion of reducing sugars, it was found that diffusion juice had a greater tendency to ferment than the juice of the beetroot obtained by rasping or pressing, thus proving that the micro-organisms had not been destroyed. The addition of antiseptics, as mercuric chloride, prevented the change. The microbes are probably developed for the most part in the battery, on occasion, as when a stoppage occurs. They coat the walls of the pipes, and may thus seed the juice as it is produced. It has been stated that the reducing sugars are produced by increase of acidity, but the acids are products of fermentation formed simultaneously with the reducing sugars, and very often accompanied with viscous substances. It is thus possible to account for the difficulty found by Panneako (this Journal, 1897, 452) in filtering acid juices.

—L. J. de W.

*Diffusion Juice [Sugar], Process for the Purification of.* Al. Coumbary and R. Farkatsch (Oest. Ung. Zeits.). Bull. de l'Assoc. des Chim. 1898, **15**, [8], 831—832.

The first operation is to remove mechanical impurities carried away in the juice as pulp, earth, &c.

The second operation is likewise purely mechanical, having as its object the removal of pectic and gummy matters which hinder filtration through a cloth. For this purpose centrifugal separators are used, when the above named substances being heavier, are deposited, while the lighter juice passes away by the separator. Experiments having shown that bubbles of air present in the non-sugar hindered the separation, these are removed by passing the juice, before it enters the centrifugal, through a closed apparatus connected with an air pump.

The object of the third operation is to remove substances originally in solution but rendered insoluble by heat. These are principally albuminous matters. The removal is found to be facilitated by the addition of 0·2 per cent. of milk of lime (calculated on the beetroots).

Diffusion juice thus treated is subjected to carbonating. Owing to its higher purity it is possible to treat it with only 1 per cent. of lime. The previous purification permits of the carbonating being carried to a very low alkalinity without fear of alteration. The suppression of the second, and, above all, of the third carbonating becomes thus possible.

—L. J. de W.

*Massecuites, Determination of the Density of.* A. M. Julhiard. Bull. de l'Assoc. des Chim. 1898, **15**, [7], 715—716.

To obtain the real density, Firpitz weighs a vessel of a special form, full of water at 15° C., and then full of massecuite, after having heated it on a water-bath to remove air bubbles.

To find the dry solids it is better to proceed by dessication than to calculate from the density.

In order to calculate the weight of a massecuite from its volume, the weight of a litre is required at the temperature of the masse when the volume is taken. The measure used must be gauged at this temperature, or the result will be too high.—L. J. de W.

*Sugar, Preparation of Pure.* H. Pellet. Bull. de l'Assoc. des. Chim. 1898, **15**, [8], 813—815.

Although every confidence may be placed in the preparation of quartz plates corresponding to a given weight of sugar, or rather to a known deviation related to this, it is still indispensable to verify polarimeters by means of a solution of crystallisable sugar. For a practical and expeditious verification, chemically pure sugar is not necessary. Any good commercial sugar containing no reducing substances will suffice. The moisture and ash can be determined with great accuracy, and the organic matters may be calculated from the ash by assuming the same ratio as found from the analysis of refinery molasses, that is, about $\frac{4}{5}$ths of the ash. The difference corresponds very closely to pure saccharose.

Varying weights of this sugar are dissolved and made up to 100 c.c., and a great number of points on the scale of the polariscope verified from these. This method is more exact than that of diluting a standard solution. The same flask is used throughout.

However, pure sugar may be required for certain researches, and there are several methods proposed for preparing it.

Raffinose need not be expected in refined sugar, even when made from sugar extracted from molasses.

To prepare pure sugar it has been proposed to wash powdered refined sugar with alcohol more or less strong, and, finally, with absolute alcohol, and to dry over sulphuric acid at a temperature not above 50° C. This method does not give absolutely pure sugar, as some of the impurities held by the sugar are not dissolved by alcohol.

It has also been proposed to dissolve refined sugar in distilled water and to precipitate it with alcohol. This appears to be the only correct and exact method. 70 parts of sugar are dissolved in an equal weight of water, assisting the operation by heating to 60° C. An equal volume of 95 per cent. alcohol is slowly poured in, with stirring, the temperature being maintained at 60° C. When the precipitated sugar has settled, the liquor is decanted and the sugar washed with warm, strong alcohol. It is then collected on a filter and dried over sulphuric acid at 30° to 40° C.

Another method is to add the alcohol very gradually without causing an immediate precipitation of the sugar. On standing, the excess precipitates in magnificent crystals, which may be considered pure. To obtain absolutely pure sugar the operation may be repeated two or three times.

The procedure is as follows:—500 grms. of refined sugar are dissolved in 250 grms. of distilled water at 40° to 50° C. The solution will occupy about 566 c.c. From 560 to 570 c.c. of absolute alcohol are added very slowly, with stirring, so that the whole remains clear. The solution is allowed to cool slowly, when the sugar crystallises and adheres to the sides of the vessel. To facilitate the removal of the crystals, the liquid should be contained in a porcelain dish, and when the crystals cease to increase in size, the dish is placed in warm water to detach them. The liquor is decanted and the crystals washed with alcohol and dried. About 230 to 250 grms. should be obtained.

The sugar, on keeping, absorbs a little moisture. On weighing out a sample this should be determined and allowance made for it.—L. J. de W.

*Inversion of Sugar by Normal Salts in Presence of Glucose.* H. C. Prinsen-Geerligs. Arch. f. Suikerindustrie, 1895; Chem. Centr. 1898, **1**, [12], 711.

The author has previously shown (Arch. f. Suikerindustrie, 1895) that cane sugar is inverted by normal salts in presence of glucose. The amount of cane sugar inverted depends chiefly on—and is nearly proportional to—the duration of the action, and on the quantity of glucose originally present. The quantity of the normal salt used, exercises little influence. The action depends on the liberation of the acid from the salt by the glucose, which itself combines with the base to form a glucosate, and therefore, if calcium carbonate or the salts of weak organic acids be employed, the inversion is more or less completely arrested. The amount of acid set free by the glucose was, however, found, by comparative experiments, to be very small. The efficiency of different salts increases with the degree of their dissociating power; thus, magnesium chloride and the chlorides of the alkaline earths act better than alkali chlorides. The action also varies according to the inverting power of the acid combined in the salt. For example, with potassium salts, the inverting power diminishes in the following series:—Chloride, bromide, iodide, nitrate, sulphate, chlorate, acetate, oxalate, tartrate. Besides glucose, dextrose, levulose, galactose, and lactose, the mono-saccharides in general exercise this inverting action, but not the di- and tri-saccharides. Alcohols also do not act in this way.—A. S.

*Glucose, Action of Normal Salts on, at a High Temperature.* H. C. Prinsen-Geerligs. Arch. f. Javasuikerindustrie, 1898; Chem. Centr. 1898, **1**, [12], 712.

In addition to the inversion of cane sugar effected by normal salts in presence of glucose (see preceding abstract), the latter also act on glucose itself at 100° C., part of the glucose being converted into levulose, and, with excess of the latter, part of the levulose is re-converted into dextrose. Lobry de Bruyn and Ekesteyn have previously shown (Arch. f. Suikerindustrie, 1896, 221; 1897, 349; and this Journal, 1896, 210) that this reaction is effected by small quantities of alkalis or of sodium acetate. The author found that alkali salts of weak organic acids act tolerably strongly in this manner, whilst the salts of strong mineral acids do so only to a very small extent. The reaction increases with the duration of the action and the concentration of the salt solution. In the course of the action a small portion of the glucose is converted into organic acids, and, as these acids absorb iodine, this explains why too much dextrose is sometimes found when using Romijn's method of analysis with iodine solution containing borax (this Journal, 1897, 765). The presence of these acids is, however, immediately revealed by the formation of iodoform. The presence of salts of organic acids in cane sugar molasses also explains the fact that, with the formation of much invert sugar, the rotation of the molasses is only very little to the right or left.—A. S.

*Sugar Honey.* A. Rohrig. Zeits. für öffentl. Chem. **4**, 174—178; Chem. Centr. 1898, **1**, [12], 711.

The raw material now used for the preparation of sugar honey [Zuckerhonig] is the invert sugar syrups obtained by various processes. The hydrolytic decomposition of the cane sugar by means of hydrochloric acid, carbonic acid, or organic acids may be either partial or complete, according to the concentration of the acids. In the first case there is formed invert sugar syrup, which is useful for the improvement of wine, as food for bees, and for the manufacture of liqueurs. This syrup will keep for 12 months without crystallising, even with a concentration up to 80 per cent. of sugar. In the latter case there is formed a mixture of dextrose and levulose, which possesses the property of solidifying, and which, in outward appearance, closely resembles natural honey. Three invert sugars of the latter kind showed on analysis:—Cane sugar, 40·02, 33·00, and 34·72 per cent.; invert sugar, 39·47, 36·04, and 35·01 per per cent.; water and non-sugar, 20·29, 30·92, and 30·11 per cent. The two varieties of commercial sugar-honey showed:—Invert sugar, 63·58 and 60·56 per cent.; cane sugar, 7·8 and 4·3 per cent.; non-sugar, 8·8 and 12·95 per cent.; and water, 19·5 and 22·16 per cent. The non-sugar may be almost completely precipitated by alcohol, and consists mainly of a mixture of different dextrins. The analytical detection of sugar honey in natural honey cannot at present be carried out with certainty.—A. S.

*Starch, The Hydrolysis of, by Acids.* H. Johnson. Proc. Chem. Soc. 1898, [193], 106—107.

It has been generally believed that the hydrolysis of starch by acids is similar in character to that effected by diastase, except that the maltose, which is the final product of the action of diastase, is transformed by acids into dextrose. An examination of the products of acid hydrolysis shows, however, that these are not identical with those of diastase conversion, and that when starch is hydrolysed by dilute acids, there is neither production of amyloins (molecular aggregates of maltose and the amylin group) nor of maltose. The nature of the products resulting from the reaction can be shortly described as follows:—

The cupric-reducing powers and specific rotations of the intermediate substances (fractionated by means of alcohol), as well as those of the total products of conversion, can be expressed exactly in values of dextrose and the amylin group. The relation between the specific rotation and the cupric-reducing power is constant throughout the whole of the reaction, and, given one of these values, the other may be calculated by the equation, $[\alpha]_{D3\cdot86}x^\circ = 195 - (195 - 52\cdot8)K_{3\cdot86}/100$, in which $x$ = the specific rotation, and K = the cupric-reducing power in terms of the percentage of dextrose.

The specific rotations of the intermediate substances (separated from the dextrose by precipitation with alcohol) vary between $[\alpha]_{D3\cdot86}$ 80° and 190°. It should be remembered that the intermediate compounds in diastase conversion have rotations which vary from $[\alpha]_{D3\cdot86}$ 190° to 150°, so that the substances from the acid conversions with rotations which fall as low as 80° must differ in character from those obtained by diastase.

The behaviour of the intermediate substances from the acid conversions, when submitted to dialysis, show that they are definite compounds, and not mixtures of dextrose and other carbohydrates, as, after purification, they dialyse without undergoing a change in specific rotatory power. Moreover, they are unfermentable in the presence of Saaz and Apiculatus yeasts. When treated with phenylhydrazine acetate they yield gummy precipitates, and on further hydrolysis with acids are completely transformed into dextrose. Their solubility in alcohol decreases as their specific rotation increases.

The products of acid conversion also differ in a marked degree from those of diastase conversion when submitted to the action of diastase. The fall in specific rotation in the case of the products of acid conversion is extremely limited at any time during the reaction.

The action of dilute acids on starch can be expressed in its simplest form by the following equations.

$$(C_{12}H_{20}O_{10})n + 2(H_2O)m = \left\{\begin{matrix}(C_{12}H_{20}O_{10})n-m \\ (C_6H_{12}O_6)m\end{matrix}\right. + (C_6H_{12}O_6)m,$$

$$\left\{\begin{matrix}(C_{12}H_{20}O_{10})n-m \\ (C_6H_{12}O_6)m\end{matrix}\right. + (C_6H_{12}O_6)m + 2(H_2O)_{n-m} = 2(C_6H_{12}O_6)n.$$

Taking into consideration their properties, the substances intermediate between starch and dextrose may be regarded as molecular aggregates of dextrose and the amylin group $(C_{12}H_{20}O_{10})n$; the name *gluco-amylins* will accurately describe them. Gluco-amylins with rotations of about $[\alpha]_{D3\cdot86}$ 80° or 90° have been recognised in commercial glucose under the name of gallisin.

The formation of gluco-amylins by acid hydrolysis is thus explained: acids, being able to hydrolyse free maltose, exercise this action also on the maltose in molecular aggregates, and therefore amyloins at the moment of their formation would be transformed into dextrose and gluco-amylins. The molecule of starch is probably formed by the condensation of a large number of molecules of dextrose. In the first place, two molecules of dextrose condense to form maltose, and then a large number of maltose molecules further condense to form starch.

*Beetroots for Seeding Purposes, Pellet's Method of Analysis of.* L. Lindet. Bull. Soc. d'Encouragement, 1898, **97**, [2], 117.

*See under* XXIII., *page* 496.

*Strontium Hydrate [Sugar], Complete Analysis of,* V. Zamaron. Bull de l'Assoc. des Chim. 1898, **15**, [8]. 816.

*See under* XXIII., *pages* 492 *and* 493.

*Grape Sugar, Milk Sugar, and other Bodies capable of reducing Fehling Solution; Volumetric Method for the Estimation of.* E. Riegler. Zeits. Anal. Chem. 1898, **37**, [1], 22.

*See under* XXIII., *page* 499.

*Molasses, Analyses of Beetroot and Cane.* H. Pellet. Bull. de l'Assoc. des Chim. 1898, **15**, [7], 705.

*See under* XXIII., *page* 498.

*Starch, Comparison of the Standard Methods for the Estimation of.* H. W. Wiley and W. H. Krug. J. Amer. Chem. Soc. 1898, **20**, 253.

*See under* XXIII., *page* 498.

### PATENT.

*Syrups, Process for Purifying Black-Strap or Refuse.* E. W. Gerbracht and F. G. Wiechmann, New York, U.S.A. Eng. Pat. 29,630, Dec. 14, 1897.

Black-strap, or refuse syrups resulting from the processes of refining sugars, especially beet sugar, is purified by subjecting it, at a density of about 20° B., to the action of an oxide or hydrate of a metal, as lime or baryta; boiling *in vacuo* to remove offensive matters by distillation, and then treating the mixture with an acid, as phosphoric or sulphurous acid, which combines with the metallic oxide or hydrate; and again boiling *in vacuo* to distil off part of the organic acids.

The resulting syrup is finally subjected to the usual processes for filtering, clarifying, and concentrating.

—L. J. de W.

*Refining of Raw Sugar, Improved Process for the.* C. D. Abel, London. From Eugen Langen's Erben, Cologne, Germany. Eng. Pat. 1285, Jan. 17, 1898.

The raw sugar is mashed with water instead of with syrup, and introduced into the vacuum pan, and heated so as to expel the air contained therein, after which the syrup, which is to be boiled down with the sugar, is introduced, and the mixture is then boiled down so as to deposit the dissolved sugar upon the existing crystals. The result is, by avoiding frothing, to shorten the process from seven to four hours.—L. J. de W.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Germination, Influence of Formaldehyde [Formaline] on.* R. Windisch. Landw. Versuchsstat. **49**, 223; Zeits. für das ges. Brauw. **21**, [6], 68.

The experiments were conducted with barley, wheat, oats, and rye, 200 selected corns of each being embedded in thick Swedish filter paper and steeped for 24 hours, 100 c.c. of steeping liquid being used in each case.

From the tabulated results it appears that the seeds employed were very sensitive to the influence of even greatly diluted solutions of formaldehyde, the germinative energy varying inversely with the amount of that reagent used, though the influence of a given strength differed with the nature of the seeds. For example, a 0·02 per cent. solution injured the germination of barley, wheat, and rye, on the first day, whereas a favourable influence was evidenced in the case of oats. A 0·03 per cent. solution exhibited a similar action; whilst one of 0·08 per cent. strength greatly retarded the course of germination, the effect being greatest in the case of rye and wheat, but smaller on barley; oats germinated normally.

Considerable damage was effected by a 0·12 per cent. solution, only 9·25 per cent. germinating in the case of wheat; rye, 20·5 per cent.; barley, 47 per cent.; oats, normal but greatly retarded. With a 0·2 per cent. solution, only three corns of rye, but none of either wheat or barley, germinated at all; and oats were greatly injured, the germination being delayed for a considerable time, though 64½ per cent. of the corns grew. The germs were in all cases destroyed by a 0·4 per cent. solution.—C. S.

*Malting Experiments on the Small Scale.* G. Luff. Zeits. für d. ges. Brauwesen, **21**, [1], 6—8; [2], 16—19; [3], 27—29.

To derive the utmost advantage from malting experiments, the author considers that these should be conducted on principles at least approaching those underlying the practical process, and should be preceded by a due consideration of the percentage of tailings, impurities, and germinating corns.

In the estimation of starch he proposes to amend the usual method by making up the filtered extract from 3 grms. of barley to 250 c.c., and then inverting in the measuring vessel. The concentration never exceeds 1 per cent., but double the usual amount of copper is used. To ensure accuracy, the water content of the barley should be estimated twice over, once in the coarsely crushed state and again after reducing to fine powder for the starch estimation, the difference being borne in mind in expressing the results.

1. *Steeping.*—One thousand grms. of barley were freed from dirt, &c., by repeated immersion and stirring in fresh quantities of water, the floating corns being collected, dried in the air, weighed, and their dry matter determined. If their weight does not exceed 1—2 *per mil* they may be returned to the bulk.

The washed barley was then placed in a perforated metal cylinder 18 cm. long and 12·5 cm. wide, terminating in a cone 11 cm. in length, and immersed in the steeping water at a constant temperature of 10°—11° C., the water being changed twice the first day, and once a day afterwards, the cylinder being left to drain 10 minutes each time and then weighed. The actual percentage of water to be absorbed by the barley before steeping can be regarded as complete, varies with the class of barley and the conditions of floor management, but in all cases the actual absorption can be roughly determined from the increase in weight of the cylinder referred to the total dry matter of the sample. In the present experiments the barley, when pronounced by a maltster as properly steeped (time 72 hours), contained 46·5 per cent. of moisture—a proportion advantageous for the working temperature (11°—12½° C.) of the floor. The barley contained 66·22 per cent. of starch, and 11·56 per cent. of protein substances (dry).

The Ashton diaphanoscope was employed to test the modification of the barley, a batswing burner set broadside to the instrument, at a distance of 10 cm., being used. Thirty-seven per cent. of the corns were opaque except at the apex, 11 per cent. being transparent over about half the total area and 10 per cent. mainly transparent. The figures yielded by the same barley in practical work were 50, 10, and 9 per cent. respectively.

The capacity of barley for absorbing moisture is apparently indefinite, a sample that took up 48·6 per cent. in 73 hours having absorbed 54·2 per cent. after 217¼ hours' steeping; but an immoderate percentage of water greatly injures germination.

2. ***Malting.***—To conform as closely as possible to practical conditions, *i.e.*, exposure of a stratum of suitable thickness to an atmosphere saturated with moisture (which facilitates "sweating" in the heap), at a constant temperature, the best kind of germinating vessel is one of tin plate, 18 cm. high and 17 cm. in diameter (for 1,000 grms. of barley), radiation being prevented by an outer jacket lined with cork cuttings. Instead of a bottom, the vessel should rest on a Solenhofer plate, and, if not kept on a malting floor, should be placed in a room, or outer vessel, ventilated, and filled with air saturated with moisture and at a constant temperature. The apparatus used in these experiments was, however, unprotected in the manner suggested. In place of turning, the sample was emptied twice a day by the hand from one vessel to another.

With a floor temperature of 11°—12½° C., and a germinating period of 8½ days, the sample attained a maximum temperature of 16·87° C. on the fifth and sixth days, decreasing to 15° C. at the close; whereas the ordinary malt-heap on the same floor was somewhat warmer, rising to 19·37° C., and, after receding to 15·6°, closing at 18·75° C. One important result of this difference was revealed in the smaller loss by evolution of gas sustained by the sample, *viz.*, 5·98 per cent. as compared with 11·68 per cent. loss in the heap, the final condition of the two malts being the same; in fact, the small sample proved superior in extract (75·05 : 73·27 per cent.) to the bulk. This saving of material by working at a lower temperature will be of great practical importance, provided the large scale experiments on this point now in progress confirm the above results.

Tests made with regard to the influence of the period of germination showed that handling the malt ten days on the floor instead of eight did not appreciably affect the modification, but increased the loss by gasification about 1·2 per cent.

3. *Withering.*—The sample was spread out on a wide board and left to wither for 24 hours in an airy room without heat.

4. *Kilning.*—The apparatus employed, consisted of a cylindrical metal vessel 10½ ins. in diameter and 4 ins. deep, fitted with a lid and a false bottom, and immersed in an oil bath to the level of the latter. The oil bath was heated by gas, and warmed the air-supply to the curing chamber, the air being delivered through a number of pipes projecting from the bottom of the chamber downwards through the bath, the temperature being regulated automatically by an arrangement controlling the supply of gas. The operation lasted 24 hours, 12 of which were occupied by the withering, 10 in raising the temperature to the maximum, and two by the final stage of curing, the temperature being adjusted to produce a malt yielding a wort equal in colour-depth to 0·8—0·9 c.c. of $\frac{1}{10}$ iodine. During the preliminary stage, a rise of temperature occurs, and outward indications point to a continuance of enzymic action in this stage. When the curing is completed, the malt is removed from the chamber to prevent further deepening of the colour, and is freed from the culms after cooling; the weight and dry matter of the latter being ascertained at once, whilst the malt itself is left exposed to the air for 1—2 days before examination. All the weights obtained are referred to the dry matter of the barley, in order to obtain comparable figures.—C. S.

*Malt, The Ready-Formed Sugars of: with some Observations on the Action of Diastase on Barley Starch.* A. R. Ling. J. Fed. Inst. Brewing, 1898, **4**, 187—202.

ALTHOUGH as early as 1844 the question of the occurrence of sugars in the seeds of cereals was discussed, and experimental evidence, *pro* and *con*, is to be found in the literature, the first definite proof was furnished in 1875, when Kühnemann (Ber. **8**, 202 and 387) isolated cane sugar from barley, in which he also recognised the presence of reducing sugars. He showed that germinated barley contains more sugar than ungerminated barley. Kühnemann's observation was confirmed by the researches of Brown and Heron, C. O'Sullivan, Kjeldahl, and others.

It is pointed out that no chemical or physiological reasons are known to account for the formation of cane sugar from the reserve materials of barley. It was, however, shown by Brown and Morris (J. Chem. Soc. Trans. 1890, 458) that, during the germination of barley, the starch of the endosperm is to some extent transformed into maltose by the diastase of secretion, this maltose being absorbed by the columnar epithelium of the embryo and converted by the metabolic processes of the living cells of the latter into cane sugar. Brown and Morris cultivated excised barley embryos on a solution of maltose, and thereby demonstrated that the amount of cane sugar increased in these.

C. O'Sullivan extracted barley with alcohol, and in this way isolated a small quantity of raffinose from it. By the same means he rendered it highly probable that both germinated and ungerminated barley contain, besides cane-sugar, maltose, dextrose and levulose.

*Estimation of Ready-formed Sugars in Malt.*—The first to devise a general laboratory method for this purpose was Heron (this Journal, 1888, **7**, 259), and subsequently, in 1891, another method was proposed by Moritz and Morris in their "Text Book of the Science of Brewing." The first stage of both these methods is the same, namely, the malt is mashed with water at the ordinary temperature for three hours. The author shows that diastatic action occurs on the starch of the malt during extraction with cold water, amounting, in some cases, to upwards of 2 per cent. of maltose, calculated on the malt. This being so, it became necessary to discover some agent which would arrest diastatic action during the extraction of the malt. The experiments of Morris (this Journal, 1896, **15**, 562) indicate that a 0·05 per cent. solution of salicylic acid has a higher extractive value than that of water, and the use of a more concentrated solution is undesirable (compare also Jalowetz, Alg. Zeit. f. Bierbrau u. Malzfab. 1892, **20**, 1103).

The author then shows that a starch solution containing 0·0073 per cent. of hydrochloric acid is unaffected by diastase (compare Kjeldahl, Res. Comptes Rend., Carlsberg, 1881, **1**, 148). The reason that such a quantity of acid does not produce the same effect when added to malt extract is that it is replaced by the weaker acids present. The use of a sufficiently strong solution of hydrochloric acid to inhibit diastatic action is inadmissible as an agent for the extraction of the sugars from malt, on account of its action on the saccharose present. Experiment showed, however, that no diastatic action is possible with a starch solution containing 0·0168 per cent. of potassium hydroxide. Further experiments were therefore carried out, in order to ascertain if the ready-formed sugars could be extracted by a solution containing 0·0168 per cent. of potassium hydroxide in excess of that neutralised by the acidity of the malt. The results are summarised in the following table:—

| —— | Reducing Sugars as Maltose. |
|---|---|
| | Per 100 c.c. |
| 10 per cent. cold-water malt extract (extraction period, three hours). | 0·918 |
| Same extract after acting on barley starch for three hours. | 1·172 |
| Maltose formed by diastatic action in last experiment. | 0·254 |
| Malt extract corrected for maltose formed by diastatic action in three hours. | 0·664 |
| Malt extracted with 0·0168 per cent. KOH in excess for three hours. | 0·611 |
| Ditto, ditto .................................... | 0·669 |
| Ditto, the extraction continued for 18 hours..... | 0·660 |

The reducing sugars are calculated as maltose, in order that the results may be comparative. It is shown, however, that the sugars extracted from malt by an alkaline solution of the concentration above mentioned, yields, on treatment with phenylhydrazine acetate, an insoluble osazone, probably glucosazone, together with a smaller quantity of an osazone soluble in hot water, which may be maltosazone.

An experiment is quoted showing that the presence of 1 per cent. of lead acetate does not prevent diastatic action.

The author claims that he has demonstrated that the sugars present in the cold-water extract of a malt comprise a certain amount of maltose formed by diastatic action. He believes he has brought forward sufficient facts to elaborate a new method for the estimation of the ready-formed sugars, free from the objection mentioned.—J. L. B.

### *Sugar and Starch Formation in Barley and Malt.*

I. Gruss. Wochenschr. für Brau. 1898, 81—84.

The author has investigated the distribution of the sugars in barley and malt by acting on sections with boiling Fehling's solution and examining them under the microscope. In order to detect cane sugar, the sections are acted on by invertase before being subjected to the action of the Fehling solution.

He finds that no cane sugar is present in the aleurone layer of barley, but that this sugar appears there as soon as germination commences, and in greater quantity than in the other portion of the corn. Reducing sugar is only present in the aleurone layer immediately under the furrow of the germinating grain. The reducing sugars in the endosperm of the germinating corn are present in greatest quantity in the central portion, and least in the endosperm cells immediately contiguous to the aleurone layer.

The author thinks that the explanation of these observations is that the reducing sugar is converted into cane sugar in the aleurone cells or those lying immediately under it.

The first stages of germination take place without any corrosion of the starch granules, and probably at the expense of the sugars of the grain; the starch granules, however, are soon attacked and converted into sugar, but, as stated above, this only accumulates to any extent in the centre of the endosperm.

The author finds the effect of a low temperature on germinating barley is to lower the percentage of the reducing sugars.—A. L. S.

### *Malt Kilning, The Bavarian Method of; Notes on.*

C. Bleisch. Zeits. für d. ges. Brauw. **21**, [8], 89—93.

The principal aim of the method is to develop the special colour and roast aroma characteristic of Bavarian beer, and this is mainly attained by exposing the malt to a high temperature at the finish.

In the course of his researches into the changes undergone by the malt on the kiln, the author finds that about 7—8 per cent. of the water present is in a more or less combined form. He therefore considers that the water content of malt plays a somewhat analogous part to that in sodium phosphate, and as the latter is altered in composition only when the final molecules of water are driven off by heat, so the degradation of starch in the malt during kilning is effected in the presence of the mechanically retained water, the sugar and intermediate products constituting the material for the subsequent development of the aroma being thereby elaborated.

The most suitable temperature for this reaction being about 62° C., it is therefore advisable to keep the water content, exceeding 7½ per cent., as near this temperature andfor as long a time as possible, in order that the chemical action referred to may be completely developed. It follows from this consideration that both floors of the kiln play their part in the work of developing the aroma.

The most suitable water content for the malt, when transferred from the upper to the lower floor of the kiln, is about 15 per cent., the malt being still "tough." A gradual increase above 62°—70° C. should take place during the last 12—14 hours on the kiln, the rise from 85°—87° C. to 100°—106° C. being very slow (3½—4 hours), and an exposure of 1—1½ hours to the last-named temperature being the maximum permissible, since longer heating thereat is injurious to the fermentability of the malt. The author regards warm-withering (25°—37° C.) as greatly preferable to cold-withering on the floor, being less dependent on atmospheric conditions and effected away from light and air.

As a normal depth for loading the malt on the kiln, the author recommends about 9½ ins., or more if the ventilation be good. The practice of introducing cold air below the upper floor he considered an advantage, in that it prevents the too rapid removal of moisture and reduction of diastatic power by overheating.—C. S.

### *Hops, and their Influence on Microbes.*

F. W. Richardson. J. Fed. Inst. Brewing, 1898, **4**, 128—134.

Hopped and unhopped worts were fermented under comparative conditions, and the amount of alcohol produced showed that hops exert a slight but distinct retarding action upon the growth of the yeast. Pure spores of green mould (*Penicillium glaucum*) flourished freely in 5 per cent. hop decoctions, whilst cultures of *Bacillus liquidus* and *B. fluorescens nonliquefaciens* were destroyed after a few hours' exposure at 72° F. in the same medium.

With a 1 per cent. decoction of hops, after 12 hours' exposure at 72° F., *B. typhosus*, *B. salivarius*, and *Micrococcus pyogenes* (Rosenbach) were killed; but *B. coli* was living and active, and it required 72 hours to destroy this hardier microbe.

With a 5 per cent. decoction of hops, also with an aqueous infusion from hop resins amounting to 20 per cent. on the weight of the liquid, and with an exposure of 70 hours, all the above-mentioned organisms were killed. A similar result was obtained with Bass's beer after 90 hours at the same temperature. After an exposure of 96 hours at 72° F., 1, 2½, and 5 per cent. solutions of gallotannic acid had quite killed all the microbes of the six species referred to.—J. L. B.

### *Yeast, The Bacteriology of.* (*Part I.*) R. F. Wood-Smith. J. Fed. Inst. Brewing, 1898, **4**, 115—120.

The investigation was carried out with the object of ascertaining the bacteria normally present in English top-fermentation yeast. To this end the author suspended a platinum wire loopful of yeast, collected 3 to 4 ins. below the surface of the rising mass in the squares, in sterilised distilled water. A portion of this was grown on nutrient agar-agar, a medium which is stated to afford a relatively greater nutrition to bacteria than to yeasts. The bacterial colonies were separated from the yeasts; these were cultivated on the various media usually employed for the purpose, and identified in the ordinary manner. The following bacteria were separated:—

1. After being pitched 4 to 7 hours: *Sarcina lutea*, *Proteus vulgaris*, *B. mesentericus*, *B. coli communis* (by carbol plate separation).
2. After 20 to 25 hours: *Sarcina lutea*, *B. coli communis*, *B. fluorescens*, and an unidentified sporogenous bacillus.
3. After 35 to 40 hours: *Sarcina lutea*, *Staphylococcus albus*, *B. fluorescens*, a sporogenous bacillus not identified.
4. Prior to letting down: *Sarcina lutea*, *Proteus vulgaris*, *B. subtilis*, *Staphylococcus cereus flavus*, *B. acidi lactis*, *B. fluorescens*, a white streptococcus, and *Staphylococcus albus*.—J. L. B.

### *Pure Yeast Colonies, The Separation of, by Plate Cultivation.* R. F. Wood-Smith. J. Fed. Inst. Brewing, 1898, **4**, 121—122.

The author states that the object of this method is to obviate the somewhat laborious process of separating individual yeast cells by Hansen's method, by removing the uncertainty which exists when recourse is had to plate cultivation in the ordinary way, for this purpose. The preliminary details are the same as in Hansen's method: suspensions of the yeast are prepared in sterile water, the number of cells present carefully determined by the hematimeter, and a more dilute suspension made, containing about 50 cells per c.c. Malt wort is now prepared of sp. gr. 1,100, and to it is added 1 per cent. of calcium chloride and 10 per cent. of gold leaf gelatin, the whole being rendered alkaline to the extent of 0·5 per cent. of sodium hydroxide. This mixture is heated until it is quite liquid, when it is shaken, sterilised, and poured into sterilised Petri dishes. After the medium has set, about 0·5 c.c. of the yeast suspension, containing approximately 50 cells, is brushed over the surface. Sub-cultures of yeast obtained by this method were treated according to Hansen's directions, and in all cases were found to be perfectly pure.

The 1 per cent. of calcium chloride was added, with the object of primarily strengthening the growth of the yeast colonies, since yeast cells readily absorb calcium salts, and by so doing appear to grow more actively and strongly in the first instance, though subsequently they become weakened. The excess of sodium hydroxide was added with a view of inhibiting the growth of many bacteria, which generally prefer a neutral or slightly acid medium. This excess in no way interferes with the growth of the saccharomycetes. The addition of calcium chloride and alkali is stated to be of great value in getting the colonies to grow perfectly discrete and with sufficient activity.—J. L. B.

### *Distillery Yeast, The Acidifying of.* P. Rosski. Zeits. für Spiritusind. 1898, 98—99.

The author had trouble with his fermentations, which he traced to a deficiency of acidity of his pitching yeast. He finds that the lactic acid ferment must not be exposed to too high a temperature; also that the more acid his pitching yeast contains, the cleaner are his fermentations. —A. L. S.

### *Distillery Yeast, The Acidifying of.* K. Morawski. Zeits. für Spiritusind. 1898, 99.

The author finds that the lactic acid ferment must not be exposed to too high a temperature, and that it must not be added before saccharification is complete. The addition of acid is not so important when the worts are prepared from good malt.—A. L. S.

### *Yeast, A New Nutrient Substance from.* Siebel. Wochenschr. für Brau. 1898, 162.

The author prepares a product from yeast having a composition closely resembling condensed milk, and which, he states, may be used in its place. Pressed washed brewery yeast is partly dried, and mixed with a fourth part of glucose and a little starch, when it forms a syrup. Mixed with water it forms a milky emulsion, which can be made to closely resemble separated milk.—A. L. S.

### *Yeast, Vitality of Dried.* H. Will. Zeits. fur d. ges. Brauw. **21**, [7], 75—77.

The conserves examined in 1896 (this Journal, 1896, 155, 453), *viz.*, asbestos, Nos. 7 and 8; charcoal, Nos. 9 and 10; and cellulose, No. 13 C., and found to still contain living yeast cells, were re-examined in 1897, 11 years and 2 months after putting up. Parallel cultures in sterile hopped beer wort, at 25° C., showed that the yeast had survived in No. 7 (wild yeasts only), No. 9 (culture yeast with traces of wild yeasts), and No. 10 (wild yeasts with a trace of culture yeast). Bacteria were present in all these, as well as in No. 8, but No. 13 C. was perfectly barren. Diplococci predominated, but No. 10 contained acetic bacteria, as in the previous year.

In the cultures 10 days old, living yeast cells were directly discernible in No. 7 alone; in the charcoal conserves only with difficulty and in traces.

Fermentation was brisk, and, in some cases, prolonged, notwithstanding the paucity of organisms.

The positive results of the examination harmonised both qualitatively and quantitatively with those obtained in the tenth year of storage, though the culture yeasts in the charcoal conserve No. 10 had undoubtedly diminished.

A number of pure cultures were prepared, and exhibited some interesting forms of wild yeasts, a few of which were characterised by their rapid tendency to form large agglomerations in wort cultures.

One of the culture yeasts prepared as a pure culture from charcoal conserve No. 9, was grown on a large scale, and tested in the brewery (Sedlmayer's), from which the original sample was obtained. It was there used for pitching nine times, yielding a beer of fine flavour, and "breaking" well in fermentation, reproduced well, and developed to large regular cells, thus demonstrating the capacity of yeast for retaining its good qualities after prolonged storage in the dried condition.—C. S.

### *Fermentation, The Influence of Pressure on.* R. E. Evans. J. Fed. Inst. Brewing, 1898, **4**, 249—258.

The author has carried out some experiments to determine the relative attenuation of a wort fermented under pressure, compared with one fermented in the normal way. He found that under conditions of pressure there was an increase in the attenuation, the amount of residual nitrogen being in each case practically the same. The gain in the attenuation implies an increased activity of the yeast. Experiments were tried to ascertain if any improvement could be effected in a bad yeast by a series of cultivations under pressure. Some sterilised wort was pitched with an impure yeast, and, after shaking, divided into three portions. The first was fermented under a pressure of 15 ins. of mercury; the second in a closed vessel with the tube dipping ½ in.

below the mercury; the third in a vessel plugged with cotton wool. When fermentation had proceeded, a microscopic examination showed that the yeast was good in the first flask, not so good in the second, and bad in the third. The fermented liquor was poured off and fresh sterilised wort added to the yeast remaining in the three flasks. When the second fermentation was finished, the treatment was repeated with a fresh quantity of wort. At the end of the third fermentation the contents of the first flask indicated that the yeast was becoming more active, and this, in the author's opinion, clearly shows the influence of air in promoting acidity and the growth of harmful organisms. The general appearance of the yeast throughout the successive cultivations was in favour of that from the wort fermented under 15-inch pressure, both for regular size of cells and the gradually diminishing quantity of wild yeasts and bacteria.—J. L. B.

*Fermentation with Top Yeast at Low Temperature.* C. Bleisch. Zeits. für d. ges. Brauwesen, **21**, [4], 39—40.

An attempt was made by the author, in a bottom-fermentation brewery in Belgium, to brew a top-fermentation beer, stable in bottle, from 9—10 per cent. wort—stronger wort being precluded on account of cost. The plan successfully adopted was as follows:—The ordinary triple thick-mash decoction method, with a longer exposure than usual to the higher ranges of temperature; pitching the yeast at an initial temperature of 9°—10° C. (maximum) in the tun, and an average room temperature of 6½°—7½° C. Fermentation set in after about 12 hours, and by the third day the curly head had subsided to a thick crust of yeast, which was skimmed off. In about 70—80 hours the beer exhibited an apparent attenuation of 43°, and was then racked into cask. During primary fermentation the temperature never exceeded 12½° C.; but cooling down to 5°—6° C. before racking, with the idea of restricting excessive secondary fermentation, was found unadvisable. The temperature of the storage cellar was 2°—2½° C., and here the beer clarified in about a fortnight, cooling down simultaneously to 4°—5° C., whilst the attenuation rose to 60°—63° C. After bunging down for a further three days, the beer was ripe and clear, but was nevertheless filtered to ensure perfect brightness.

A frequent change of yeast was found necessary, not on account of sluggish but of wild fermentation, the operation then proceeding more rapidly and further than usual, and the beer, though clarifying quickly, becoming afterwards hazy, the flavour being at the same time thin. An endeavour to use pure culture yeast failed by reason of the deficient reproduction, the yeast crop being too small for pitching. The beer was of refreshing and full flavour, sparkling in bottle and remaining bright therein for 10—11 days at room temperature. The flavour was almost undistinguishable from that of bottom-fermentation beer, and the same applied to a heavy 17 per cent. beer brewed in the above manner. The short time (4—5 weeks) in cask is a great advantage over the ordinary lager process, economising both time and money, and the system is peculiarly adapted for the production of cheap, light beers.—C. S.

*Attenuation [Beer], Note on.* G. Clissold. J. Fed. Inst. Brewing, 1898, **4**, 124—127.

The author recounts some difficulties he has met with in the attenuation of beers, and he considers that the final gravity depends far more on the species of the yeast and the conditions of its cultivation, in so far as they tend to change the species, than on the malt or its manipulation in the mash tun.—J. L. B.

*Must, Influence of the Constituents of, on Fermentation.* G. Gelmi. Staz. sperim. agr. ital. **30**, 302; Zeits. für d. ges. Brauw. **21**, [5], 57.

To different samples of a white and a red wine must, the author either added a mixture of citric acid, sugar, and peptone, or else neutralised the existing acid by calcium carbonate, and investigated the fermentation products subsequently obtained. The results showed that the addition of acid increased the amount of alcohol formed, and consequently stimulated the activity of the ferments; the use of calcium carbonate, on the other hand, reducing the formation of alcohol to below the normal level, as represented by the wine from untreated must.

In particular experiments the loss of carbon dioxide during the stages of fermentation was ascertained by weighing the vessels at intervals.—C. S.

*Wines, Use of Carbonic Acid to Preserve and Improve.* R. Pini. (From Gazz. vinic. ital.) Bull. de l'Assoc. des Chim. 1898, **15**, [7], 741—743.

Pasteur recommended the addition of carbonic acid to wine to preserve it from diseases. The author attributes the rapid alteration of light wines to the absence of carbonic acid lost by evaporation in the cask. According to Berthelot, carbonic acid preserves the aroma and bouquet of wines, whilst the air causes them to disappear. Mestre states that he has obtained good results with carbonic acid in "la casse." The author regards the addition of artificial carbonic acid as necessary, or at least advantageous, to wines subjected to pasteurisation, that is, which have been heated to 60° C., with the object of ageing them. By this heating the greater part of the carbonic acid contained in the wine escapes, and it becomes flat and insipid. But if the wine, as it runs from the pasteuriser, is put into a cask filled with carbonic acid, and a jet of the gas is introduced along with the wine, a double advantage is gained: the wine is placed in an antiseptic medium, or at least, out of contact of the air, and it acquires a pleasant fresh taste. Schaffer also recommends it for wines which, by transport, refilling, or filtering, have lost most of their carbonic acid, and therefore their freshness. Kulisch says that the addition of carbonic acid to Alsatian wines removes the very pronounced earthy taste they possess. It must also be of advantage for the wines of the South of Italy and Algeria, which have also this defect. The author states that he succeeded with carbonic acid in restoring the original quality to Rhine and Moselle wines, which had been altered by too long keeping. He attributes the freshness and piquancy of the Tuscany wines to the slow development of carbonic acid in the casks. Chianti is exported in closed bottles containing a thin layer of oil to prevent the loss of the natural piquancy and the delicate aroma peculiar to this wine. The author thinks that the same result would be obtained by charging the wine with carbonic acid, even in casks, and exporting it in them.—L. J. de W.

*White Wines, Production of, from Red Grapes.* V. Martinand. Comptes Rend. 1898, **126**, 656.

In this process (this Journal, 1898, 260)—

1. The grapes are extracted as completely as possible.
2. The must is cooled, in order to check fermentation.
3. Aerated until exactly decolorised.
4. Filtered, to separate the particles of precipitated colouring matter.
5. Put to ferment.

—H. T. P.

*Spirit from Cellulose and Wood.* E. Simonsen. Zeits. angew. Chem. 1898, [10], 219—228.

Parallel experiments were made on the lines indicated in this Journal, 1898, 365, so as to determine the respective influence of the four factors—pressure, amount of water, length of time, and acidity—in the inversion first of cellulose and then of wood.

*Influence of Pressure and Amount of Acid.*—With an acidity of 0·3 per cent. of sulphuric acid, the yield of sugar rose from 2·5 per cent., calculated on the dry cellulose with a pressure of 1·3 atmosphere, until the maximum was reached with a yield of 43·1 per cent. at a pressure of 9 atmospheres. With the acidity 0·45 per cent., the maximum yield (45 per cent.) was attained with the pressure at 8 atmospheres, whilst with an acidity of 0·6 per cent. the maximum was 43·9 per cent. at a pressure of 6 atmospheres. With a smaller amount of acid (0·15 per cent.) the greatest quantity of sugar (38·4 per cent.) was obtained with a pressure of 12 atmospheres. On increasing the amount of acid to 0·8, 1·2, and 1·6 per cent., the yields under a pressure of 2 atmospheres were greatly reduced, being only 10·4, 11·5, and 13·1 per cent. respectively. A similar series of experiments with oxalic acid, instead of sulphuric acid, showed under all conditions a smaller yield of sugar. From this the author concludes that the most favourable conditions for the inversion of 10 grms. of cellulose with 1,080 c.c. of dilute sulphuric acid are with a strength of 0·45 to 0·6 per cent. and the corresponding pressures of 8 or 6 atmospheres.

*Influence of Time.*—Experiments made under the most favourable conditions of pressure and acidity, as determined above, showed that the maximum yield was obtained in two hours, but that there was no considerable loss when the heating was continued for four hours, as in the previous determinations.

*Influence of the Quantity of Water and Strength of Acid.*—On treating 40 grms. of cellulose for two hours under a pressure of 8 atmospheres with 4·8 grms. of sulphuric acid in varying quantities of water (250 to 1,500 c.c.), the greatest yield of sugar (45·4 per cent.) was obtained when the volume of the liquid was 1,080 c.c., corresponding to an acidity of 0·5 per cent. By increasing the water to 1,500 c.c. the yield fell to 32·5 per cent. With the total volume of liquid 250 c.c., and the acidity 0·5 per cent., a yield of 42·7 per cent. was obtained after heating for 1½ hour, and by no variation of factors could this be increased. Thus, taking into account the advantage of having a more concentrated solution of sugar produced, the most favourable conditions for inversion appear to be: 1 part of cellulose to 6 parts of sulphuric acid (0·5 per cent.) heated for 1½ hour under a pressure of 10 atmospheres.

*Respective Influence of Temperature and Pressure.*—An experiment in which the chamber between the leaden vessel of the walls of the autoclave was partially filled with chloroform vapour, so that with a pressure of 10 atmospheres the temperature was only 138° C. instead of the usual 180·3 C., gave a greatly reduced yield, and from this the author infers that the high temperature is the principal cause of the inversion.

*The Residue after Inversion.*—This varied considerably in different determinations. In one experiment (yield of sugar 45·4 per cent.) it amounted to 14 per cent. of the cellulose; in another (sugar 25 per cent.) it was only 15 per cent. The residue in the former case, when dried and again inverted, yielded a further 26·8 per cent. of reducing sugar, which was possibly owing to a chemical change taking place during the drying.

*Substances contained in the Inverted Liquid.*—In addition to the reducing sugar, which the author found to consist principally, if not entirely, of glucose, there was always present another substance or substances in solution, the amount of which varied from 10 to 60 per cent. in different experiments, being in greater proportion according to the length of time of heating. It was of an acid nature, and was apparently derived directly from the cellulose without affecting the quantity of sugar.

*Fermentation of the Sugar.*—The amount of alcohol obtained was slightly lower than was to be expected from glucose. The spirit had a characteristic, partly empyreumatic odour, and showed all the ordinary reactions for alcohol.

*Inversion of Sawdust.*—In these experiments, 100 grms. of ordinary pine sawdust (15 per cent. of moisture) were heated with 500 c.c. of water (containing sulphuric acid), making up a total volume of 750 c.c.

*Influence of Strength of Acid.*—When heated for 1 hour under a pressure of 10 atmospheres, the best yields of sugar (19·2 to 19·6 per cent.) were obtained with an acidity of 0·58 to 1·0 per cent. Above 1 per cent., the quantity of sugar produced rapidly diminished, and with the acidity at 1·5 per cent. amounted to only 5 per cent.

*Influence of Pressure.*—The best results with acidity 0·5 per cent. and time 1 hour were obtained under a pressure of from 9 to 10 atmospheres. Under 8 atmospheres the yield was somewhat less (18·2 instead of 19·6 per cent.). Between 11 and 12 atmospheres it became considerably less.

*Influence of Time.*—The most favourable time of heating was found to be 15 minutes, when the other conditions were also most favourable. The yield of sugar was then 22·5 per cent., becoming 19·6 per cent. after 1 hour, and 16·1 per cent. after 2 hours. Hence, the best conditions for the inversion of wood are: Acidity, 0·5 per cent.; pressure, 9 atmospheres; ratio of wood to liquid, 1:5; and time, 15 minutes. In one experiment, in which the autoclave was heated by direct fire, a slightly higher yield (23·4 per cent.) of sugar (as determined by the reduction of Fehling's solution) was obtained—a result contrary to what might have been expected.

*Hydrochloric Acid as the Inverting Agent.*—This offered no advantage over sulphuric acid, as the yield of sugar was somewhat less, whilst the leaden wall of the autoclave was considerably attacked.

*Inversion of the Residue.*—The residue, which was of a brown colour, and which had still the form of sawdust, yielded a further 10 to 13 per cent. of sugar on inversion. This amount is too small to make inversion of the residue practicable on the large scale, but the author suggests that the latter might be used for the manufacture of oxalic acid.

*Acidity of the Inverted Liquid.*—It was found that the greater the quantity of acid used for the inversion, the more organic acid or acids was produced from the wood.

*Comparison of the Results of the Inversion of Cellulose and Wood.*—From the foregoing determinations it appears that wood is much more readily inverted than cellulose, if the reduction of Fehling's solution be taken as the criterion. The maximum yield, which in the case of cellulose is attained after about 1½ hours, is obtained with wood in about 15 minutes. On the other hand, wood produces only about 20 per cent. of sugar, whereas cellulose yields about 45 per cent. Whether the lignin contributes to the formation of the sugar still remains an unsettled question, but the author points out that the amount obtained from the wood corresponds to the quantity of cellulose present. Moreover, the residue in the author's experiments was certainly not cellulose, whilst the lignin reaction with phloroglucinol was as marked in the case of the residue as in the original sawdust.

*Fermentation Experiments.*—In five determinations in which the autoclave was heated by direct fire, the reducing substance could not be fermented—a result which the author considers was due either to no sugar being formed, or to the presence of some other substance which interfered with the fermentation. Further experiments were then carried out, in which the autoclave was heated indirectly in the usual way, with the result that fermentation readily occurred; but under no conditions was it found possible to ferment more than 64 per cent. of the sugar present. On concentrating the various weak distillates, a spirit of 50 per cent. strength, with a pleasant smell and taste, was obtained.

The author promises a further communication on the results of experiments on a manufacturing scale.—C. A. M.

*Seeds, Note on the Influence of very low Temperatures on the Germinative Power of.* H. T. Brown and F. Escombe. Proc. Royal Soc. 1897, **62**, 160.

*See under* XXIV., *page* 503.

*Hops, The Essential Oil of.* A. C. Chapman. J. Fed. Inst. of Brewing, 1898, **4**, 224.

*See under* XX., *page* 486.

*Brewing Sugars, On the Analysis of.* G. H. Morris. J. Fed. Inst. of Brewing, 1898, **4**, 162.

*See under* XXIII., *page* 496.

*Alcohol, The Determination of Small Quantities of.* F. J. Benedict and R. S. Norris. J. Amer. Chem. Soc. 1898, **20**, 293.

*See under* XXIII., *page* 500.

*Glycerin, Oxidation of, by the Bacteria of Sorbose.* G. Bertrand. Comptes Rend. 1898, **126**, [11], 842.

*See under* XXIV., *page* 503.

### PATENTS.

*Moulds, Utilising, for the Production of Alcohol.* A. Collette fils and A. Boidin, Séchin, France. Eng. Pat. 1155, Jan. 15, 1897.

The mould *Amylomyces Rouxii*, when grown in one hectolitre of the residuary liquors of yeast factories, will produce 1 litre of alcohol. Other similar residues may be utilised for the production of alcohol in this way, and the above-mentioned mould may be replaced by the *chlamydomucor* of Java or the *aspergillus orizæ* of Japan.

These moulds may also be substituted for yeast in bread making.—A. L. S.

*Spirit, Improved Process for the Purification of.* L. Maiche, Paris. Eng. Pat. 4089, Feb. 15, 1897.

The strong spirit to be purified is diluted with 5—10 times its volume of water, and subjected to distillation in a vacuum at a temperature of about 26° C. The impurities, it is stated, distil over, leaving the pure spirit behind.—A. L. S.

*Alcoholic Liquids, A New Method of Maturing.* J. G. Hathaway, London. Eng. Pat. 8223, March 30, 1897.

Air, and air rich in oxygen is passed through the spirit to be matured, which is heated to 65°—95° F. The oxygenated air is produced by passing air over heated potassium permanganate.—A. L. S.

*Cider, Apparatus for the Manufacture of.* A. C. Briet, Paris. Eng. Pat. 11,600, May 10, 1897.

Nine diffusion vats are mounted on trucks running on circular rails, so that each vat can be brought successively under an apple-cutting machine, to receive a charge of apple cuttings. Each vat is divided by horizontal partitions, between which the apple cuttings are held, and by vertical partitions, forming compartments up and down which the juice circulates. The vats communicate by pipes furnished with stop-cocks. The liquid is made to circulate consecutively through all the diffusing vats of the battery, and is finally collected in a vat.

The apple-cutting machine consists of a drum, at the lower end of which is a revolving cutter consisting of a disc having openings on which are mounted frames containing the cutting blades.—A. L. S.

*Alcohol, The Manufacture of, by Saccharification and Fermentation by Moulds.* A. Collette fils and A. Boidin, Séchin, France. Eng. Pat. 19,858, Aug. 28, 1897.

The starchy raw material is well boiled, and the starch liquefied by a little malt, acid, or mould; it is then well boiled, and cooled, without allowing any air that has not been sterilised to come in contact with it. A pure culture of *Amylomyces Rouxii, Aspergillus orizæ*, or other mould, is then added, and by continual aeration and agitation, the mould is kept submerged. Saccharification and fermentation of the starch take place, the latter being hastened by the addition of a little yeast. This process is stated to give much better yields of alcohol than the one usually practised.
—A. L. S.

*Alcoholic Liquors, Improved Process and Apparatus for Accelerating the Ageing of.* R. M. de Tangry, Antwerp. Eng. Pat. 30,658, Dec. 28, 1897.

The spirit to be matured is passed through a series of vessels, and during its passage is gradually raised from the ordinary temperature to 55° C., and then gradually reduced to the ordinary temperature. In the upper parts of these vessels there are condensers or tubes of a pear-shaped section, with a gutter beneath each tube to collect the liquid condensing on them.

The spirit which has passed through the apparatus is collected in a vat, which has a partition nearly extending to the bottom, but leaving sufficient space for the spirit to flow readily under. By this arrangement the spirit deposits any sediment at the bottom of the collecting vat, and flows from the further side of the vat bright. The impurities of the spirit are thus separated, partly by condensation on the tubes of the condenser, and partly as a deposit at the bottom of the collecting vat.—A. L. S.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (A.)—FOODS.

*Pentosans, The Solubility of, in the Reagents employed in the Estimation of Starch.* W. H. Krug and H. W. Wiley. J. Amer. Chem. Soc. 1898, **20**, 266.

*See under* XXIII., *page* 498.

*Chocolate, Detection of Added Starch in.* Posetto. Giorn. di Farm. di Chim., &c., Jan. 1898; through Pharm. Centralhalle, 1898, **39**, [9], 152.

*See under* XXIII., *page* 499.

*Flour, Detection of Sawdust or Wood-Meal in.* G. A. Le Roy. Rev. de Chim. Industr. 1898, **9**, [100], 114.

*See under* XXIII., *page* 493.

*Moulds, Utilising for the Production of Alcohol.* A. Collette fils and A. Boidin, Séchin, France. Eng. Pat. 1155, Jan. 15, 1897.

*See under* XVII., *see adjoining column.*

### PATENTS.

*Albuminous Products, Manufacture of Soluble, from the Seeds of Plants.* O. Imray, London. From Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 11,513, May 8, 1897.

Oil cakes are ground and boiled with ether. The extracted mass is treated with just sufficient soda to dissolve the albuminous substances, heated to 80° C., and stirred for several hours. It is then filtered and evaporated to dryness *in vacuo*. The residue is the soluble sodium salt of the albuminoids, and is of an agreeable taste and odour.
—A. L. S.

*Flour and Meal, Impts. in the Manufacture of.* W. Jago, Hove, Sussex. Eng. Pat. 12,567, May 21, 1897.

The bran and flour of wheat are separated in the usual manner. The bran from 100 lb. of wheat is treated with a wort prepared from 25 lb. of malt at 160° F. for one hour, when the mixture is boiled and the water evaporated in a suitable apparatus. The dry mass is ground and mixed with the flour.—A. L. S.

### (B.)—SANITATION; WATER PURIFICATION.

*Sterilisation of Liquids by Filtration.* J. Hausser. Comptes Rend. 1898, **126**, [11], 844—846.

The Chamberland and Garros filters, as is well known, work very slowly, and are difficult to clean. In order to form a filter free from these objections, and which will sterilise most liquids, and especially water and beer, the author proceeds in the following way:—

Infusorial earth, otherwise known as *farine fossile* and *Kieselguhr*, is sifted to remove sand and other grosser impurities. It is then ignited at a temperature of about 800°—1,000° C. After cooling, it is pulverised, and, if it be necessary to have an impalpable powder, is treated by elutriation. This latter operation, however, may in general be omitted, except in certain special cases. If wine is to be filtered, it is necessary, on account of the acidity of this liquid, to complete the purification by washing with a mineral or organic acid. The substance thus obtained is insoluble in most liquids, and does not communicate to them any taste.

To prepare the filtering bed, the powder is suspended in the liquid to be filtered, and run on to any suitable filter support. The liquid flows through, leaving a perfectly regular compact layer, which is capable of retaining the smallest particles and micro-organisms. Filtering beds prepared in this way, filter, other things being equal, from 15 to 20 times as fast as the Chamberland filter. Absence of rigidity is, if anything, an advantage, since it facilitates renewal and cleaning. When the filtering power seems to be exhausted, the layer is removed by a current of water and replaced by a fresh bed. When dirty, the original properties of the powder may be restored by washing with dilute acid, drying and re-igniting. Since the first portions of the filtered liquid are not sterile, they must of course, be passed through the filter once more, and if continuous sterilisation be desired, it is important that the filtration should never be stopped, except when absolutely necessary. Experiments have shown that a depth of filtering bed amounting to four- or five-tenths of a millimetre is sufficient for sterilisation.—J. S.

*Disinfection, New Method of.* R. Walther and A. Schlossmann. J. für Prakt. Chem. **57**, [3 and 4], 173—197.

By means of a specially constructed apparatus, a mixture of formaldehyde and glycerin is sprayed into a room which is to be disinfected, until a thick fog results; about 4 lb. of the mixture are needed per 1,000 cb. ft. The room need not be hermetically closed during the operation, as the ordinary circulation of air assists in spreading the disinfectant and in enabling it to reach remote corners. Three hours' exposure was found sufficient to kill all germs in the rooms experimented on, though the test objects were purposely chosen of the most refractory nature. For example:—Pieces of linen thickly coated with a paste of white of egg and garden soil, dried in an incubator; layers of soil 3 or 4 mm. thick with potato skins under and above them; potato skins alone: these were placed, open and covered, at various heights in the room, in recesses in the wall, on the floor, under pieces of furniture, in tall glass cylinders, or in shorter cylinders under a layer of wadding, in the pockets of thick winter clothing; fæces were also sterilised by this exposure. Live guinea pigs and rabbits were also found to be freed from bacteria in their skins, their bedding straw, and their excrement. The authors attribute the very advantageous effect of adding glycerin to the formaldehyde, to its hygroscopic character and its power of adhering to and penetrating most of the ordinary porous materials found about a household. They anticipate that it may be found possible to diminish still further the necessary duration of the period of disinfection, and that their method will become a much more powerful agent than any yet known against the spread of infectious diseases not only in man but in the lower animals.—J. T. D.

*Carbon Monoxide, Estimation of, in Large Quantities of Air.* A. Gautier. Comptes Rend. 1895, **126**, 793.

*See under* XXIII., *page* 490.

*Carbon Monoxide, Estimation of, in Air.* A Gautier. Comptes Rend. 1898, **126**, [13], 931.

*See under* XXIII., *page* 490.

*Carbon Monoxide, Detection of, in Air by Means of Palladium Chloride; and Transformation of, into Carbon Dioxide at the Ordinary Temperature.* Potain and Drouin. Comptes Rend. 1898, **126**, [13], 938.

*See under* XXIII., *page* 490.

## PATENTS.

*Filter Presses, Impts. in, and in Apparatus for Opening and Closing Same.* G. M. Park, Blackburn. Eng. Pat. 6180, March 12, 1897.

*See under* I., *page* 441.

*Town Sewerage, A New or Improved Process and Apparatus for Purifying and Utilising.* G. A. de Katow, London. From Rensch and Co., Wiesbaden, Germany. Eng. Pat. 8281, March 31, 1897.

A SERIES of mechanical straining appliances are fixed in the main sewer, capable of removing from the sewage, as it flows, suspended matter of an increasing degree of fineness. The coarser matters are first separated by a fixed grating, and are swept by a revolving rake on to a travelling band. Beyond this, a movable bottom, slightly below the bottom level of the sewer proper, is provided, on which sand deposits, and is automatically discharged at intervals into a catch pit, from which it is raised by a dredger. Further along, a second, finer screen with revolving rake, removes the coarser matters still remaining in suspension; and finally, the liquid flows through two screens which remove the finest particles; these screens are fixed in grooves, so that when clogged they can be raised, one at a time, for cleaning purposes. The clarified liquid may flow direct into a stream, or it may be treated with suitable chemicals and passed through clarifying tanks. The solid refuse screened from the sewage, together with the chemical precipitate, if any, is mixed, pressed into bricks, and sold for manure.—L. A.

## (*C.*)—DISINFECTANTS.

## PATENTS.

*Disinfecting and Antiseptic Block, A New and Improved.* J. L. Mortimer and J. H. Williams, Liverpool. Eng. Pat. 6823, March 16, 1897.

POTASSIUM or sodium permanganate is heated to 300° F. with about one-third of its weight of either naphthalene, camphor, or both, and the thoroughly incorporated mixture is moulded into blocks. Instead of camphor and naphthalene, soap in a liquid state may be used, the inventors' object being to coat the crystals or particles of the permanganate with a slightly soluble or volatile substance which, by dissolving or evaporating, gradually exposes the permanganate to the solvent action of water passing through the trap, drain, sink, &c., in which the block may be placed. L. A.

*Drying and Sterilising, Impts. in or relating to.* H. Higgins, Cambridge. Eng. Pat. 7664, March 24, 1897.

A SERIES of drying chambers are each provided with a superheater of any convenient form, such as a coil of piping, and these are arranged in a series so that steam passes through each chamber in succession, being superheated in its passage between each chamber. Any chamber can be cut out by diverting the steam through a bye-pass pipe, and the temperature of each chamber is controlled by adjusting the superheating of the steam. The apparatus is stated to be applicable for sterilising clothes in workhouses and hospitals.—E. G. C.

*Sterilising Apparatus, Impts. in and relating to.* H. Higgins, Cambridge. Eng. Pats. 7667 and 7668, March 24, 1897.

THE apparatus described in both specifications consists of a steam generator, a pipe in which the steam is superheated, and a sterilising chamber, all contained within an outer metal casing, which is lagged and can be moved about on casters.

In the first specification, the steam is generated and superheated in a coil of pipe supplied with water from a tank fixed outside the casing. The superheated steam passes into the lower part of the sterilising chamber and escapes at the top, at substantially atmospheric pressure, through a tube which conveys it into the tank, where it partly condenses and heats the water. The lid of the chamber fits tightly, but is not fastened down, and carries a tube closed at the lower end to hold a thermometer.

In the second specification, the steam generator is an upright boiler placed within the casing, the steam is superheated in a coil of pipe which passes from the steam space of this boiler to the sterilising chamber, and the steam is condensed in another pipe which is coiled around the outside of the casing and returns the condensed water to the boiler. In this case, the lid is fastened down by thumb screws, and carries a safety valve and pressure gauge as well as a tube for the thermometer.—L. A.

*Antiseptic Soaps containing Mercury Albuminates, Impts. in the Manufacture of.* R. Ehrhardt, Augsburg, Bavaria. Eng. Pat. 2407, Jan. 29, 1898.

*See under* XII., *page* 469.

## XIX.—PAPER, PASTEBOARD, Etc.

*Sulphur: Amount required by the Sulphite Treatment of Wood.* Papier Zeit. 1898, **23**, [18], 646.

In the annual report of the Josefihütte pulp mills, in Bohemia, it is stated that the average proportion of sulphur used during 1897 amounted to 10·9 kilos. per 100 kilos. of air-dry cellulose (12 per cent. of moisture).—S. P. E.

*Waste Sulphite Lye and Common Salt.* Papier Zeit. 1898, **23**, [26], 951.

As in most countries salt is taxed for manufacturing purposes, whilst farmers who use it as a cattle food are exempt from the tax, a method of rendering the common salt unfit for manufacturing purposes, but not unfit for cattle food, is very much required.

An addition of 5 to 10 per cent. of waste sulphite lye is found to answer this purpose, being very difficult to remove from the salt, and not rendering it distasteful to cattle.
—S. P. E.

*Paper-making, Leather Cuttings for.* Papier Zeit. 1898, **23**, [22], 802.

A correspondent of the Papier Zeitung puts forth the opinion that leather cuttings should be very useful for paper-making, on account of their low price and the long fibres.

Leather cuttings have, however, often been tried without success, on account of the leather containing no felting fibres. The cuttings could only be used as a filling for the paper, and might be useful for thick paper or boards; but thin paper could not be made from the leather, as stronger felting fibres are required.—S. P. E.

*Peat for Paper Pulp.* Papier Zeit. 1898, **23**, [33], 1209.

C. A. Zschörner, of Vienna, has obtained an Austrian patent for a process of converting peat turf into a pulp suitable for making paper, and also for rendering the fibres capable of being spun. According to the author, previous attempts to clean and soften the fibres have failed, owing to the severe action of the chemicals, heat, and pressure employed.

By the present invention the peat, after being washed, is treated under a pressure of one or more atmospheres with a 2 per cent. caustic soda solution at a temperature of 4°—25° C. After 10 to 14 hours a dark brown lye is formed, which is drawn off, and the peat well washed with water, and subsequently treated under pressure with a 1 per cent. solution of alkali. The fibres are preferably bleached with a solution of calcium or sodium hypochlorite of about 2° B. at the ordinary temperature, but under pressure. It is stated that a large company has been formed in Vienna to manufacture peat paper, peat boards, and spun peat fibres, and that patents have been applied for in all countries of Europe and America.—S. P. E.

*Straw Boards.* Papier Zeit. 1898, **23**, [24], 379.

In many strawboard mills much mischief is done in the boiling. To obtain good boards, care should be taken that wheat and rye straw are not boiled together. Rye straw is sufficiently soft after about 3½ to 4 hours' boiling, whilst wheat straw requires quite 5 hours. 85 kilos. of lime are sufficient for 1,000 kilos. of rye straw, whilst 100 kilos. of lime and longer boiling are required for wheat straw.

The steam pressure in the boilers ought not to exceed 5 atmospheres, a higher pressure causing the straw to burn, which shows especially in such boards as have to be covered with thin paper.

It is therefore much better to keep the two kinds of straw as separate as possible.—S. P. E.

### PATENTS.

*Manufacture of Glutinous Substances or Sizes [Paper], Process for the.* A. Mitscherlich, Freiburg, Baden, Germany. Eng. Pat. 8042, March 29, 1897.

The invention relates to a process for the manufacture of size from horny matters, keratin masses, or similar bodies, and for rendering it particularly suited for sizing paper by means of rosin. Horn pieces or keratin bodies are suitably dissolved by means of dilute alkaline solutions; to this solution at 100° C. the rosin is added in such quantities as will dissolve readily, forming a transparent liquid, which on cooling sets to a tough jelly.—J. G. P.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Iodine, Rapid Process for the Solution of, in Oils.* A. Schmitt. L'Union pharmaceut. 1898, **39**, 99; Chem. Zeit. Rep. 1898, **22**, [29], 87.

As a means of saving time in the solution of iodine in oil, the author recommends the following method, which depends on the employment of glycerin and an iodide as a preliminary solvent. For example, 1 grm. of "iodine bisublimat.," 0·25 grm. of sodium iodide, and 1—2 drops of pure glycerin are triturated very intimately till the iodine has completely dissolved, and then the requisite quantity of oil is added. This process may be carried out in a few minutes; the solution of the iodine in the oil is complete and the liquid becomes quite clear, and is gradually decolorised.—A. S.

*Absolute Alcohol, Preparation of, by means of Calcium Carbide.* E. Ostermayer. Pharm. Zeit. **43**, 99; Chem. Centr. 1898, **1**, [11], 658.

The author succeeded, it is stated, in preparing "100 per cent." alcohol from ordinary spirit on the small scale by means of the method proposed by P. Yvon (this Journal, 1898, 180), but the product had an unpleasant odour and taste, due to the presence of organic sulphur compounds.
—A. S.

*"Acetone Oil" and Ketones, Preparation of [from Wool-Washings].* A. and P. Buisine. Rev. de Chim. Industr. 1898, **9**, [100], 112—114.

The authors suggest a process for the production of "acetone oil" by making use of the volatile fatty acids contained in the liquid obtained by washing wool. This liquid is allowed to ferment, producing fatty acids, ammonium carbonate, &c. After heating to drive off the ammonium carbonate, the fatty acids are liberated by the addition of sulphuric acid, and the volatile acids separated by distillation in a current of steam. Calcium carbonate is then added to the distillate, and the liquid is evaporated to dryness. The dry calcium salts of the volatile fatty acids thus obtained are distilled in the usual way, the yield being a mixture of ketones.—W. P. S.

*Quinine Glycerophosphate.* Moncour. J. Pharm. Chim. 1898, **7**, [8], 384—389.

Quinine glycerophosphate is obtained by neutralising the free acid with the calculated quantity of quinine, or by double decomposition between quinine sulphate and calcium glycerophosphate. The compound forms slender white needles melting at 154° C., less bitter than quinine sulphate and slightly soluble in hot and cold water. At 15° C., alcohol of 95 per cent. strength dissolves 3 per cent., but at 100° C. the salt is soluble in all proportions. The addition of a very small quantity of acid (citric or hydrochloric) largely increases the solubility in water. Analysis shows the compound to be the basic salt—

$$C_3H_7O_3.PO.(OH.C_{20}H_{24}N_2O_2)_2 + 4H_2O.$$

Quinine glycerophosphate should not give the tests for free phosphoric acid with ammonium molybdate or magnesia mixture, it should not give up glycerin when washed with water, and should show the correct melting point. The quinine-content is obtained by dissolving in dilute acid, precipitating with alkali, washing, and weighing; phosphoric acid is estimated in the residue after calcination with potassium nitrate and carbonate.—A. C. W.

### *Morphine-Quinoline Ester.* P. Cohn. Kaiserliche Akad. der Wissenschaft. in Wien, 1898, (7), 51.

Dichlor-quinoline acts on morphine with the liberation of hydrochloric acid, whilst codeine remains unchanged even after prolonged heating—a circumstance which explains the different behaviour of the hydroxyl groups of the former alkaloid. The author draws the conclusion that the hydrogen which appears to be substituted in codeine by the methyl group, has entered into reaction. The product obtained by the action of dichloroquinoline on morphine forms small, pointed, white prisms which melt at 158° C. Mineral acids combine with the base and form bitter salts. The new base does not give the characteristic reactions of morphine with ferric chloride, iodic acid, or with other reagents. Of the derivatives, the acid salts are mostly amorphous and soluble in water, but the normal salts crystallise readily in well-defined form. The normal sulphate shows some similarity to normal quinine sulphate. It consists of fine shining needles which dissolve in water on the addition of a few drops of hydrochloric acid. This solution is a powerful tetanic poison. Doses of 0·001 grm. have a lethal effect on cold-blooded and 0·2 grm. on warm-blooded animals. It produces complete paralysis, and arrests respiration while the heart continues to beat, and sensibility is retained. The platinum double salt, the chromate, tartrate, and picrate, are described. The ester gives substitution products with bromine, similar to those obtained with morphine, and also forms a periodide with iodine.

—J. O. B.

### *The Rhodinol Question.* J. Bertram and E. Gildemeister. Ber. 1898, **31**, [6], 749.

A reply to Poleck (this Journal, 1898, 267). The differences between the statements of Eckart and other observers led the authors in 1894 to investigate rose oil. They found that Eckart's "rhodinol" was not a pure substance; the primary alcohol isolated from it was identical with the geraniol, $C_{10}H_{18}O$, discovered by Jacobsen in 1870 in Turkish geranium oil, and thus could receive no other name than geraniol.—A. C. W.

### *Caffeine Sodium Benzoate.* F. Rolla. Boll. Chim. Farm. **37**, 100—101; Chem. Centr. 1898, **1**, [11], 678.

It is stated that on mixing together solutions of caffeine and sodium benzoate, caffeine sodium benzoate is sometimes precipitated as soon as the mixture becomes cold. The author observed that this condition of uncertainty will always be present if the sodium benzoate contain free sodium carbonate, and he therefore proposes, either that the benzoate employed be first purified, or that a few centigrms. of free benzoic acid be added to the hot solution of the benzoate and caffeine.—A. S.

### *Hops, The Essential Oil of.* A. C. Chapman. J. Fed. Inst. Brewing, 1898, **4**, 224—233.

Following a brief account of his own researches (this Journal, 1893, 783; 1895, 63) and those of some other workers on the essential oil of hops, the author describes some experiments which he has made with the object of studying the oxidation of this oil. When exposed to the air, it resinifies, and absorbs oxygen, but forms no valeric acid. In an alkaline solution of potassium permanganate it readily oxidises, producing, amongst other substances, an oily acid, which is not, however, valeric acid. The same product was also obtained by oxidation with chromic acid.

The essential oil isolated from old hops was oxidised with alkaline potassium permanganate, and valeric acid identified in the products. This acid, therefore, arises from the oxidation of one of the hop constituents (other than the oil); probably it is the so-called "soft resin." The author has also found (Brewer's Journal, 1896, 238) that neither the essential oil of hops or any one of its constituents possesses antiseptic properties. The preservative power of hops, as was originally suggested by Hayduck, is due to certain of the resins. (See this Journal, 1898, 171.)

—J. L. B.

### *Pilocarpidine.* J. Herzig and H. Meyer. Monatsh. für Chem. 1898, **19**, [1], 56—59.

The authors have found that pilocarpine contains one methyl group united to nitrogen, and pilocarpidine no methyl group so united. Petit and Polonowsky (this Journal, 1897, 461, 555, 756), on the contrary, state that the two alkaloids are isomeric, pilocarpine being tranformed into pilocarpidine on heating, without change of weight. Merck's Report for 1897 states that the pilocarpidine from jaborandi is not identical with the substance obtained from pilocarpine. The hydrochloride of the base obtained by heating pilocarpine contains one methyl group; the formula $C_{11}H_{16}N_2O_2.HCl.AuCl_3$ was found for the gold double salt. The double chloride given by pilocarpidine from Merck has the formula $C_{10}H_{14}N_2O_2.HCl.AuCl_3$. Thus pilocarpidine is not identical with the isomer of pilocarpine.—A. C. W.

### *Scopolamine and Scopoline.* W. Luboldt. Archiv. der Pharm. 1898, **236**, [1], 11—47.

*Preparation of Free Scopolamine.*—The alkaloid was liberated by treating an aqueous solution of the recrystallised hydrobromide with potassium hydroxide solution, washing out with chloroform, evaporating the solvent, and treating the residual base with ether. The ether-extracted base was then treated with the theoretical amount of water and a little alcohol, to prevent precipitation; from this medium it crystallised in colourless, clear, rhombohedric crystals containing one molecule of water and corresponding to the formula $C_{17}H_{21}NO_4H_2O$. The melting point was 56° C., and the solubility in water, at 15° C., 1·4946 in 100. The specific rotation was $\alpha_D = -1°·37$. The action of strong sulphuric acid upon the base was found to result in the removal of a molecule of water from two molecules of scopolamine; thus, $2C_{17}H_{21}NO_4 - H_2O = C_{34}H_{40}N_2O_7$.

*Scopoline* was obtained by the prolonged boiling of scopolamine hydrobromide with barium hydroxide solution; the base was extracted by chloroform and crystallised from petroleum spirit or ether, from which it separated in long transparent needles, m.p. 109° C. The hydrochloride, $C_8H_{13}NO_2HCl + H_2O$, occurred as crystalline tablets; the hydrobromide as needles; the hydriodide in columnar crystals; the sulphate, $(C_8H_{13}NO_2)_2H_2SO_4$, as a hygroscopic crystalline powder. By oxidation with a permanganate, scopoline gave a base *Scopoliginine*, which contained a $CH_2$ group less than scopoline, and corresponded to the formula $C_7H_{11}NO_2$. Scopoliginine melted at 205° to 206° C., but sublimed above 100° C. It occurred in prisms. The salts of this base were prepared and examined, and, by treatment with methyl iodide, scopoliginine was reconverted into scopoline.

The analogy between scopoline and tropine shown in the products of oxidation was further visible in the results of methylation and decomposition. A long series of methyl derivatives and their salts were obtained and examined. Scopoleines, resembling in character the tropeines of Ladenburg, were also prepared by similar methods. Furthermore, it is pointed out that the empirical formula of scopoline, $C_8H_{13}NO_2$ compared with that of tropine, $C_8H_{15}NO$, shows that, in the former, two atoms of hydrogen have been replaced by one atom of oxygen, probably as a ketone group.—J. O. B.

### *Glycerophosphates, Critical Study of the Method for the Estimation of the.* H. Imbert and J. Pagès. J. Pharm. Chim. 1898, **7**, [8], 378. (See also this Journal, 1898, 66, 73, 78, and 266.)

*See under* XXIII., *page* 500.

### *Mustard Oil, The Estimation of.* E. Haselhoff. Zeits. Untersuch. der Nahr. und Genussm. 1898, **4**, 235.

*See under* XXIII., *page* 500.

### *Cocaine Hydrochloride, Maclagan's Ammonia Test for the Purity of.* C. F. Boehringer and Soehne. Pharm. Centralhalle, **39**, [9], 141.

*See under* XXIII., *page* 494.

*Cantharides, Assay of.* H. G. Greenish and H. Wilson. Pharm. J. 1898, **60**, 256.

*See under* XXIII., *page* 501.

*Goupia Tomentosa, The Volatile Constituents of the Wood of.* W. R. Dunstan and T. A. Henry. J. Chem. Soc. April 1898, 226.

*See under* XXIV., *page* 504.

## PATENTS.

*New Chemical Compound [Condensation Product of Gallic and Salicylic Acids] suitable for Medicinal Purposes, Manufacture or Production of.* Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 9898, April 20, 1897.

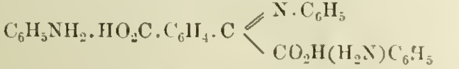
The process is claimed for producing a condensation product of gallic and salicylic acids, which consists in acting on an equimolecular mixture of the acids or their salts with phosphorus oxychloride or other dehydrating agent. The product of the reaction, which is probably a mixed anhydride, $OH.C_6H_4.CO.O.CO.C_6H_2(OH)_3$, is claimed as a new product, and its use is also claimed for medicinal purposes.—A. C. W.

*Aldehydobenzoic Acid and Intermediate Products, Impts. in the Manufacture of.* G. B. Ellis, London. From La Société Chimique des Usines du Rhône, anc. G. P. Monnet et Cartier, Lyons, France. Eng. Pat. 10,183, April 23, 1897.

On oxidising naphthalene or naphthalene derivatives with permanganate, *o*-oxalylbenzoic acid—

$$(1.2)HO_2C.C_6H_4.COCO_2H$$

is obtained. When this substance is treated with aniline in an aqueous solution, it forms a compound having probably the following formula:—



$$C_6H_5NH_2.HO_2C.C_6H_4.C\begin{cases} N.C_6H_5 \\ CO_2H(H_2N)C_6H_5 \end{cases}$$

On boiling this substance in a neutral solvent, such as toluene or xylene, it is converted into anilidobenzylidene-*o*-carboxylic acid, melting at 174° C., which, on hydrolysis, is converted into aldehydobenzoic acid. In practice, the solution obtained by oxidising naphthalene or naphthalene derivatives with permanganate, after filtering off the manganese dioxide, is acidulated with acetic acid. To this is then added a quantity of aniline, such that three molecular proportions are present to every one of the *o*-oxalylbenzoic acid. The mixture is warmed for half an hour to 80°—100° C., and, on cooling, the new aniline salt separates out in yellowish leaflets, any phthalic acid formed in the first part of the operation remaining in solution. The aniline salt thus obtained is suspended in five times its weight of xylene, or 10 times its weight of toluene, and cohobated for half an hour. Carbonic acid is given off, and, on cooling, anilidobenzylidene-*o*-carboxylic acid—

$$C_6H_5N:CH.C_6H_4.CO_2H$$

crystallises out. On warming this compound with 10 times its weight of 10 per cent. hydrochloric or sulphuric acid on the water-bath, solution takes place, and, after filtering, the filtrate is extracted with ether. On removing the latter, aldehydo-*o*-benzoic acid, melting at 98° C., is obtained.
—T. A. L.

*Sterile Iodoform, Production of.* Chemische Fabrik vorm. E. Schering, Berlin, Germany. Eng. Pat. 11,226, May 5, 1897.

The claim is for a mixture of iodoform with a solid polymeride of formaldehyde. Iodoform thus treated, containing only 0·5—0·025 per cent. of formaldehyde, remains sterile after several days' exposure to the air, and has a strong smell of the aldehyde. Under polymers of formaldehyde are included trioxymethylene, α-trioxymethylene, and a solid polymer prepared by mixing 50 parts of 40 per cent. formaldehyde solution with 25 parts of concentrated sulphuric acid, and cooling. After several days' standing the precipitated white powder is washed and dried. This substance is insoluble in ammonia, and melts at 162° C. in a closed tube.—A. C. W.

*Salicylo-acetic Acid, Process for the Manufacture of.* J. D. Riedel, Berlin, Germany. Eng. Pat. 11,596, May 10, 1897.

The processes for the manufacture of salicylo-acetic acid are claimed, which consist in the employment of the reaction of monochloracetates upon salicyl nitrile or amide, and saponification of the product.—A. C. W.

*m-Hydroxy-p-amidobenzoic Acid, Manufacture of Esters of.* Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 12,179, May 17, 1897.

The processes for the manufacture of esters of *m*-hydroxy-*p*-amidobenzoic acid, which consist in reducing the hydroxynitrobenzoic acid with tin and hydrochloric acid, and treatment of the resulting amido acid in alcoholic solution with a mineral acid, or in esterifying and then reducing, are claimed, as also the ethyl and methyl esters so produced.—A. C. W.

*Bismuth Oxyiodide-pyrogallate [Antiseptic], Manufacture of.* H. Imray. From F. Hoffmann—La Roche and Co., Basle, Switzerland. Eng. Pat, 13,993, June 8, 1897.

The process claimed consists in digesting molecular quantities of bismuth oxyiodide with pyrogallol, or in precipitating a solution of pyrogallol and an iodide with an acetic acid solution of bismuth nitrate. The product (bismuth-oxyiodide pyrogallate) is claimed as a new article of manufacture.—A. C. W.

*Camphor [Substitute], Further Impts. in the Manufacture of Substitutes for, and Products made therefrom.* J. E. T. Woods, Brixton. Eng. Pat. 14,328, June 12, 1897.

The patentee claims a method for the purification of turpentine hydrochloride, which consists in neutralising the crude product, filtering, and distilling by superheated steam in the presence of oxidising agents such as permanganate of soda or potash. The second claim is for the crystallisation of the melted purified hydrochloride under "the coercive action of excited electro-magnets, by which the axis of crystallisation can be changed at will, and the material made to resemble refined natural camphor." The purification of the residual oil obtained in the first process by neutralisation and steam distillation in the presence of oxidising agents is claimed, together with the conversion of this "terebene ether into modified substances of a fragrant aroma" by steam distillation in the presence of nitrous or nitric oxide. The final claim is for combinations of the camphor substitute, or the purified "terebene ether," with melted soap, "useful as a disinfecting detergent."—A. C. W.

*Sulphonic Acids of Guaiacol and of Creosote, and of Metallic Salts of these Sulphonic Acids; Manufacture of.* H. Imray, London. From F. Hoffmann—La Roche and Co., Basle, Switzerland. Eng. Pat. 14,376, June 14, 1897.

According to the quantity of sulphuric acid employed, and the temperature at which the sulphonation is performed, it is possible to obtain from guaiacol or from creosote one or other sulphonic acid. The methods employed are as follows:—Molecular proportions of fused pure guaiacol and concentrated sulphuric acid, or one part of guaiacol and 1—3 parts of concentrated sulphuric acid, are mixed and stirred at a temperature not exceeding 70°—80° C. When sulphonated, the melt is poured into water, treated with barium carbonate, filtered from barium sulphate, and the filtrate treated with dilute sulphuric acid. The guaiacol acid I. thus obtained forms on evaporation of its aqueous solution, a thick coloured syrup, which cannot be distilled without decomposition, and does not crystallise from any solvents. Its salts are prepared by saturating the acid with the mono- or bicarbonates, and in dilute aqueous solution give a violet-blue coloration with ferric chloride, changing to bright yellow on addition of ammonia. Guaiacol sulphonic acid II. is formed by employing the same proportions of guaiacol and sulphuric acid, but heating to 150°—

160 C., the acid and salts being obtained in a similar manner to that given above. The salts of this isomeride crystallise from water, in which they are easily soluble, and give a greenish-blue coloration with ferric chloride, changing to a brilliant Bordeaux-red on addition of ammonia. The salts of both acids are colourless and nearly tasteless, and are preferable to guaiacol in that they have no caustic action on the mucous membrane. By treating creosote in the same way, two acids may be obtained, which are very similar to those produced from guaiacol, and possess the following advantages over creosote: they are nearly odourless, have only at first a bitter taste, and afterwards the taste is agreeable and sweetish; they are neutral, and have no injurious action on the mucous membrane, and are extremely easily soluble in water.—T. A. L.

## XXI.—PHOTOGRAPHY.

### PATENT.

*Developing, Toning, and Fixing Photographs; Impts. in Method of and Apparatus for.* G. A. de Katow, London. From A. Schwarz, Berlin-Schoneberg, Germany. Eng. Pat. 21,679, Sept. 22, 1897.

This patent contains a description of automatic machinery for developing, toning, and fixing a continuous roll of photographic paper; the special feature being that provision is made for controlling the time during which the film is subjected to the action of each several reagent, so that the intensity of development or the shade of toning may be altered at will. This adjustment is effected either by changing the speed at which the paper travels through the apparatus, or by making it pass through more or less of the active liquid. The latter object again is attained either by connecting the bottom of the developing or toning bath by means of a flexible tube with a portable reservoir which can be raised or lowered, or else by arranging the guide roller, which is underneath the surface of the liquid, in such a manner that it can be moved and fixed at any point between the bottom and the top of the vertical trough. The counter-shaft of the whole machine is provided with a clutch at its centre and a hand-crank at its free extremity, so that in the event of the paper breaking, the torn portion may be instantly wound backwards out of the baths, thus preventing the loss by over-development, &c. of that part of the film which at the time of the accident happens to be under treatment.—F. H. L.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### PATENTS.

*Explosives and Explosive Substances, Impts. in or relating to the Manufacture of.* E. A. G. Street, Paris. Eng. Pat. 13,724, June 3, 1897.

This invention aims at the production of a stable explosive of the chlorate class, without danger during course of manufacture. Advantage is taken of the fact that certain oils, when heated, dissolve nitro- and azo-compounds. If the solubility of the nitro- or azo-compounds be small, other substances more soluble, and formed by combination of two nitro- or two azo-compounds, or one nitro- and one azo-compound, may be used, the oil being always in sufficient quantity to preserve the fluidity of the whole combustible mixture during the course of manufacture.

The oily solution is mixed with one or more finely pulverised alkaline chlorates or perchlorates, "with or without addition of a carbonated or hydrocarbonated substance." The oily mixture surrounds the chlorate, avoiding the hygroscopicity of the salt, and the oil is found not to exude. Castor, linseed, and olive oils are found to be suitable solvents for mononitronaphthalene, and a mixture of equal parts of oil and nitro-body can be prepared at 65° C., and dinitrobenzene will give a clear liquid with its own weight of oil at 90° C. Azo-compounds "(azobenzene, azoxybenzene, amidoazobenzene, diamidoazobenzene)" are found to dissolve as readily as the nitro-compounds. Picric acid, which is found not sufficiently soluble in oil, when melted with mononitronaphthalene in the proportion of 1·32 parts to 1 part, produces a mixture which will dissolve in its own weight of oil at 105°—110° C. Other combinations are given. It is found that a good explosive should contain about 10—20 per cent. of oily mixture as described; 80—65 per cent. of chlorate of an alkali; 10—15 per cent. of fecula, starch, or carbon; and, if the latter be dispensed with, 20—30 per cent. of oily solution; 80—70 per cent. of chlorate.—W. M.

*Blasting Powder, An Improved.* S. Fülop and M. J. Lackovic, both of Buda-Pest. Eng. Pat. 13,822, June 4, 1897.

This powder consists of a mixture in the proportion of horse dung, 28 parts; saltpetre, 39 parts; fine gunpowder, 23 parts; and sulphur, 10 parts.—W. M.

*Matches and Flaming Fuses, Impts. in.* W. Salter, Wimbledon, Surrey. Eng. Pat. 228, Jan. 4, 1898.

In this invention the disadvantages attending the use of phosphorus and sulphur are obviated by substituting for them in the deflagrating composition, aromatic gums, which cause diffusion of agreeable antiseptic and aromatic odours during the combustion. The stems of the matches are also impregnated with essential oils, in the case of wax stems the essential oils being mixed with the stearin. A suitable deflagrating mixture is—

| | Parts. |
|---|---|
| Potassium chlorate | 98 |
| Oxide of iron | 6 |
| Whiting | 6 |
| Powdered glass | 6 |
| Potassium carbonate | 2 |
| Gum acroides or "gum olibanum, either or both" | 6 |
| Glue or gelatin | 6 |
| Gum arabic or gum tragacanth, or both conjointly | 6 |

—W. M.

*Lucifer Matches, Impts. in.* W. L. Gordon, New York. Eng. Pat. 1007, Jan. 13, 1898.

This patent is for producing a headless match, free from phosphorus, by immersing the stems in a solution of the following composition: 87·5 parts of sodium chlorate, 12·5 parts of gum arabic, 7·5 parts of a metallic sulphate, preferably copperas, and 125 parts of water. The ends are then thoroughly dried and the match impregnated with an inflammable waterproofing solution as far as the middle portion of the stem. Ignition is effected by scratching against a suitably prepared surface. The waterproofing solution may preferably consist of 25 parts of rosin, 67·5 parts of liquid turpentine, 125 parts of oleic acid, and 37·5 parts of linseed oil.

The coating upon which the match is to be ignited by friction is to consist of 25 parts of amorphous phosphorus, 12·5 parts of "antimony," 3·75 parts of glass powder, 25 parts of water, and 6·25 parts of gum arabic.—W. M.

*Fuse Cord for transmitting Ignition without Flame or visible Fire, Impt. in the Process for the Manufacture of.* G. C. Marks. From Westfälisch-Anhaltische Sprengstoff-Actien Gesellschaft, Wittenburg, Prussia. Eng. Pat. 2225, Jan. 27, 1898.

The invention relates to the class of fuse where the flame is suppressed by impregnating the fuse threads with oil, with which are mixed salts having a high percentage of water. The authors find it advantageous not to employ the usual fibrous substances, such as cotton, jute, and the like, but to mix them with nitrocellulose or use nitrocellulose alone, impregnating them with oils to which the usual drying agents have been added. The more effectually to suppress flame, albuminous and albuminoid substances are added; and to prevent the absorption of water and keep the fuse rigid, solutions of lacs, varnishes, &c., may be employed. For the salts containing a high percentage of water, a mixture of these and chromates, manganates, chlorates, and perchlorates may be substituted, or the latter alone may be used. It is an advantage to use diazo-compounds, and it is claimed that the oil and salts in the fuse cord will prevent the decomposition of the diazo-compound during storage. The fuse may be rendered impervious to moisture by means of chrome glue.—W. M.

*Smokeless Powder, Process for the Manufacture of.* B. Schratzenthaller, Vienna. Eng. Pat. 3976, Feb. 17, 1898.

This invention relates to the production of a smokeless powder, in the form of plates, by immersing paper boards in suitable nitrating mixtures, subsequently freeing the product from acids by washing, and immersion in dilute ammonia, and then further washing. For producing grains, the paper is first reduced to the state of powder, nitrated, and, when purified, worked up into form.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### *APPARATUS, ETC.*

*Vacuum Fractionating Apparatus.* R. Steinlen. Chem. Zeit. 1898, **22**, [18], 157—158.

The apparatus in question can be put together in the laboratory by the use of materials which are easily procured. In Fig. 1, *a* represents the extended neck of a Drechsler's wash bottle, into which a stopcock *b* with wide opening is fused, the lower end *c* being bent to form an obtuse angle and passing through a caoutchouc stopper, which is well secured by surrounding it with a luting consisting of a concentrated solution of calcium chloride, prepared by allowing the latter to absorb moisture from the air. The tube *d*, channeled through the ground stop-cock e, is provided at its outer terminal with a widened shoulder, to which a Liebig condenser is attached. The laterally bent tube *f* is fitted with a three-way tap *g*, which may, however, be replaced by a T piece with india-rubber connections and screw clamps (see Fig. 2). The distillate is collected in test tubes arranged on a circular wooden stand, the disc *h* being moveable, so that when operating upon larger quantities of liquid, beakers can be used instead of test tubes. The fractionation of the distillates is effected by turning *a*, which rotates in air-tight fittings, so that the liquid flows into the second tube. If after fractionation it be desired to subject each portion separately to rectification, the bell-jar A is exhausted continuously and independently of the distilling flasks by regulating the stop-cocks *b* and *g*. On closing the former, the communication between A and the distilling flask is stopped, whilst by turning the three-way tap *g* in a certain direction, it is possible to connect the flask with the atmosphere with a view of introducing a fresh charge. If it be desired to restore the vacuum in the flask, the operation of connecting the latter with the bell-jar does not materially influence the column of mercury indicated by the manometer.

Fig. 1.

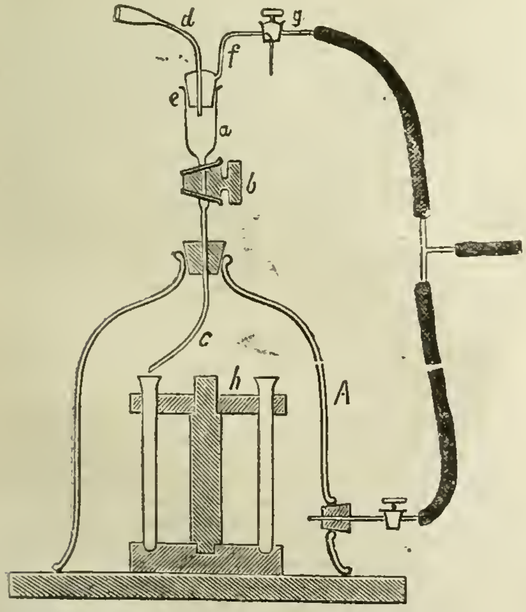
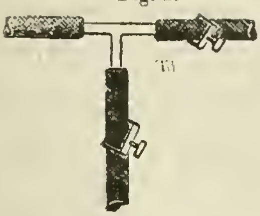
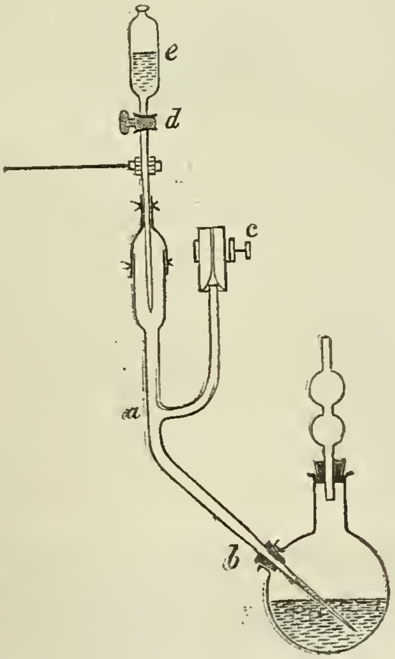


Fig. 2.



With very volatile substances or liquids, which can be distilled only under a greatly diminished pressure without undergoing decomposition, the charge must be introduced into the flask by excluding the air. For this purpose the device illustrated in Fig. 3 is recommended. The oblique tubulure *a*, which is drawn out to a fine point, passes through the stopper *b* into the lower part of the flask. It has a capillary branch tube fitted with rubber connection and screw clamp, and carries a somewhat wider tube, into which a separating funnel *e* is entered. The flask is charged by closing *c* and opening *d*. The latter is then closed, and *c* regulated, so that only a slight current of air passes through the liquid in order to accelerate the distillation.
—D. B.

Fig. 3.



*Gas Balance for Indicating Continuously the Proportion of Carbonic Acid Gas in the Flow of Furnace Gases.* Report of a Committee of Investigation of the Franklin Institute. J. Franklin Inst. 1898, **145**, 205—213.

The instrument, termed an "Econometer," is the invention of M. Arndt, of Aix-la-Chapelle (see Eng. Pat. 6211 of 1895; this Journal, 1896, 472, and 1897, 935). The smoke gases of steam boiler furnaces consist chiefly of oxygen, nitrogen, carbonic oxide, and carbonic acid, of which the first three are of nearly the same specific gravity as air, whilst the specific gravity of carbonic acid is 1·52. The greater the proportion of carbonic acid in smoke gas, the heavier, therefore, it is. When the precise amount of air necessary for the perfect combustion of the carbon in the fuel is supplied, the carbonic acid should amount to 20 per cent. of the volume of the smoke gases. When a greater amount of air is supplied, a smaller percentage of carbonic acid is found in the smoke gases. The econometer consists of a balance-beam, carrying at one end a gas container having at the bottom an open neck, through which passes upwards into the container a gas pipe through which the filtered smoke gas can enter the same. The neck is surrounded by a cup which communicates with a suction apparatus, by means of which the gas can be withdrawn from the container. The other end of the balance-beam carries a vessel and weights which counterpoise the gas-containing vessel when air is drawn through the apparatus, and the pointer attached to the beam is at the zero mark on a scale. The scale is graduated so that the oscillations of the pointer in front of it show directly the percentage of carbonic acid in smoke gas passing through the gas vessel at the time. Variations in the proportion of carbonic acid in the smoke gas affect the density of the latter, and the gas-containing vessel moves up and down with fluctuations in the density of the gas passing through it, and these movements are reflected by the pointer on the scale. The instrument is in a nearly air-tight case, and works continuously, so that a fireman may observe the conditions of combustion at any time. A table supplied with the instrument, shows the surplus quantity of air passing through the flues, and the loss of fuel when the gases leave the boiler at 270° C., for given percentages of carbonic acid found by the instrument in the smoke gases.

Tests on a Babcock and Wilcox boiler, burning anthracite coal were made, and satisfied the investigating committee of the substantial accuracy of the instrument. The following is a summary of the results of the tests :—

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Average of gas balance readings, numbering from 10 to 15 in each instance. | 3·95 | 3·915 | 3·81 | 8·52 | 10·38 | 7·02 | 3·857 |
| Percentage of carbonic acid found by analysis of a sample of smoke gas taken during the period covered by the readings. | 4·02 | 4·84 | 4·12 | 9·06 | 10·53 | 6·57 | 4·38 |

—J. A. B.

## INORGANIC CHEMISTRY—QUALITATIVE.

*Carbon Monoxide, Detection of, in Air by Means of Palladium Chloride; and Transformation of, into Carbon Dioxide at the Ordinary Temperature.* Potain and Drouin. Comptes Rend. 1898, **126**, [13], 938—940 (see under XXIV., pages 484 and 503).

If a given volume of the air is aspirated in small bubbles through a tube filled with 10 c.c. of a solution of palladium chloride (containing 1 mgrm. of palladium) and two drops of hydrochloric acid, a portion of the palladium is reduced to the metallic state. A fair idea of the quantity of carbon monoxide in the air may then be formed by filtering off the precipitated palladium and comparing the tint of the filtrate with that of the original solution. The authors also find that carbon monoxide mixed in small quantity with air, is slowly transformed into carbon dioxide. This transformation however, is retarded and limited by the presence of carbon dioxide itself.

In spite of the fact that large quantities of carbon monoxide are continuously being discharged into the air, only traces of this gas can in general be found. According to the authors this is due to its transformation into carbon dioxide.—J. S.

*Alkali Carbonates in Presence of Excess of Bicarbonates or Borax, Qualitative Examination for Traces of.* A. Leys. Bull. de l'Assoc. des Chim. 1898, **15**, [7], 723—724.

In order to distinguish alkali carbonates from bicarbonates, a salt of magnesium is used, the sulphate, for example. Normal carbonates give a white precipitate, whilst with bicarbonates the mixture remains clear. This reaction, which gives excellent results with the salts separately, fails with mixtures of them, as is the case in certain products sold as "milk preserver," or where borax is present.

A saturated solution of calcium sulphate, however, remains bright for a certain time when pure sodium bicarbonate is added, but gradually becomes dim from separation of microscopic crystals. With pure borax no turbidity is seen even on standing. Neutral carbonate, even in very small quantity, gives a precipitate immediately.

Commercial bicarbonate of soda sold as pure,' when treated with a quantity of water insufficient to dissolve it, gives an immediate precipitate with calcium sulphate, so delicate is the reaction, and it is only on a third treatment with water that a pure solution of the bicarbonate is obtained.

Both solutions should therefore be used. When magnesium sulphate gives no precipitate, calcium sulphate should be tried before concluding that normal carbonate is absent.—L. J. de W.

## INORGANIC CHEMISTRY.—QUANTITATIVE.

*Carbon Monoxide, Estimation of, in Large Quantities of Air.* A. Gautier. Comptes Rend. 1898, **126**, [11], 793—795.

The author was on the point of publishing a method which he has used for the past seven or eight years, and which permits of the detection of 1 volume of carbon monoxide in 20,000 volumes of air, when a somewhat similar method was described by Nicloux (this Journal, 1898, 377). The method is based on the oxidation of the carbon monoxide at a temperature of about 60° by iodic anhydride, iodine being set free. Nicloux determines the amount of iodine liberated by the depth of colour produced in carbon bisulphide or chloroform, whilst the author fixes the iodine by copper, and weighs it directly. Details of the apparatus and of the results obtained will be given in a subsequent communication. It should be observed that acetylene also liberates iodine above 60° C., and that sulphuretted hydrogen and sulphur dioxide must first be removed by passing the air through an alkaline solution.—J. S.

*Carbon Monoxide, Estimation of, in Air.* A. Gautier. Comptes Rend. 1898. **126**, [13], 931—937. (Compare this Journal, 1898, 377.)

Carbon monoxide begins to act on iodic anhydride at 30° C.; the action becomes more pronounced at 40°—45° C., and is complete at 60°—65° C. According to the author, this reaction, *viz.*, $5CO + I_2O_5 = 5CO_2 + I_2$, takes place, no matter what the dilution of the carbon monoxide may be. At first the attempt was made to estimate small quantities of carbon monoxide in air by passing the air, freed from $CO_2$, over the iodic anhydride heated to 65°—70° C., and (1) collecting and weighing the iodine in absorption tubes filled with finely divided copper or silver, or (2) by applying certain volumetric methods. Neither of these proved sufficiently accurate, and therefore the author had recourse to Muntz and Aubin's method (Ann. de l'Institut Agronomique, 1881—82), which consists in collecting the carbon dioxide formed, in caustic potash solution (freed from carbonate by means of baryta) and subsequently liberating it again with sulphuric acid, and measuring over mercury. The volume of carbon dioxide found is equal to the volume of carbon monoxide oxidised. Test analyses are given in detail, showing the accuracy of the method. A volume of air containing about one-thousandth part of carbon monoxide (9·83 c.c.) was analysed, and the volume of carbon dioxide found was 9·73 c.c. Air from the laboratory contained 0·0199 and 0·0297 c.c. of CO per litre.

The method is not accurate in presence of certain hydrocarbons, notably acetylene and ethylene; and hence the author proposes to modify it in a suitable manner.—J. S.

### *Rare Earths and Bases of the Fourth Group, New Method for the Simultaneous Estimation of.* G. Possetto. Giorn. Farm. Chim. **48**, 49—54. Chem. Centr. 1898, **1**, [10], 634—635.

The metals of the second group, &c., are separated by precipitating with sulphuretted hydrogen in acid solution, and to the filtrate is added ammonia and ammonium sulphide. The resulting precipitate may contain the hydrates of iron, chromium, aluminium, cerium, lanthanum, thorium, zirconium, yttrium, uranium, glucinum, and didymium, the sulphides of manganese, zinc, nickel, and cobalt, and the oxalates, borates, phosphates, &c., of barium, strontium, calcium, and magnesium. The precipitate is analysed in the following manner:—It is thoroughly washed and treated with warm hydrochloric acid, thus leaving undissolved only the sulphides of nickel and cobalt, which may be separated in the usual way. The acid solution is treated with a few drops of nitric acid, boiled, cooled, and rendered slightly alkaline with concentrated potash. The precipitate contains all the elements present except glucinum, which may be precipitated from the filtrate by adding water and warming. The main precipitate is boiled with potash to dissolve the zinc and aluminium, and the latter detected in the solution by means of sulphuretted hydrogen and ammonium chloride respectively. The residue is allowed to stand for 12 hours in ammonium carbonate solution; in this way the bicarbonates of the rare earth metals are formed and are dissolved by the ammonium carbonate, whilst the manganese, iron, and chromium, and the oxalates, borates, and phosphates of the alkaline earths remain undissolved. In this residue iron, chromium and manganese may be detected. The remainder of the residue is boiled with soda solution, filtered, the residue dissolved in hydrochloric acid, the solution precipitated with ammonia and ammonium sulphide, filtered, and the filtrate added to the first filtrate, and tested as in the following group by means of ammonium carbonate. The solution containing the bicarbonates of the rare earths is boiled until the carbonates are precipitated, and the precipitate analysed as follows:—A small portion is treated with hydrogen peroxide. If the colour change from white to yellow, cerium is present. A further portion is brought into contact with a crystal of iodine, when if lanthanum be present, the colour changes from white to blue. Another portion is dissolved in very dilute hydrochloric acid, and heated gradually to 100° C. with a strip of turmeric paper. If zirconium be present, the paper is coloured orange-red. A second portion of the hydrochloric acid solution is tested with potassium ferrocyanide, a red coloration being produced in presence of uranium. A third portion of the acid solution is treated with sodium bicarbonate solution. If didymium be present, there is formed a white precipitate insoluble in excess of the reagent.

The remainder of the precipitate of carbonates is dissolved in hydrochloric acid, the solution neutralised with potash and boiled with potassium sulphate, thus precipitating the double sulphates of thorium, zirconium, and cerium, Yttrium sulphate remains in the solution, and may be separated as yttrium hydrate by means of ammonia. The double sulphates are boiled with hydrochloric acid, the solution filtered from the insoluble zirconium, and the cerium and thorium in the filtrate precipitated as oxalates. The precipitate is dissolved in concentrated ammonium oxalate solution, the cerium oxalate separated out by diluting with water, and the thorium in the filtrate being precipitated as hydrate by means of ammonia.—A. S.

### *Yttria Earths, New Method of Fractionating the.* G. Urbain. Comptes Rend. 1898, **126**, [11], 835—838.

Yttrium ethyl sulphates in the form of beautiful hexagonal prisms are obtained by decomposing the sulphates of the yttria earths with barium ethyl sulphate. The author then records the results of the fractional crystallisation of these substances in a series of tables. In general this method leads to the same results as were previously obtained by the author and Budischowsky (Comptes Rend. 1897) on the fractional crystallisation of the aceto-acetates, and by Schützenberger and Boudouard (this Journal, 1897, 429) by the fusion of the nitrates and the fractionation of the sulphates.

From the measurements given in the tables, it appears that on the fractionation of the ethyl sulphates, the metals separate out in the following order:—Yttrium, terbium, holmium and dysprosium, erbium, ytterbium.—J. S.

### *Cerium, Determination of, in Presence of other Rare Earths.* G. von Knorre. Zeits. f. angew. Chem. 1897 [22], 717—725.

A continuation of the author's former paper (this Journal, 1898, 72). Figures given in the paper show that the method is applicable to the determination of cerium in presence of salts of lanthanum, didymium, thorium, and ferric iron, as also to the determination of ceric in presence of cerous salts, even when these other substances are present in relatively large amount. To make the method applicable to the determination of cerous cerium, the author has sought a method of converting this sharply into the ceric condition. Oxidation by nitric acid and lead peroxide, or by electrolysis, proved far from complete, but potassium (or preferably ammonium) persulphate was found to furnish exact results under certain conditions. To the solution of cerous salt, acidified with sulphuric acid, an excess of ammonium persulphate is added, and the liquid boiled; after cooling, the titration with hydrogen peroxide and permanganate is conducted as described in the former paper. Too much sulphuric acid must not be used, or the ceric salt is partially reduced, no doubt through the persulphuric acid breaking up into sulphuric acid and hydroxyl, which then reduces some of the previously formed ceric salt. By direct experiment the author was able to prove the formation of hydrogen peroxide, when strong sulphuric acid acts on ammonium persulphate. Should the solution be already strongly acid, the greater part of the free acid should be neutralised before adding the persulphate; and nitric acid, if present in any large quantity, should be removed. Moreover, persulphate and hydrogen peroxide are not without action on one another, so that the excess of persulphate should be as far as possible removed from the solution before the hydrogen peroxide is added: this is readily done by a few minutes' boiling of the acidified solution. The process is carried out as follows:—Acidify the cerous solution with just enough sulphuric acid to prevent the precipitation of basic ceric sulphate on boiling; add in the cold ammonium persulphate solution, and heat to boiling for one or two minutes; cool to 40° or 60° C., add a second portion of persulphate, and boil again; cool again, add a third portion of persulphate, and this time boil for 15 minutes; add a little more sulphuric acid, and allow to cool (for 0·2 grm. of cerium about 3 grms. of ammonium persulphate will be needed, of which half should be used at first, and a quarter at each of the subsequent additions). The titration with hydrogen peroxide and permanganate is carried out as soon as the solution is cold; but to avoid action between the peroxide and any undestroyed persulphate, it is desirable to add the peroxide from a burette and to stop as soon as the disappearance of the yellow tint shows that reduction is complete—in this way a large excess of peroxide is avoided. The experimental figures show the process to be accurate, not only for pure cerous salts, but for mixtures containing large excess of salts of thorium and didymium. Instances are given of the application of the method to the determination of cerium in Welsbach mantles, in commercial thorium nitrate, in the fluid used for the impregnation of mantles, and in monazite sand. In the last case, the sand was treated with strong sulphuric acid till copious fumes of the acid were evolved, and the residue after cooling was diluted with water. The solution contained the whole of the earths, together with iron salts, and phosphoric and titanic acids, while the residue was made up of silicates, magnetite and titanic iron. The titanic and phosphoric acids in the solution were found to interfere with the process, and the solution should therefore be treated as follows:—To the not very dilute solution add excess of oxalic acid, and then dilute very considerably; the bulk of the oxalate precipitate comes down at once, the whole of the rest of it on dilution, without carrying down any phosphoric or titanic acid, which almost always come down if the solution be greatly diluted before precipitation. Convert the washed and dried oxalates into oxides by ignition, dissolve in concentrated sulphuric acid (should any residue remain, evaporate on the water-bath with hydrogen peroxide, and again heat with strong sulphuric acid); then carry out the determination by the process as given above.

The author has confirmed the work of Clève (1885), and finds that by adding to a cerous salt a great excess of hydrogen peroxide in the cold, and then making just alkaline with ammonia, a precipitate of a composition corresponding to Clève's cerium peroxide, $CeO_3$, is obtained; but he considers that the action of sulphuric acid on this does not proceed according to Clève's equations, $2CeO_3 + 2H_2O = 2CeO_2 + 2H_2O_2$ and $2CeO_2 + H_2O_2 = Ce_2O_3 + H_2O + O_2$, but that the peroxide is at once converted into cerous sulphate, with formation of a corresponding amount of hydrogen peroxide, $2CeO_3 + 3H_2SO_4 = Ce_2(SO_4)_3 + 3H_2O_2$.—J. T. D.

*Ammonia Soda, Analysis of Waste Liquors from Distillation.* K. W. Jurisch. Zeits. angew. Chem. 1898 [12], 273—274.

For complete analysis of the liquors running from the stills, the author uses the following method:—

Either a continuous sample is taken, or an average one is obtained from a mixture of equal portions of liquor withdrawn at definite equal intervals.

A.—25 c.c. of the sample are titrated with N-hydrochloric acid. Warming is necessary to bring all the $CaCO_3$ into solution, and litmus is used as indicator. The acid consumed, say A c.c. per 100 c.c. sample, indicates the amount of $CaO_2H_2$, $CaCO_3$, and $NH_3$.

B.—100 c.c. of the sample are shaken with cold water and 35 grms. of ammonium chloride, and diluted with cold water to 1 litre. 500 c.c. are filtered off and titrated with normal acid for $CaH_2O_2$ and $NH_3$. If 100 c.c. sample require B c.c. of acid, then $CaCO_3$ = (A—B) c.c.

C.—From the litre a further 100 c.c. are filtered and boiled with ammonium carbonate until ammonia ceases to be evolved. The precipitated arragonite is filtered and titrated with normal acid. This gives the $CaCl_2$ originally present, and also that formed under B from the $CaH_2O_2$. Let 100 c.c. of sample take C c.c. of acid.

D.—100 c.c. of the sample are distilled with caustic soda and the evolved ammonia collected and titrated. If it require D c.c. of N acid, then (B—D) c.c. gives the amount of $CaH_2O_2$.

E.—1 c.c. of the sample (10 c.c. of a solution obtained by diluting 10 c.c. of the sample to 100 c.c.) are titrated with $\frac{1}{10}$N silver nitrate solution. This gives the total chlorine due to $CaCl_2$ and NaCl. ($NH_4Cl$ is absent in presence of $CaH_2O_2$). Let 100 c.c. of the sample require E c.c. of $\frac{1}{10}$N $AgNO_3$. The following quantities are thus obtained for 100 c.c. of the sample:—

| | | | |
|---|---|---|---|
| $CaO_2H_2$ | = | [B − D] × 0·037 | grm. |
| $CaCO_3$ | = | [A − B] × 0·050 | „ |
| $NH_3$ | = | D × 0·017 | „ |
| $NH_4Cl$ | = | 0·0 | „ |
| $CaCl_2$ | = | [C − B + D] × 0·0555 | „ |
| NaCl | = | [E − 10(C − B + D)] × 0·00585 | „ |

If the sample contains no caustic lime, then B gives only free $NH_3$; test C gives both free ammonia and that combined as $NH_4Cl$. Test E gives the chlorine present as $CaCl_2$, NaCl, and $NH_4Cl$.

The ratio $\frac{C}{C - B + D}$ gives the number of equivalents of lime used per each equivalent of ammonium chloride decomposed.

The following table shows the magnitude of the above values:—

Analyses of March 23, 1885.

| | Time. | | | | | | | | | | | | Mean. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 9.0. | 9.30. | 10.0. | 10.30. | 11.0. | 11.30. | 12.0. | 12.30. | 4.0. | 1.30. | 5.0. | 5.30. | |
| A | 71·9 | 91·7 | 98·8 | 125·4 | 35·2 | 48·8 | 131·4 | 22·4 | 41·2 | 120·0 | 195·6 | 73·8 | 88·51 |
| B | 34·7 | 40·8 | 70·43 | 99·81 | 15·4 | 21·4 | 108·22 | 11·6 | 10·4 | 100·4 | 164·57 | 58·2 | 61·58 |
| C | 114·0 | 105·93 | 161·81 | 180·28 | 135·08 | 147·99 | 178·99 | 103·4 | 110·0 | 182·98 | 192·97 | 153·6 | 147·09 |
| D | 0·52 | 0·36 | 0·42 | 0·33 | 0·72 | 0·81 | 0·23 | 25·0 | 9·4 | 0·42 | 0·4 | 0·6 | 3·27 |
| E | 2,585·0 | 1,470·0 | 2,110·0 | 1,610·0 | 2,570·0 | 2,600·0 | 1,435·0 | 2,560·0 | 2,385·0 | 1,720·0 | 640·0 | 2,000·0 | 1,973·75 |
| C + D − B | 79·82 | 65·49 | 91·8 | 80·8 | 120·4 | 124·4 | 69·0 | 116·8 | 109·0 | 83·0 | 28·8 | 96·0 | 88·78 |

—J. T. C.

*Copper Bullion, Assaying of.* Eng. and Mining J. 1898, **65**, [8], 223.

The following method is stated to give slightly higher results, and at the same time to be quicker and more economical, than most other methods. Weigh out "1 A.T." into a suitable beaker and cover with 120 c.c. of distilled water, add 100 c.c. of nitric acid (C.P.), and when violent action ceases, boil until red fumes disappear; dilute to 300 c.c. and filter. In the bottom of a 2·5-in. scorifier spread 5 grms. of test lead, and also sprinkle the filter paper with 2·5 grms. of finely-divided test lead, then gather the edges of the paper together and press it into the scorifier, so as to flatten the paper with the lower portion in the layer of lead. This is said to ensure the contact of every particle of gold with metallic lead, and thus to prevent mechanical loss. To the hot filtrate from the gold, add a slight excess of salt, and stir vigorously at intervals, until the silver chloride separates, which should not take more than 30 minutes. It has previously been thought necessary to let the solution stand over night, and also to produce a dense precipitate of lead sulphate, which, in settling, will collect the silver, but, except in the case of very poor bullion, these operations are unnecessary. Even when the solution is not stirred, from 1—2 hours is sufficient for the silver chloride to separate, so as to allow of its being completely filtered off, the proper time being indicated by the appearance of fine clear blue lines on the surface of the solution where the chloride has commenced to settle. It is preferable to filter through a double paper, and in this operation there is often recovered a little gold which has escaped the first filtration. Sprinkle the filter paper containing the precipitate with 2·5 grms. of finely-divided test lead, transfer to the scorifier containing the gold, and place the latter over the muffle until the paper is dried, and burnt. When combustion has ceased, place the scorifier in the opening of the muffle for a few seconds to complete the incineration, cover the partially fused mass with 15 grms. of lead and 0·5 grm. of borax and scorify at a low heat. The resulting button weighs only about 4 grms., and may be cupelled in a very small cupel, its comparatively small weight greatly diminishing losses by absorption and volatilisation. Parting is performed by the usual treatment with dilute and strong nitric acid.—A. S.

*Strontium Hydrate [Sugar], Complete Analysis of.* V. Zamaron. Bull. de l'Assoc. des Chim. 1898, **15**, [8], 816—819.

In testing strontium hydrate, $Sr(OH)_2 + 8H_2O$, the following determinations are made :—The water of substitution, the insoluble in boiling water, the hydrate of strontium ($SrO.9H_2O$), the lime, the sulphides.

The strontium hydrate is ground in a porcelain mortar pretty finely, so as to have a homogeneous sample, and the material is kept in a stoppered flask with wide mouth.

To determine the water of substitution, 5 grms. are weighed out and heated to 100°—110° C. The substance is placed for this purpose in a porcelain crucible contained in a Scheibler's dessicator (hermetically closed), and the whole placed in the oven. After 20 to 24 hours, the water of substitution and water of hydration are removed, with the exception of the last equivalent of water, which can only be driven off at 900° to 1,000° C.

For the insoluble, 2 grms. are placed on a filter and washed with boiling water until the washings are no longer alkaline to litmus. The filter is incinerated and weighed.

In the filtrate the strontium is determined as carbonate, by precipitation with ammonium carbonate and ammonia. The strontium carbonate multiplied by 1·8 gives the strontium hydrate in 2 grms. The precipitate must be well washed. To determine the lime, the above precipitate is dissolved in nitric acid, and the solution evaporated until nitrous fumes cease to come off. The vessel containing the nitrates is allowed to cool over sulphuric acid, to avoid absorption of water, and when cold, 30 c.c. of a mixture of ether of 65° and an equal volume of absolute alcohol, are added. After 20 minutes the liquid is filtered, and a few drops of sulphuric acid added. In the presence of lime a white precipitate forms, which may be weighed as calcium sulphate. The weight multiplied by 0·411 gives the weight of CaO for 2 grms. of substance.

The sulphur is determined in a solution of 20 grms. in boiling water, by adding a standard solution of nitrate of silver. The weight of silver sulphide multiplied by 0·481 gives the weight of strontium sulphide.—L. J. de W.

### *Phosphoric Acid, Estimation of very Small Quantities of.* A. Jolles and F. Neurath. Monatsh. für Chem. 1898, **19**, [1], 5—15.

The authors have devised and examined several methods for the estimation of traces of phosphoric acid.

I. Solution of the phosphomolybdate precipitate in sodium carbonate, and estimation of ammonia by Nessler's reagent. The phosphomolybdate precipitate, which contains $6NH_3$ to $P_2O_5$, was washed with a mixture of equal parts of 45 per cent. alcohol and 10 per cent. nitric acid. This had been found to give better results than other solutions not containing ammonium salts. The results were unsatisfactory.

II. Solution of the phosphomolybdate precipitate in ammonia, evaporation of the solution on the water-bath, and colorimetric estimation of ammonia in the residue. The results were not satisfactory. After evaporation of the ammoniacal solution, a white residue is left, having the composition $(NH_4)_3PO_4.5(NH_4)_2Mo_2O_7$. This salt (0·1 per cent. solution) gives colour reactions with salts of certain heavy metals which are not given either by ammonium phosphate or molybdate. The most delicate of these is a bluish-green coloration with ferrous salts. A method (III.) founded on this coloration gave uncertain results.

IV. Direct colorimetric determination with potassium or sodium molybdate. Colorimetric methods with ammonium molybdate have been proposed by Nördlinger and Lepierre. These are not sufficiently delicate, and have the disadvantage that precipitation soon occurs. Potassium molybdate gives the most delicate reaction. At 80° C. the coloration reaches its greatest intensity; 0·0000025 grm. of $P_2O_5$ in 20 c.c. may be recognised at this temperature. In a test tube are placed 20 c.c. of the colorless solution to be examined, 1 c.c. of potassium molybdate (8 grms. of the salt in 50 c.c. of water mixed with 50 c.c. of nitric acid of sp. gr. 1·2) is added, and the mixture warmed to about 80° C. In similar tubes 20 c.c. of sodium phosphate solutions containing 0·001, 0·00075, 0·0005, &c. grms. of $P_2O_5$ are treated in a similar manner. By a comparison of the colorations produced, the amount of phosphoric acid may be determined with tolerable accuracy. The presence of inorganic salts in such quantities as they occur in natural waters is without influence on the determinations. Larger quantities, especially of iron and silicic acid, influence the accuracy of the method. If more than 0·001 grm. of $P_2O_5$ be contained in the 20 c.c. of solution employed, turbidity will result. Such solutions should be diluted.—A. C. W.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

### *Flour, Detection of Sawdust or Wood Meal in.* G. A. Le Roy. Rev. de Chim. Industr. 1898, **9**, [100], 114—115.

When flour adulterated with sawdust is moistened with an alcoholic solution of phloroglucinol and gently heated, the woody particles are coloured an intense red, and are easily detected with the naked eye, or with the help of a lens, whilst the flour itself remains colourless. The solution of the reagent should be made strongly acid with phosphoric acid, and may be preferably composed of 10 parts of phloroglucinol, 150 parts each of alcohol (90—95 per cent.) and distilled water, and 100 parts of syrupy phosphoric acid.—W. P. S.

### *Pyruvic Acid, Two Colour Reactions for.* L. Simon. Bull. Soc. Chim. 1898, **19**, [7], 294—297.

This is an application of Legal's reaction for acetone. On mixing 1 c.c. of a solution of pyruvic acid with the same volume of potash (sp. gr. 1·15 to 1·20), and adding several drops of a freshly-prepared and fairly concentrated solution of sodium nitroprusside, a strong violet-red coloration is obtained. The limit of the reaction is 1 part of pyruvic acid in 50,000. The addition of a small quantity of acetic acid makes the violet shade more pronounced, but on adding an excess, the colour vanishes. The salts and esters give the same reaction, but a negative result is obtained with the phenylhydrazine compound. Phenylglyoxylic acid and its derivatives do not give any coloration, but the reaction is not characteristic of pyruvic acid alone, since it is given by many other substances, such as aldehydes, ketones, acetone, &c.

It is otherwise with the colour obtained on adding ammonia and nitroprusside to the solutions of pyruvic acid. The test is applied in the same manner as the preceding one, and the slow appearance of a violet-blue coloration appears to be quite characteristic of that acid or its derivatives, with the single exception of acetophenone, which is distinguished by giving a blue colour with Legal's reagent. A gentle heat accelerates the appearance of the colour, but it disappears altogether on boiling, or on the addition of an excess of acetic acid. The limit of sensibility is about 1 in 10,000.

On adding amines of the fatty acid series to pyruvic acid and sodium nitroprusside, a pronounced violet colour rapidly appears. On the addition of acetic acid this changes to blue and then disappears. This reaction is quite distinct from that obtained with ammonia. The blue colour appears more rapidly, but is more evanescent, especially in the presence of acetic acid.—C. A. M.

### *Acetaldehyde, A Colour Reaction for.* L. Simon. Bull. Soc. Chim. 1898, **19**, [7], 297—299; see also this Journal, 1898, 381.

Ordinary aldehyde shares with many substances the property of giving a red coloration with potash and sodium nitroprusside (Legal's reaction), but the following test has hitherto been found by the author to be characteristic of aldehyde alone. On adding to a dilute solution several drops of aqueous trimethylamine, and then several drops of a very dilute solution of sodium nitroprusside, a bright blue colour slowly appears. This is very intense with a solution containing 1 part of aldehyde in 1,000, is plainly visible in a solution of 1 in 10,000, whilst the limit of sensibility appears to be 1 in 25,000. The test is thus more sensitive than Legal's reaction. The colour is more evanescent than that given by Schiff's reagent. It disappears in about 15 minutes in the case of solutions of the strength named above. By means of this test the author could detect traces of aldehyde in ether, alcohol, and acetone. The last, when pure, gives a red colour with the reagent, but this is completely masked by the blue of the aldehyde when present in sufficient quantity (1 c.c. per litre). The colour is slightly more stable in the presence of alcohol. Not only does ammonia not give any blue coloration itself with aldehyde and nitroprusside, but it causes the colour to disappear when formed. The author suggests that this may be the explanation of the instability of the coloration. Potash displaces the amine, and the blue then gives place to the red of Legal's reaction. Acetic acid first changes the colour to violet and then causes it to disappear. Monomethylamine used instead of trimethylamine gives a violet colour in concentrated solutions of aldehyde, but the test is much less sensitive than the foregoing.—C. A. M.

*Phenylhydrazine, A New Colour Reaction for.* L. Simon. Bull. Soc. Chim. 1898, **19**, [7], 299—301.

When a solution of phenylhydrazine is momentarily warmed with several drops of aqueous trimethylamine and several drops of a solution of sodium nitroprusside added, a colour varying from blue to green is produced, which becomes more pronounced on the addition of a little concentrated potash solution. The addition of a little acetic acid before or after the potash modifies the reaction, the colour obtained being of a sky-blue shade. This reaction is capable of detecting phenylhydrazine in a solution of 1 in 50,000. The presence of ether or alcohol does not affect the reaction, but chloroform and benzene have a disturbing influence. Acetone gives its own coloration (the red of Legal's reaction). The presence of mineral or organic acids retards the appearance of the colour until the addition of the potash. Added in excess, acetic acid causes the colour to disappear. The presence of ammonia does not interfere with the test. The blue colour appears even in the cold, but more slowly. On heating the blue-coloured liquid, the tint finally becomes red when potash is present, and clear yellow when it is absent.

This reaction is not obtained with hydrazones, and appears to be exclusively characteristic of phenylhydrazine and its substitution derivatives in which the substitution occurs in the phenyl group. For example, negative results are obtained with formyl-phenylhydrazine—

$$(C_6H_5.NH.NH.COH)$$

and benzoyl-phenylhydrazine ($C_6H_5.NH.NH.CO.C_6H_5$), whilst blue colorations are given by ortho-cresylhydrazine ($CH_3.C_6H_4.NH.NH_2$) and *p*-bromo-phenylhydrazine ($C_6H_4Br.NH.NH_2$).

The blue colour is easily distinguishable from that given by aldehyde with the same reagent by its persistence in the presence of potash, ammonia, and acetic acid. Legal's reaction can also be used to detect traces of phenylhydrazine, but is not so characteristic or sensitive as the preceding test. Neither ammonia nor monomethylamine can take the place of trimethylamine in the reaction.—C. A. M.

*Cocaine Hydrochloride, Maclagan's Ammonia Test for the Purity of.* C. F. Boehringer and Soehne. Pharm. Centralhalle, **39**, [9], 141—145.

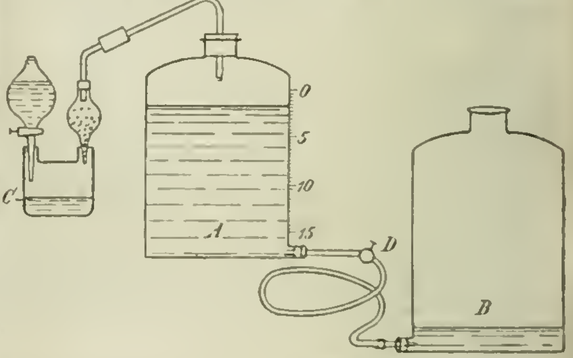
Fritz Guenther having adversely criticised the ammonia test of Maclagan, the authors reply at length to his strictures. They assert full confidence in the test, and that any sample of cocaine hydrochloride not responding to it will be found to contain the toxic isotropyl-cocaine in such proportions as to render the salt unfit for medical use. They consider that no uncertainty exists, as the impurity which prevents the free crystallisation of the liberated alkaloid, isotropyl-cocaine, alone has this inhibitory influence. So far from the test not being delicate, they find that it is possible by its aid to detect as little as 0·2 per cent. of the impurity. To render the test as definite as possible, the following details are given for its application:—One decigramme of the salt should be dissolved in 85 c.c. of water and 0·2 c.c. of ammonia solution (of sp. gr. 0·96) added, and the mixture stirred vigorously, the sides of the beaker being well rubbed with the glass rod immediately after the addition of the ammonia, and the solution then well beaten up.—J. O. B.

## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Calcium Carbide, Analysis of.* H. Bamberger. Zeits. f. angew. Chem. 1898, [11], 243.

When the yield of acetylene from calcium carbide is estimated gravimetrically from the loss in weight on evolution of the gas, in an apparatus similar to those used for the estimation of carbonic acid in carbonates by difference, the impurities in the gas may also be determined by attaching to the outlet of the calcium chloride tube a train of bulbs containing oxidising liquids. The solubility of phosphuretted hydrogen and sulphuretted hydrogen in the confining liquid renders impracticable their estimation when the yield of gas is determined volumetrically.

R. Fuchs and R. Schiff (Chem. Zeit. **21**, 875) estimate the yield of acetylene from carbide by collecting the gas over water, protected from it by a layer of oil. The movements of the oil and water, however, cloud the glass walls. The author therefore uses a modification, the apparatus for which is shown in the figure. A is a bottle of about 20 litres capacity, graduated in divisions of 0·1 litre, and is connected by flexible tubing with another bottle, B, of similar capacity. A concentrated solution of common salt, saturated at the temperature of the room, with acetylene, is used as the confining liquid in A. The gas is generated from the weighed granulated carbide in the two-necked bottle, C, which is provided with a tap-funnel and a tube packed with cotton-wool. The level of the solution in A is read after C is connected to A; the cock D is then opened, and water is allowed to drop slowly on the carbide until all the gas has been evolved therefrom, when the level of the solution in A is again read, and the volume of gas at the prevailing temperature and pressure is thus found.



—J. A. B.

*Carbon and Nitrogen, Simultaneous Estimation of, by Combustion in a Vacuum.* K. A. H. Mörner. Zeits. Anal. Chem. 1898, **37**, [1], 1—21.

The method adopted is that of Klingemann (this Journal, 1893, 869), based on the method of Frankland and Armstrong.

The fine copper oxide is moistened with a very small quantity of water before being intimately mixed and ground with the substance to be analysed. Before the combustion proper is commenced, the tube is heated by a hot-water trough placed in the furnace, until the whole of the moisture is given off, the pump being in action throughout. In this way the last traces of air are removed.

The author's experience is, that in the analysis of substances containing nitrogen, the gases resulting from the combustion cannot be assumed to be free from oxide of nitrogen, even when a copper spiral is used. For the gas analysis, he therefore proceeds as follows:—

1. The volume of the gases is measured.
2. The carbonic acid is absorbed by potash lye, and the residual gas measured.
3. A sufficient amount of oxygen is measured.
4. The oxygen is mixed over a little water, with the gases, and, after a time, treated with potash lye, and the

volume measured. The contraction, divided by 1·75, gives the oxide of nitrogen.

5. A portion of hydrogen, far too small to combine with the oxygen, is measured.

6. The measured quantity of hydrogen is mixed with the gas and exploded. The volume is measured. The contraction indicates the nature of the combustible gases.

7. The carbonic acid formed by the explosion is absorbed by potash, and determined by measuring the residual gas.

8. An excess of hydrogen is measured.

9. The hydrogen is mixed with the gas and exploded. The residual gas is measured. From the contraction the oxygen along with the hydrogen and nitrogen are calculated.

The description makes the analysis appear tedious, but it can be performed and the calculation made in four hours, and the exactitude of the nitrogen determination is assured.

Details of a number of analyses are given in the paper, and a full description of the apparatus used.—L. J. de W.

*Indigo, Analysis of.* C. Kurz. Rev. Gén. des Mat. Col. 1898, **2**, 91—92.

Finely-ground Indigo is mixed with small pebbles and extracted in a Soxhlet apparatus with phenol, or, better, with naphthalene. The indigotin is rapidly dissolved, and is less attacked by the solvents than it is with aniline and acetic acid.—E. B.

*Weighting in Black Silks, Determination of the.* R. Gnehm and E. Blumer. Rev. Gén. des Mat. Col. 1898. **2.**

A weighed quantity of the silk (not less than 1 grm.) is submitted to the following treatment:—

(1) The sample is treated with a 1 per cent. solution of hydrochloric acid at 60° C. The red solution is filtered off, and the treatment is repeated until only a tinge of red is extracted. As a rule two or three successive treatments suffice, but in some cases it was necessary to extract as many as seven times.

(2) After well washing, the silk is treated for an hour with a 2 per cent. solution of sodium carbonate to 80° C., and this treatment is repeated until the extract ceases to show a reaction for ferrocyanide. Usually the operation may be repeated twice.

(3) The sample is boiled for an hour and a half with a solution of soap (25 grms. per litre).

After this it is well washed, and the nitrogen in it is estimated by Kjehldahl's process.

The operations mentioned should not be repeated a larger number of times than is absolutely necessary, because small mechanical losses are difficult to avoid.

The authors tested their process on the following samples, the amount of weighting in which was known to them:—

I. A sample of Japanese tram weighted 96 per cent. above pari.

II. A sample of Japanese tram weighted 50 per cent. above pari.

III. A sample of yellow Italian organzine weighted 53 per cent. above pari.

(*a*) In the first analysis the silk to be assayed was treated five times with hydrochloric acid, three times with sodium carbonate, once with soap solution. Amount of nitrogen in residue, 6·91 per cent.

(*b*) In the second analysis the silk was treated as follows:—four times with hydrochloric acid, twice with sodium carbonate, once with soap. Amount of nitrogen in residue, 7·08 per cent.

In the second analysis the second sodium carbonate extract still showed traces of ferrocyanide.

*Calculation of the Amount of Weighting in Assay (a.)*

The loss in weight by discharging in this case taken as 18 per cent.

| | | |
|---|---|---|
| Weight of silk employed | 0·2742 | grm. |
| Weight of nitrogen found | 0·018974 | „ |

Fibroïn = 5·455 × 0·088974 = 0·10350 grm.

$$\text{Sericin} = \frac{18 \times 0{\cdot}1035}{82} = 0{\cdot}02272$$

For the weight of the raw silk we therefore have—

| | |
|---|---|
| Fibroïn | 0·10350 |
| Sericin | 0·02272 |
| | 0·12622 |
| + 11 per cent. moisture | 0·01388 |
| Weight of raw silk | 0·14010 |

If S represents the weight of the weighted silk, and R that of raw silk, the amount of the weighting is given by the formula—

$$\text{Weighting} = \frac{S - R}{R} \times 100,$$

or in figures—

$$\frac{0{\cdot}2742 - 0{\cdot}14010}{0{\cdot}14010} \times 100 = 95{\cdot}71 \text{ per cent.}$$

The analysis of Sample II. yielded 49·66 per cent. (in place of 50 per cent.), and that of Sample III., 52·81 per cent. (in place of 53 per cent., assuming the loss in discharging to be 22 per cent.).

The authors finally point out that, although the method recommended may in some cases give results differing from the actual amount of weighting of from 5 to 10 per cent., it is sufficiently accurate for technical purposes.

*Weighting in Silk, Determination of the.* R. Gnehm and G. Schwartz. Rev. Gén. des Mat. Col. 1898, **2**.

It was noticed on examining a large number of silk samples submitted to the authors that, besides metallic substances, organic compounds had been employed for weighting. Under these conditions, the only method which would give satisfactory results was to determine the nitrogen in the silk. The method could only give good results after all other nitrogenous substances contained in the silk, such as Prussian blue, glue, ammonium phosphate, &c., had been removed, and from the quantity of nitrogen formed, the weight of the fibroïn was determined.

The first to recommend this process were (1) Saint Claire, (2) Moyret, (3) Persoz. The credit of having given the detailed description of the process of analysis is, however, due to O. Steiger and H. Grünberg.

In following the process described by these chemists the authors have carried out numerous analyses, and generally speaking, obtained results with silks dyed in colours which were very close to the actual weighting. The results obtained with black silks were, however, very far from being satisfactory; this is due to the treatment to which the silk is submitted before the determination of the nitrogen.

For the purpose of removing foreign substances, such as logwood, ferrous and ferric oxides, yellow prussiate, dyestuffs, &c., Steiger and Grünberg recommended the following method:—

(1) The silk is treated at a temperature of 66° C. with a 5 per cent. solution of HCl; this treatment is repeated until an appreciable quantity of Brazil wood enters into the solution. It is frequently necessary to repeat the operation five times in succession.

(2.) The silk is warmed for half an hour at a temperature of 80° C., with a 2 per cent. solution of $Na_2CO_3$.

Souple, or raw silk, must be boiled after this treatment for one hour in a soap solution containing 25—30 grms. per litre, after which the samples are well washed and dried.

By carefully following this method the figures obtained are frequently much too high, and surpass the true figures by 100 per cent. This difference is due to the loss of fibroïn caused by the treatment of the fibre with the HCl. In order to prove this the authors carried out a large number of experiments, of which a number are given as examples. The estimation of the nitrogen was first made by Kjeldahl's method. In order to be sure that the silk was completely discharged, the sample was treated with a tepid solution of soap.

The results of the two analyses were :—

| | Nitrogen. | |
|---|---|---|
| | 1. | 2. |
| | Per Cent. | Per Cent. |
| Silk analysed directly............ | 17·65 | 18·17 |
| " " after soaping........ | 18·75 | 17·80 |

These comparative results show that the mordants employed were completely discharged.

Weighed samples were then treated according to the method with a 5 per cent. solution of HCl at 60° C.; the loss in weight was determined. The nitrogen in the residue was also determined by Kjeldahl's method. The treatment with HCl was repeated four times for dyed silks.

(1.) After the first treatment with HCl the results obtained were:—

| | | Per Cent. |
|---|---|---|
| Loss in weight.................... | = | 2·2 |
| Nitrogen in residue.............. | = | 17·65 |

(2.) A weighed quantity of silk, after having been submitted to the first treatment, lost after the second treatment with a 5 per cent. of HCl—

| | | Per Cent. |
|---|---|---|
| Loss in weight.................... | = | 3·48 |
| Nitrogen in residue.............. | = | 17·15 |

(3.) In the third treatment the results were as follows :—

| | | Per Cent. |
|---|---|---|
| Loss in weight.................... | = | 3·35 |
| Nitrogen in residue.............. | = | 16·45 |

(4.) The sample having gone through three previous treatments, was again treated with HCl, and the following results were obtained :—

| | | Per Cent. |
|---|---|---|
| Loss in weight.................... | = | 5·0 |
| Nitrogen in residue.............. | = | 15·16 |

The total loss in weight amounts to 14·03 per cent. The experiments were repeated several times, without any different results being obtained. It is also remarked that, besides the losses caused by the chemical action of the HCl, there may be loss of a purely mechanical nature. The silk is, in fact, destroyed by the repeated action of the acid to such an extent that during the following operations of washing, filtration, &c. the material falls to pieces.

The foregoing experiments show that fibroïn is attacked by HCl of 5 per cent. strength, and the use of the acid of this strength is not permissible in estimating the amount of weighting. This fact explains the high results which the authors obtained in the determination of the weighting of the black silks.

*Soaps, Estimation of Fat and Alkali in, and Examination of the Fatty Acids by Means of the Refractometer.* C. Huggenberg. Zeits. f. offentl. Chem. **4**, 163—168; Chem. Centr. 1898, **1**, [12], 756.

THE author employs a separating burette, fitted with a stopcock at its lower part, and closed above by means of a glass stopper, which has a circular opening on its side, corresponding to a similar opening in the neck of the burette, so that by certain arrangements of the stopper, the gas-pressure in the inside can be adjusted. The apparatus holds about 160 c.c., and has three pear-shaped enlargements. The total height is 53 c.m., and topmost mark is 150 c.c. from the stop-cock. The graduations begin at 2 c.c., and are only marked on the narrow portions of the apparatus; from 60—64 c.c., 88—90 c.c., and 148—150 c.c.; the burette is marked in $\frac{1}{10}$ c.c divisions. 3·5 grms. of the soap are weighed out, dissolved by warming with 30—40 c.c. of water, and the solution is poured into the apparatus, which already contains 25 c.c. of N-sulphuric acid. The liquid is allowed to cool, and ether poured in up to the middle of the topmost bulb. After allowing to settle, the acid aqueous liquid is carefully run off, the ethereal solution washed two or three times with water, and the latter again run off. Ether is poured in up to 148 or 149 c.c., the liquid thoroughly mixed, and the total volume read off to within 0·1 c.c. The water in the hole of the stop-cock is entirely displaced by running down 1 c.c. of the ethereal solution, and then 25 or 50 c.c. of the latter are run off into a small tared flask, the ether evaporated off, and the residue dried on the water-bath to constant weight ($\frac{1}{2}$—$\frac{3}{4}$ hour). In calculating to fatty anhydride, 3·25 parts of water are deducted from every 100 parts of hydrate. The weighed fatty acids are dissolved in 10 c.c. of alcohol, and the acid number determined by titration with $\frac{1}{2}$-N alcoholic potash. For the estimation of resin acids, 6—7 grms. of soap are taken, the ethereal layer is run off down to the 2 c.c. mark, and the rosin estimated in the mixed resin and fatty acids thus obtained, by means of Twitchell's method. The alkali is estimated by titrating back with normal alkali the excess of acid in the previously run-off aqueous layer. For determining the composition of the fatty acids, the author recommends a preliminary examination by means of the butyro-refractometer, followed by a complete examination according to the known methods of fat analysis.—A. S.

*Beetroots for Seeding Purposes, Pellet's Method of Analysis of.* L. Lindet. Bull. Soc. d'Encouragement, 1898, **97**, [2], 117—119.

BEETROOTS are selected for seeding purposes on the basis of their sugar content. The roots can be taken up without injury, the foliage cut off, and then pierced transversely in an oblique direction to obtain material for analysis. They are then stored until the following spring, and when planted produce foliage and seeds.

The selection being individual, a large number of roots have to be examined in a short time, and the present article deals with some of the modifications which are used to enhance rapidity of analysis. Pellet (this Journal, 1896, 746) extracts the sugar from the pulp by instantaneous aqueous diffusion in the cold. He has simplified this process on the supposition that the specific gravities of the beetroots to be analysed are the same. The root is stabbed in such a manner that a constant weight of material is obtained (compare this Journal, 1894, 669). An automatically-measured quantity of water, containing basic lead acetate, is run on to the pulp contained in an ordinary glass, the making up to volume being thus avoided. The analysis is further hastened by reading the solutions in a "continuous tube" (*ibid.*, 1894, 669), which can be filled and emptied without removing it from the barrel of the polariscope. Following these directions, a seed factor, using two polariscopes, was able to make 350,000 analyses of beetroots in 41 days.—J. L. B.

*Brewing Sugars, On the Analysis of.* G. H. Morris. J. Fed. Inst. Brewing, 1898, **4**, 162—176.

IN the methods given in the "Text-Book of the Science of Brewing," by E. R. Moritz and the author, the calculation of the percentages of the different sugars is based on the assumption that 1 grm. of dextrose, levulose, maltose, and "gallisin" reduce 2·205, 2·037, 1·345, and 0·992 grms. respectively of copper oxide, these numbers being based on the values given by C. O'Sullivan. Heron has pointed out that the equivalent of 1 grm. of dextrose, levulose, or invert sugar is 2·26 grms. of copper oxide, thus maintaining that these three sugars have the same reducing powers.

The determination of the amount of available sugar is one of the most important estimations in the analysis of brewing sugars; and, as this depends largely on the reduction of Fehling's solution, the author has given in detail the mode of procedure. The conditions under which all determinations should be made are:—(*a*) the use of Fehling's solution, containing 34·6 grms. of recrystallised copper sulphate, 173·0 grms. of Rochelle salt, and 65 grms. of anhydrous sodium hydroxide per litre. The copper sulphate and alkaline tartrate solutions are kept separate, and mixed in equal volumes immediately before being used. (*b*) The degree of dilution of the copper solution, after taking into account the volume of the sugar added, should be 1 part of Fehling's solution to 1 part of water, 50 c.c. of the undiluted Fehling being used in each experiment, and made up to 100 c.c. (*c*) An amount of the reducing sugar should be taken which will give a weight of copper oxide lying within the limits of 0·15 to 0·35 grm. (*d*) The diluted Fehling's solution should be heated in a beaker in a boiling water-bath until the temperature is constant, then the weighed or measured solution of reducing sugar added, and the heating in the water-bath continued for exactly 12 minutes, the beaker being covered with a clock-glass during the whole period of heating. The filtration should be performed as rapidly as possible, either through a Soxhlet tube under reduced pressure, or through a carefully-folded filter paper. In the former case, the reduced cuprous oxide is oxidised to cupric oxide and then reduced to copper in a stream of hydrogen. In the latter case, the filter and its contents are burnt in the usual way, and weighed as cupric oxide. A correction must be made for the slight amount of reduction which Fehling's solution always undergoes on heating, and if filter papers be used, it is necessary to determine and allow for the copper retained in the tissue of the paper. Working under these conditions, it was found that 1 grm. of dextrose, levulose, and maltose, reduced 2·578 to 2·338, 2·310 to 2·211, 1·380 to 1·362 grms. respectively of copper oxide, the exact amount depending upon the quantity of copper reduced in the standard volume of Fehling's solution. The method of calculation previously adopted (*loc. cit.*) must, therefore, be modified, and in place of a fixed factor, one corresponding to the amount of reduction in each case be taken.

Attention is drawn to the fact that commercial invert sugar and glucose contain a certain amount of unfermentable matter, having a cupric-reducing and optical action. It has been usual to make a correction for this matter in the case of glucose, but not, however, with invert sugar. Several determinations are given, showing the properties of this unfermentable substance in both sugars.

The following is the author's scheme for the analysis of brewing sugars:—The ash is determined by burning a weighed quantity of the sugar in the usual way, and calculating the amount obtained as a percentage of the sample. The albuminoids are estimated through the medium of Kjeldahl's nitrogen process, and calculated from the ammonia obtained by the usual factors. The water is estimated by dissolving 10 grms. of the sugar in 100 c.c. of water, taking the gravity, and calculating the total solids by the 3·86 divisor. The solids so obtained require to be corrected for the higher solution density of the ash, which, according to Heron, confirmed by the author, may be taken as 8. The most convenient way of doing this is to multiply the solid matter of the 10 per cent. solution by 10, to convert it into a percentage, and then to deduct from it the percentage of ash. 100, minus the number thus obtained, gives the percentage of water. The reducing power is carried out as described above. A polarimetric reading of a 10 per cent. solution, which should be made with boiling water and allowed to stand 18 hours, is taken at 68° F. in a 200-mm. tube, and the specific rotatory power calculated, after correcting for any cane sugar which may be present. Both the values so obtained then require to be corrected for the reducing power and opticity of the unfermentable residue. The percentages of the sugars are calculated by means of the equation—

$$xD + xL = a$$

$$[\alpha]_D D + [\alpha]_D L = b \times 100$$

in which $xD$ = the gram-value of dextrose expressed in either CuO or Cu, $xL$ = the gram-value of levulose expressed in the same way, $a$ = the CuO or Cu reduced by 100 grms. of the sample, $b$ = specific rotatory power ($[\alpha]_D$) calculated on the sample.

In order to determine the amount of uninverted cane sugar, 50 c.c. of the 10 per cent. solution are digested with a small quantity of washed and pressed yeast at 125° F. for five hours (compare Ling and Baker, this Journal, 1898, 111—114). The solution is cooled, a little alumina added, made up to 55 c.c., filtered, and the rotation determined at 68° F.; the reading is increased by one-tenth, to correct for dilution, and the difference between the corrected and the original reading divided by 5·02 gives the cane sugar in solution, from which the percentage may be calculated. (5·02 is the number of divisions of the Soleil-Ventzke-Scheibler polarimeter, which a solution of 1 grm. of cane sugar in 100 c.c. of water, read in a 200-mm. tube, lost on being converted into invert sugar by yeast or acid). To estimate the unfermentable matter, 50 c.c. of the 10 per cent. solution are placed in a 100-c.c. flask and sterilised; 2 or 3 grms. of washed and pressed yeast are added, and the mixture fermented at 75° F. When fermentation is over, alumina is added and the whole made up to 100 c.c. After filtration, the copper reduction is determined on 25 c.c., and the rotation observed in a 200-mm. tube. The results are expressed on the same basis as those of the original solution, and deducted from the latter. In order to directly obtain the percentage of the unfermentable residue, a portion of the liquid may be evaporated to expel the alcohol, then made up to the original volume and the gravity taken. From this the solid matter is obtained by the 3·86 divisor, and the result multiplied by 20, in order to bring it to a percentage on the original sample. It is then necessary to subtract the percentage of albuminoids, and twice that of the ash, to get the amount of solid matter contained in the unfermented residue. This residue also contains the non-volatile products of fermentation, and these must be corrected for. According to Pasteur, the non-volatile products amount to from 4 to 5 per cent. of the sugars fermented; therefore 4 per cent. of the total sugars obtained as above is deducted, and the remainder gives approximately the true unfermentable matter. With commercial sugars it is, however, unnecessary to make this determination, it being sufficient to take the difference between the sum of the sugars, ash, albuminoids, and water, and 100 as representing the unfermentable residue. In the case of glucoses this residue includes the "gallisin" and dextrin. The constants of the former are not, in the opinion of the author, sufficiently well established to admit of direct estimation. The latter can, if necessary, be directly determined by making a second fermentation with a small quantity of cold-water malt extract, and calculating the difference between this and the ordinary fermentation as dextrin. It is only necessary to do this when partially converted products are being dealt with.

The extract is estimated from the 10 per cent. solution in the usual way.

The paper concludes with the following optical constants and cupric-reducing tables used in the preceding calculations:—

| — | Rotation in 10 per Cent. Solution at 20° C. $[\alpha]_D$ Absolute. | Reading in the 200-mm. Tube in the Soleil-Ventzke-Scheibler Polarimeter for 1 Grm. in 100 c.c. |
|---|---|---|
| | ° | Divisions. |
| Dextrin | 202·0 | 11·66 |
| Maltose | 138·0 | 7·97 |
| Cane sugar | 66·5 | 3·84 |
| Dextrose | 52·8 | 3·05 |
| Levulose | − 92·0 | − 5·31 |
| Invert sugar | − 19·6 | − 1·13 |

*Reducing values of Varying Quantities of Carbohydrates under Standard Conditions.*

DEXTROSE.

| Sugar in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. | Sugar in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. |
|---|---|---|---|---|---|
| 50 | 0·1030 | 0·1289 | 130 | 0·2585 | 0·3241 |
| 55 | 0·1134 | 0·1422 | 135 | 0·2675 | 0·3354 |
| 60 | 0·1238 | 0·1552 | 140 | 0·2762 | 0·3463 |
| 65 | 0·1342 | 0·1682 | 145 | 0·2850 | 0·3573 |
| 70 | 0·1443 | 0·1809 | 150 | 0·2934 | 0·3673 |
| 75 | 0·1543 | 0·1935 | 155 | 0·3020 | 0·3787 |
| 80 | 0·1644 | 0·2061 | 160 | 0·3103 | 0·3891 |
| 85 | 0·1740 | 0·2187 | 165 | 0·3187 | 0·3996 |
| 90 | 0·1834 | 0·2299 | 170 | 0·3268 | 0·4098 |
| 95 | 0·1930 | 0·2420 | 175 | 0·3350 | 0·4200 |
| 100 | 0·2027 | 0·2538 | 180 | 0·3431 | 0·4302 |
| 105 | 0·2123 | 0·2662 | 185 | 0·3508 | 0·4399 |
| 110 | 0·2218 | 0·2781 | 190 | 0·3590 | 0·4501 |
| 115 | 0·2313 | 0·2900 | 195 | 0·3668 | 0·4599 |
| 120 | 0·2404 | 0·3014 | 200 | 0·3745 | 0·4689 |
| 125 | 0·2496 | 0·3130 | 205 | 0·3822 | 0·4792 |

MALTOSE.

| Maltose in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. | Maltose in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. |
|---|---|---|---|---|---|
| 70 | 0·0772 | 0·0966 | 190 | 0·2072 | 0·2593 |
| 75 | 0·0826 | 0·1031 | 195 | 0·2126 | 0·2661 |
| 80 | 0·0880 | 0·1102 | 200 | 0·2180 | 0·2729 |
| 85 | 0·0934 | 0·1169 | 205 | 0·2234 | 0·2797 |
| 90 | 0·0988 | 0·1237 | 210 | 0·2288 | 0·2865 |
| 95 | 0·1042 | 0·1305 | 215 | 0·2342 | 0·2933 |
| 100 | 0·1097 | 0·1373 | 220 | 0·2397 | 0·3000 |
| 105 | 0·1151 | 0·1441 | 225 | 0·2451 | 0·3068 |
| 110 | 0·1205 | 0·1509 | 230 | 0·2505 | 0·3136 |
| 115 | 0·1259 | 0·1576 | 235 | 0·2559 | 0·3203 |
| 120 | 0·1313 | 0·1644 | 240 | 0·2613 | 0·3272 |
| 125 | 0·1367 | 0·1712 | 245 | 0·2667 | 0·3340 |
| 130 | 0·1422 | 0·1779 | 250 | 0·2722 | 0·3407 |
| 135 | 0·1476 | 0·1848 | 255 | 0·2776 | 0·3475 |
| 140 | 0·1530 | 0·1916 | 260 | 0·2830 | 0·3543 |
| 145 | 0·1584 | 0·1983 | 265 | 0·2884 | 0·3610 |
| 150 | 0·1634 | 0·2051 | 270 | 0·2938 | 0·3678 |
| 155 | 0·1692 | 0·2119 | 275 | 0·2992 | 0·3747 |
| 160 | 0·1747 | 0·2186 | 280 | 0·3047 | 0·3814 |
| 165 | 0·1801 | 0·2254 | 285 | 0·3101 | 0·3882 |
| 170 | 0·1855 | 0·2323 | 290 | 0·3155 | 0·3950 |
| 175 | 0·1909 | 0·2390 | 295 | 0·3209 | 0·4017 |
| 180 | 0·1963 | 0·2458 | 300 | 0·3264 | 0·4085 |
| 185 | 0·2017 | 0·2526 | 305 | 0·3318 | 0·4154 |

LEVULOSE.

| Sugar in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. | Sugar in Mgrms. | Cu weighed in Grms. | CuO weighed in Grms. |
|---|---|---|---|---|---|
| 50 | 0·0923 | 0·1155 | 130 | 0·2390 | 0·2997 |
| 55 | 0·1027 | 0·1287 | 135 | 0·2477 | 0·3106 |
| 60 | 0·1122 | 0·1407 | 140 | 0·2559 | 0·3209 |
| 65 | 0·1216 | 0·1524 | 145 | 0·2641 | 0·3311 |
| 70 | 0·1312 | 0·1645 | 150 | 0·2723 | 0·3409 |
| 75 | 0·1405 | 0·1761 | 155 | 0·2805 | 0·3587 |
| 80 | 0·1500 | 0·1881 | 160 | 0·2889 | 0·3622 |
| 85 | 0·1590 | 0·1993 | 165 | 0·2972 | 0·3726 |
| 90 | 0·1686 | 0·2114 | 170 | 0·3058 | 0·3828 |
| 95 | 0·1774 | 0·2224 | 175 | 0·3134 | 0·3930 |
| 100 | 0·1862 | 0·2331 | 180 | 0·3216 | 0·4032 |
| 105 | 0·1952 | 0·2447 | 185 | 0·3297 | 0·4134 |
| 110 | 0·2040 | 0·2558 | 190 | 0·3377 | 0·4234 |
| 115 | 0·2129 | 0·2669 | 195 | 0·3457 | 0·4335 |
| 120 | 0·2215 | 0·2777 | 200 | 0·3539 | 0·4431 |
| 125 | 0·2303 | 0·2887 | 205 | 0·3616 | 0·4534 |

—J. L. B.

*Starch, Comparison of the Standard Methods for the Estimation of.* H. W. Wiley and W. H. Krug. J. Amer. Chem. Soc. 1898, **20**, 253—266.

THE authors have arrived at the following conclusions from the comparative study of the standard methods which have been used in the estimation of starch, and of some modifications made by themselves:—

1. All the methods described for determining the percentage of starch by polarisation, whether with soluble starch or dextrose, cannot be relied upon to give accurate results. Although such methods are rapid, the sources of error are so great as to nullify the advantage of any time saved.

2. The methods depending on hydrolysis under steam pressure are undesirable on account of the danger of caramelisation. This, however, appears to be entirely prevented by the addition of a small quantity of an organic acid. Salicylic is preferred to lactic acid, tartaric acid being excluded on account of its optical properties. When these organic acids are added, the pentosans of the cereals and its products are hydrolysed to a considerable extent, thus increasing the reducing power and the consequent percentage of starch. A correction should be made by determining the quantity of pentose sugar present and diminishing the amount of starch in proportion.

3. The direct weighing of the starch, according to Lindet's method, after solution of the protein surrounding the starch granules by means of pepsin, has advantages for practical work, especially in starch factories. Small particles of fibre, &c., however, pass through the bolting cloth, and are consequently weighed as starch.

4. The combination of the Lindet method, in so far as the solution of the protein is concerned, and digestion by diastase, have given good results; and the authors are of opinion that a satisfactory method of starch estimation can be worked out in this direction.

5. The diastase method without pressure gives satisfactory results when the diastase is freshly prepared and used in proper quantities and at an appropriate temperature. The diastase exerts only a small, solvent action on the pentosans, and the final hydrolytic products obtained are not contaminated with any appreciable amounts of pentose sugars. The use of taka-diastase is suggested, since it can easily be prepared free from reducing sugars. The materials on which the diastase acts should be ground very finely, and extracted with ether to prevent the retarding action of fat on the enzyme. The treatment with diastase should always be repeated after boiling and cooling to about 50° C. The residue must not show any starch granules when stained under the microscope with iodine.—J. L. B.

*Pentosans. The Solubility of, in the Reagents employed in the Estimation of Starch.* W. H. Krug and H. W. Wiley. J. Amer. Chem. Soc. 1898, **20**, 266—268.

IN analytical work involving the exact determination of starch in cereals, fodder, &c., diastase should be used, and the application of any acid avoided until those bodies which may be acted upon by it, have been removed by filtration, or else a correction must be applied (see preceding abstract). The authors bring forward experiments to show that the increased amount of starch found by the use of high-pressure methods is due to the action of the organic acid on the pentosans and hemicellulose present, which results in the formation of reducing substances.

J. König (Landwirthschaftlichen Versuchs. Stat. **48**, 81) has stated that diastase acts as a solvent for pentosans and hemicelluloses. This would, if correct, render the diastase method as unreliable as the high-pressure method. The authors, however, have investigated the point, and record numbers which, in their opinion, prove that diastase has no solvent action on the pentosans, and that it furnishes a reliable means for determining the exact amount of starch in certain materials.—J. L. B.

*Molasses, Analysis of Beetroot- and Cane-.* H. Pellet. Bull. de l'Assoc. des Chim. 1898, **15**, [7], 735—708.

*Analysis of Molasses by Fermentation.*—200 grms. of molasses are diluted in about 500 c.c. of water at 40° to 50° C. The whole is poured into a flask of 1,200 to 1,500 c.c. capacity, and the alkalinity is neutralised, if necessary, and 2 grms. of sulphuric acid added, say 200 c.c. of a 10 per cent. solution; 10 grms. of yeast are mixed in a mortar with 100 c.c. of cold water, then added to the contents of the flask, and more cold water added until the whole stands at a mark at 950 c.c. The flask is kept at a temperature of 25° to 30° C.

If the molasses be acid as in the case of cane molasses, the proportion of sulphuric acid is diminished so as to have an acidity equal to 2 grms. per litre, calculated as sulphuric acid. Before the fermentation begins, the flasks are weighed. Each flask is closed by a cork carrying an S tube with a little water or a drop of mercury in the bend, so as to be able to follow the progress of the fermentation, and to see more readily when the operation is finished. To prevent frothing, it is recommended that a little oil be added. Some molasses froth abundantly. The flasks are weighed every six hours. The fermentation is complete when the flasks cease to lose weight; the supernatant liquid then becomes clear. If the flask be shaken also, to set free the excess of gas in solution, and it be then held to the ear, no sound due to disengagement of gas will be heard. The flasks are then cooled and made up to a litre, not including the oil. The contents are agitated and filtered; 500 c.c. of the filtrate are distilled to obtain the alcohol, adding again a few drops of oil to prevent frothing. At least half should be distilled off, and the remainder tested further for alcohol. The distillate is made up to 500 c.c. and tested with a hydrometer at a temperature as near 15° C. as possible, to obtain the content per 100 grms. of molasses. The operation is made in duplicate and the mean of the results taken. Discrepancies occasionally arise, possibly owing to the presence of germs inimical to fermentation. The remainder of the filtrate containing alcohol may be tested for reducing power and polarisation. If raffinose be present in sensible quantity, and Paris bakery yeast have been used, it is recognised immediately by the dextrorotatory polarisation, and, on inversion, a rotation is obtained which does not vary, or which diminishes according to the nature of the products of transformation of raffinose by fermentation. The density of the liquid may also be taken before and after fermentation, but this should be done on the liquid in the same condition as to turbidity. The fermentation lasts from 32 to 48 hours, but may vary from various causes. If treble the quantity of yeast is used, the fermentation may be complete in 12 hours, giving practically the same yield of alcohol.

If, in the estimation of the crystallised sugar, there are indications that the molasses contain raffinose, a comparative test should be made on raffinose with the yeast employed. Certain yeasts ferment raffinose partially, giving 40 to 50 per cent. of the weight in alcohol. Bottom yeast ferments it completely with sufficient time. Paris bakers' yeast does not ferment raffinose, but changes it into melibiose. To obtain comparable results it is therefore absolutely necessary to indicate the quality of the yeast employed, and its action on raffinose, and, if need be, to try also the distillery yeast, which may have other properties.

Barbet boils the solution for 10 to 15 minutes after the addition of sulphuric acid to sterilise it, and Arachequesne inverts the sugar by boiling with the requisite amount of dilute sulphuric acid. The author finds with Barbet that the reducing sugars of cane molasses may ferment and produce a notable quantity of alcohol. The unfermented reducing substances corresponded to 1·5 to 2·2 per cent. on an average of 20 to 22 per cent. of reducing substances in the molasses.

The analysis of the vinasse, which has a density of 1·033 to 1·039, is carried out as follows:—To 200 c.c. add sufficient basic acetate of lead so that no further precipitate is formed, say 120 to 180 c.c., make up to 500 c.c., agitate and filter. To 100 c.c. of the filtrate add 10 c.c. of a saturated solution of sodium carbonate, agitate, and filter. 25 to 50 c.c. of this filtrate is heated with Fehling's solution for the estimation of the reducing sugars.—L. J. de W.

*Grape or Milk Sugar, and other Bodies capable of Reducing Fehling Solution, Volumetric Method for the Estimation of.* E. Riegler. Zeits. Anal. Chem. 1898, **37**, [1], 22—25.

This process is based on the determination of the copper in a definite volume of Fehling solution, before and after reduction, by the iodometric method of de Haen.

The copper solution is prepared by dissolving 34·64 grms. of crystallised copper sulphate in 500 c.c. of water; the Rochelle salt solution, by dissolving 173 grms. of Rochelle salt and 50 grms. of sodium hydroxide in 500 c.c. of water.

To test the copper solution, once for all, 10 c.c. of each of the above solutions are run into a 200-c.c. flask, with about 100 c.c. of water and 2 c.c. of pure concentrated sulphuric acid, and the whole well shaken; 1 grm. of potassium iodide in 10 c.c. of water is then added, mixed thoroughly, and allowed to stand 10 minutes; about 3 c.c. of starch solution is added, and the liberated iodine titrated with $\frac{1}{10}$N thiosulphate solution, until the blue colour vanishes and does not reappear for five minutes. A return of the blue colour after this time is disregarded.

From the equation—

$$2CuSO_4 + 4KI = 2K_2SO_4 + Cu_2I_2 + I_2$$

it follows that each c.c. of the thiosulphate solution represents 0·00635 grm. of copper. If the solutions are properly prepared, 10 c.c. of the copper solution will require 27·8 c.c. of thiosulphate solution, representing 0·1765 grm. of copper.

For the determination of grape sugar, 10 c.c. of the copper solution, 10 c.c. of the alkaline Rochelle salt solution, and about 30 c.c. of water are placed in a 200-c.c. beaker, mixed and heated to boiling over wire gauze, and exactly 10 c.c. of a sugar solution, not over 1 per cent. strength, are run into the boiling liquid by a pipette, and the boiling maintained for a few seconds. The precipitate is allowed to subside somewhat, and then passed through a filter of thick paper, and the filter and precipitate washed thoroughly with about 80 c.c. of water. The filtrate is received into a 200-c.c. flask and titrated as already described. If $v$ be the number of c.c. of thiosulphate solution used $(27·8—v)$ 0·00635 is the amount of copper reduced by the sugar. The quantity of sugar is then found by reference to Allihn's table.

Before estimating milk sugar in milk, the albuminous bodies must be removed. This may be done with extraordinary ease and completeness with asaprol. 15 grms. of asaprol and 15 grms. of crystallised, powdered citric acid are added to 500 c.c. of water, and the solution is filtered. Of this solution, which will keep indefinitely, 15 c.c. are added in a 100-c.c. flask, to 10 c.c. of the milk, and then made up to the mark with water. The mixture is vigorously shaken, warmed to 60 c., and filtered. 20 c.c. of the clear filtrate are boiled for six minutes with 10 c.c. of the copper solution, 10 c.c. of Rochelle salt solution, and about 30 c.c. of water in a 200-c.c. beaker. After filtering and titrating, the calculation $(27·8—v)$ 0·00635 gives the copper reduced, and Soxhlet's table gives the quantity of milk sugar corresponding to it, and, in this case, the amount contained in 2 c.c. of the milk. When the copper reduced is less than 100 mgrms., it is sufficiently close to take each mgrm. of copper as equal to 0·725 mgrms. of milk sugar.

It is obvious that the precipitate on the filter may be dried and used for the determination of the fat and the total nitrogen. The results of the sugar determinations are in complete agreement with those obtained gravimetrically.
—L. J. de W.

*Chocolate, Detection of added Starch in.* — Posetto. Giorn. di Farm di Chim. &c., Jan. 1898; through Pharm. Centralhalle, 1898, **39**, [9], 152.

The author states that with a reagent composed of iodine 5 grms., potassium iodide 10 grms., dissolved in water 100 grms., decoctions of pure chocolate give only a fugitive blue coloration, not persisting for more than 12 minutes, whilst samples adulterated with starch give a marked and lasting blue colour. 2 grms. of the sample are boiled with 20 c.c. of water for two minutes and allowed to cool; another 20 c.c. of water and 0·5 c.c. of the reagent are added without shaking. The test is stated to be sufficiently delicate to detect the addition of 1 per cent. of chestnut meal; with larger quantities of added starch, the reaction is very distinct.—J. O. B.

*Meat Mixtures, &c., Estimation of Starch in.* H. Weller. Zeits. f. Untersuchung d. Nahr. u. Genussmittel, 1898, **3**, 167—171.

The following method is recommended for the quantitative estimation of starch in meats and similar articles of food. 40 grms. of the finely chopped meat are rubbed down in a mortar and placed in a 200-c.c. flask, with about 100 c.c. of distilled water, 0·3 grm. of zinc chloride, and 0·5 grm. of concentrated hydrochloric acid (sp. gr. 1·19). The flask with its contents is placed in a boiling water-bath for half-an-hour and is frequently shaken. After cooling, the flask is filled up to the mark, shaken, and the turbid fluid strained through a piece of coarse linen. 50 c.c. of this fluid are then put into a 100-c.c. flask, 0·3 grm. of zinc chloride and 0·5 grm. of concentrated hydrochloric acid are again added and the whole is once more heated to boiling. When cooled, the contents of the flask are made up to 100 c.c. with a cold saturated solution of mercuric chloride, shaken and filtered. The starch in the filtrate may then be determined by polarisation or by weighing after precipitation with absolute alcohol.—W. P. S.

*Ash, Determination of [in Drugs], as an Analytical Method.* D. B. Dott. Pharm. J. 1898, **60**, (1447), 282—283.

The further investigation of the proportion of ash in many drugs is advocated and details of manipulation in certain

cases is given. The addition of ammonium nitrate, often made as an aid to complete combustion, is not admissible where great accuracy is required, as it may alter the composition and weight of the ash. The proportion of nitre sometimes recommended to be used with a carbonate, as a flux, is dangerous with a platinum crucible, the metal being attacked and a distinct amount of platinum found in the course of the analysis. The ash of *Opium* is noteworthy from the very high percentage of sulphate it contains, probably more than any other plant-stuff. *Lobelia*, concerning which no figures have hitherto been published, gave 12·58 per cent. of ash. Genuine *saffron* gave from 5·1 to 5·4 per cent. of ash; adulterated samples as much as 31·8 per cent. One sophisticated specimen gave but 3·8 per cent. *Indigo* should give from 5 to 6 per cent.; specimens containing 19·36 and 32·07 per cent. have been met with. *Silk* is often weighted with inorganic salts, as much as 30 per cent. being sometimes found. The silk sutures used by surgeons were found to give but 1·31 per cent. of ash. The *alkaloidal salts* normally do not give more than a trace of ash, but the determination of the residue on ignition is an essential part of their examination.
—J. O. B.

### *Alcohol; The Determination of Small Quantities of.* F. G. Benedict and R. S. Norris. J. Amer. Chem. Soc. 1898, **20**, 293—302.

The following method depends on the oxidation of the alcohol by an excess of chromic acid in a concentrated sulphuric acid solution, and the determination of the excess of chromic acid by titration against a solution of ferrous ammonium sulphate, the end point being established by a few drops of standard permanganate solution. The reagents required for the determination are:—1. A solution of chromic acid in concentrated sulphuric acid, prepared by dissolving 2·2064 grms. of potassium bichromate in just enough distilled water to effect solution, and then making up to 500 c.c. with concentrated sulphuric acid, care being taken not to let the temperature rise to above 100° C. This corresponds to a solution containing 3 grms. of chromic anhydride in a litre. 2. 10 grms. of ferrous ammonium sulphate are dissolved in water and after the addition of 30 c.c. of concentrated sulphuric acid, the volume is made up to a litre. 3. A potassium permanganate solution containing 3·3 grms. of the salt in a litre of water. 4. About 2 grms. of 5 per cent. alcohol is weighed into a 50 c.c. flask and concentrated sulphuric acid slowly added, keeping the mixture cool to prevent volatilisation until the required volume is reached. Some precautions are given for weighing out small quantities of alcohol. To find the value of each solution in terms of the other, 10 or 20 c.c. of the chromic acid was poured into 100—200 c.c. of water and enough of the ferrous ammonium sulphate solution added to completely reduce it, the excess of this salt being then determined with potassium permanganate. The amount of chromic acid required to oxidise a given weight of alcohol was determined by placing 10 c.c. of chromic acid solution in a small flask together with a measured amount (about 3 c.c.) of the standard solution of alcohol. The mixture was heated to 98° C. and kept at this temperature for five minutes. Under these conditions no aldehyde was formed. After cooling it was poured into 200 c.c. of water, ferrous ammonium sulphate added from a burette until the red colour of the chromic acid had disappeared, and the excess of iron titrated back with permanganate. The value of 1 c.c. of the standard alcohol solution is thus found in terms of ferrous ammonium sulphate.

For the determination of alcohol in dilute solutions the authors roughly estimate by means of a pycnometer, and then accurately weigh a quantity containing about 0·1 grm. of alcohol into a 50 c.c. flask, making up to volume with concentrated sulphuric acid. About 5 c.c. of this solution are transferred to a flask, 20 c.c. of chromic acid added, and heated to 98° C. for five minutes. The subsequent titration is similar to that above described. The amount of alcohol per c.c. is calculated thus: the amount of ferrous ammonium sulphate solution used (after making allowance for the permanganate required for the back titration) is subtracted from the number of c.c.'s of ferrous ammonium sulphate equivalent to the chromic acid solution employed, and the result divided by the number of c.c.'s of the alcohol taken. This gives the volume of ferrous ammonium sulphate solution in c.c.'s equivalent in reducing power to 1 c.c. of the solution of alcohol. Multiplying this by the factor, which expresses the amount of alcohol equal to 1 c.c. of ferrous ammonium sulphate, the weight of alcohol in 1 c.c. of the sulphuric acid solution is found.

When it is desired to determine small quantities of alcohol in a current of air, a known volume of the filtered air is drawn through sulphuric acid. Complete absorption takes place, and the alcohol is estimated as previously described.—J. L. B.

### *Glycerophosphates, Critical Study of the Methods for the Estimation of the.* H. Imbert and J. Pagès. J. Pharm. Chim. 1898, **7**, [8], 378—384. See also this Journal, 1898, 66, 73, 78, 179, 266, 368, 386.

If a solution of glycerophosphoric acid or one of its salts be made neutral to Helianthin, the ester is then present as a monometallic salt, $(OH)_2C_3H_5O.PO(OH).OM$. This salt will require the same quantity of alkali to give a neutral reaction towards phenolphthalein that would be required to make glycerophosphoric acid neutral to Helianthin, and the acid required to make neutral to Helianthin a solution neutral to phenolphthalein is proportional to the alkali required by the reverse process. The phosphates behave in the same manner, so that in a mixture of glycerophosphate and phosphate the total phosphoric acid will be determined. The reactions of caustic soda upon solutions of monometallic phosphates and glycerophosphates in the presence of excess of calcium chloride are represented by the following equations:—

$$2PO.(OH)_2.OM + 3CaCl_2 + 4MOH = Ca_3P_2O_8 + 6MCl + 4H_2O.$$

$$2C_3H_5(OH)_2O.PO.(OH).OM + 2CaCl_2 + 2MOH = 2C_3H_5(OH)_2O.PO.O_2Ca + 4MCl + 2H_2O.$$

In the latter case the quantity of alkali required is the same as in the absence of the calcium chloride. Thus, if a mixture of monometallic phosphates and glycerophosphates be made neutral to phenolphthalein in the presence and in the absence of calcium chloride, the excess of alkali required in the first operation is proportional to the phosphoric acid present as a soluble phosphate; NaOH is equivalent to $H_3PO_4$.

When caustic soda is added to a monometallic phosphate in the presence of calcium chloride, phenolphthalein being the indicator, owing to the formation of polycalcium phosphates, more than 2 mols. are required to 1 mol. of phosphoric acid. This difficulty may be avoided by adding excess of calcium chloride, making neutral to Helianthin, then adding excess of soda and phenolphthalein. Acid is now added until the solution appears to be neutral to the phthalein, but if a drop be withdrawn and tested with a drop of phenolphthalein solution it will be found still to give the rose coloration; addition of acid must be continued until this is no longer the case. Analyses made by this method on mixtures of known composition gave satisfactory results, which, however, were somewhat uncertain when the amount of phosphate was less than 5 per cent. of the glycerophosphate.

The calcium chloride method does not permit of the determination of glycerophosphoric acid in the presence of silicates and borates.—A. C. W.

### *Mustard Oil, The Estimation of.* E. Haselhoff. Zeits. f. Untersuch. der Nahr. und Genussm. 1898, **4**, 235—239.

The first part of this paper deals with a number of investigations which were made to ascertain whether certain methods for the estimation of mustard oil were reliable. The results of these experiments showed that the methods of Forster, Schlicht, and Passon (this Journal, 1892, 779) were equally good, and sufficiently accurate for ordinary purposes.

The author also estimated the mustard oil in rape seed and in rape-seed cake by the above methods. It was found

that the heating of the seeds in the manufacture of the cakes did not increase the amount of oil, as Schuster and Mecke state (Chem. Zeit. 1892, **16**, 1954). The amount of mustard oil found in rape seeds varied from 0·184 to 0·348 per cent., and in rape-seed cake from 0·155 to 0·357 per cent.
—W. P. S.

*Cantharides, Assay of.* H. G. Greenish and H. Wilson. Pharm. J. 1898, **60**, [144], 256.

*Determination of Total Cantharidin.*—20 grms. of cantharides in No. 40 powder are mixed in a small mortar with 25 c.c. of a menstruum composed of glacial acetic acid, 1 vol.; rectified spirit, 2 vols.; chloroform, 3 vols. The moistened mass is covered for at least an hour, and then allowed to dry spontaneously, or at a slightly raised temperature. The dried mass is then extracted with boiling chloroform in a Soxhlet tube. After extraction, the chloroform solution is mixed in a separator with a little water, and the acetic acid nearly, but not quite, neutralised with solution of potash. The mixture is well shaken, the chloroformic layer separated and evaporated. The residue thus obtained is washed with petroleum spirit, which removes the fat, with some cantharidin, and leaves crystals of crude cantharidin, which are dried and cautiously washed with absolute alcohol until white, the alcoholic washings being evaporated and the residue reserved. The cantharidin removed by the fatty solution of petroleum spirit is thus recovered; 20 c.c. of 10 per cent. caustic potash solution are added, and the mixture warmed until the fat is saponified. The soap solution is transferred to a separator, covered with a layer of petroleum spirit, and acidulated with hydrochloric acid. The fatty acids rise to the surface, and are dissolved in the petroleum spirit, leaving the cantharidin in the lower aqueous layer, which is quickly separated and shaken out with chloroform; the residue of the alcoholic washings are added to this chloroform solution, which is then washed with lime water containing an excess of calcium hydroxide suspended in it, and the chloroform caused to separate by the addition of salt solution. The aqueous solution containing the cantharidin as a soluble lime compound, is filtered, acidified with hydrochloric acid, and shaken out with chloroform; on evaporation, crystals of pure cantharidin are obtained, which, added to those at first separated, are weighed after drying in a desiccator.

*The free cantharidin* is determined by a similar process to that given above for total cantharidin, omitting the preliminary treatment with acetic acid.

*The combined cantharidin* is deduced from the difference of the total and free active principle. The amounts of total cantharidin found in two samples of beetles by this method, were 0·56 and 0·68 per cent.

The communication contains a complete series of determinations of the amount of cantharidin in the various official pharmaceutical preparations.—J. O. B.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Coal-Gas: Alleged Injury to Vegetation through Leakages into the Soil.* O. Pfeiffer. J. f. Gasbeleucht. **41**, 137—139.

The saturation of the soil with coal-gas, through leakages or breaks in the pipes, is often blamed for the sickliness and withering of trees in towns. Official investigations in Berlin, in 1871, and later researches of Bohm, in which trees and shrubs were artificially exposed to the influence of coal-gas, showed that injurious effects were produced, and that the evil effects proceeded from absorption at the rootlets. Freytag, on the other hand, found that neither hydrogen, paraffins, nor alkylenes affected vegetation, though small amounts of tar-products, especially of phenol vapour, were actively injurious; and Poselger maintained that coal-gas was not at all deleterious to plant life. Many other causes may certainly contribute to the low vitality of the trees of towns, and the question can hardly be satisfactorily settled in any particular case unless some means be found of detecting the presence of coal-gas in the soil where the sickening of trees leads it to be suspected. The author has experimented with a sample of soil which had been impregnated with coal-gas by passing the gas over it for 10 days in a 4-inch gas-pipe. Neither ammonia nor cyanogen could be detected, and though diazobenzene-sulphonic acid gave indications of phenolic bodies, yet investigation showed that similar results were obtained from many of the products of decay of animal and vegetable substances. Ultimately, the determination of the alkylenes by absorption with bromine was found to give satisfactory results. 500 grms. of the earth were soaked for several hours in 750 c.c. of water, and filtered till quite clear. To 100 c.c. of the filtrate, acidified with sulphuric acid, a solution of bromine (saturated bromine water diluted to 10 times its original bulk) was added from a burette till the yellow colour of the bromine was persistent (a few drops of carbon bisulphide in the liquid make this point more exactly determinable). By standardising the bromine water solution before use, successive titrations can be compared with one another. Blank experiments with distilled water and with fresh earth showed that ½ c.c. of bromine was enough in all cases to tint the liquid; and titration of the gas-impregnated earth, after many months' exposure to air and drying, showed that it still retained the whole of the alkylenes.—J. T. D.

*Mutual Replacement of Two Acids, The Causes of the.* A. Colson. Comptes Rend. 1898, **126**, [11], 831—833.

The author has tried the action of dried hydrogen sulphide on dried silver phosphate, and comes to the conclusion that temperature plays an important part in the reactions. This, he considers, is at variance with the laws of Berthollet.—J. S.

*Ammonium Nitrate and Ammonia, Balanced Ratios between.* B. Kuriloff. Zeits. physik. Chem. **25**, 107—111; Chem. Centr. 1898, **1**, [11], 655.

If a current of dry ammonia gas be passed over solid ammonium nitrate, a liquefaction of the salt ensues. The vapour pressure of the ammonia above the liquid, decreases continually at 0° C., with absorption of ammonia, and remains constant at 360 mm., when only 0·14 mol. of ammonia is present to 1 mol. of ammonium nitrate. At − 10°·5 C., the constant vapour pressure is 240 c.c. The constant dissociation pressure corresponds to a saturated solution of an ammonia compound in liquid ammonia. The composition of the liquid was determined at temperatures between 109°·8 and − 60° C., and the results were set forth in the form of curves. Between − 40° and + 160° C., the curve indicates the saturated solutions of ammonium nitrate in liquid ammonia. At − 40° C., the solid ammonium nitrate disappears, and in its place occurs a compound of the probable formula $NH_4NO_3.3NH_3$. The latter probably remains permanent until the temperature sinks to − 80° C., when it is replaced by solid ammonia.—A. S.

*Nascent Hydrogen.* D. Tomassi. Monit. Scient. 1898, **51**, [675], 182—183.

The author considers that the reductions caused by hydrogen at the moment of its liberation from its compounds are wrongly attributed to its being in an allotropic condition, such as is usually connoted by the term "nascent"; for, in that case, he argues, the same reactions ought always to follow whatever the origin of the gas; but this is not borne out by experiment. Thus, silver chloride, bromide, or iodide, suspended in water acidulated with sulphuric acid, can be reduced by the hydrogen liberated from water by electrolysis, but show no signs of reduction when the water is decomposed with sodium amalgam. Or, if a solution of potassium chlorate be acidulated with sulphuric acid, and zinc added, the chlorate is reduced to chloride; whereas, if sodium amalgam be added, no reduction takes place. Nor does sodium amalgam bring about the reduction of chloric acid or of the chlorates of sodium, barium, copper, lead, or mercury. In the case of potassium perchlorate, none of the usual reducing agents have any effect; zinc or magnesium with sulphuric acid, or zinc with potash or soda, or in a boiling solution of copper sulphate, all failing to bring about reduction; but, on the other hand, this is readily effected by sodium hydrosulphite—a compound from which hydrogen is not liberated. Similarly, a solution of nickel sulphate, to which potash and potassium cyanide have been added, acquires a reddish tint on the addition of zinc, whilst hydrogen is liberated; but if magnesium, or a magnesium-platinum couple, replace the zinc, the red colour is no longer produced, although hydrogen is still liberated. Kern found (Bull. Soc. Chim. **26**, 338) that by the action of magnesium on ferric chloride, ferric hydroxide was produced, and this fact the author confirms, with the addition that he obtains the same result by using sodium amalgam instead of magnesium. According to Stahlschmidt, nascent hydrogen derived from the decomposition of water by zinc dust reduces potassium nitrate to nitrite, reduces iodides and iodates, but does not reduce chlorates; and De Wilde has established the fact that sodium amalgam reduces potassium bromate, but is without action on the chlorate.

On these and similar facts the author bases his opinion that the reducing power of nascent hydrogen varies according to the chemical reaction by which the hydrogen was produced, and he considers that if the gas has a greater affinity in the nascent than in the ordinary condition, this is entirely due to the hydrogen at the moments of its liberation from a compound being accompanied by the heat produced during that liberation. Hence, if nascent hydrogen be represented by the symbol $H + a$ (in which $a$ denotes this amount of heat), the value of $a$ would vary with each chemical reaction, and, as a general rule, the reducing power of nascent hydrogen would be proportional to that value, provided that the reaction between the hydrogen and the substance to be reduced, could once be started. There are certain cases, however, in which the reduction is due, not to the hydrogen, but to the metal which served to generate it. The reduction of potassium chlorate by means of sulphuric acid and zinc, or by electrolysis of its solution with a zinc anode, is an instance of this. If such a solution be electrolysed with both electrodes of platinum, oxidation occurs at the anode, with the formation of perchlorate, whilst no trace of chloride is found at the cathode; but if a zinc anode be used, chloride is formed at the anode, but not at the cathode. From this the author concludes that the reduction in this case must be attributed, not to the hydrogen, but to the zinc uniting with the oxygen of the chlorate in accordance with the equation $KClO_3 + 3Zn = KCl + 3ZnO$.
—C. A. M.

*Palladium Dichloride, Action of Carbon Monoxide on.* E. Fink. Comptes Rend. 1898, **126**, 646—648. (See under XXIII., pages 484 and 490.)

When anhydrous palladium dichloride, mixed with finely divided asbestos, is heated to 260° C. in a current of dry carbon monoxide, a sublimate, partly yellowish, partly brownish in colour, is obtained. The crude product contains Pd, CO, and Cl in varying and uncertain proportions. It is more or less crystalline, and by fractional solution in carbon tetrachloride yields the following compounds:—

(i.) *Yellow needles*, melting at 132° C., and having the composition expressed by the formula $C_3O_3Pd_2Cl_4$. The compound is decomposed by water into CO, $2CO_2$, $Pd_2$, and 4HCl. When heated to 263° C., CO is evolved, and a brownish liquid remains, which solidifies, on cooling, to a mass of *yellowish-red* needles, of the composition $COPdCl_2$. This substance melts at 197° C., and in contact with water is decomposed into Pd, $CO_2$, and 2HCl.

(ii.) *Colourless needles*, $C_2O_2Pd_2Cl_2$, which melt at 142° C., and, when heated to about 218° C., evolve CO and yield the compound $C_3O_3Pd_2Cl_4$ already described under (i.).—H. T. P.

*Chromium Silicide, A New.* C. Zettel. Comptes Rend. 1898, **126**, 833—835.

140 grms. of copper and 140 grms. of aluminium are fused in a Perrot's furnace, in a crucible of a refractory earth, and 200 grms. of chromium sesquioxide are then added in small portions at a time. After having been kept fused and stirred with an agitator made of a refractory earth, for half an hour, an additional small quantity of aluminium filings is added. A metallic ingot is obtained containing chromium, copper, aluminium, and the well crystallised compound $SiCr_3$. This compound is very stable and resists the action of acids, with the exception of hydrofluoric acid. It can consequently be separated from the fused mass by treatment with *aqua regia*.—J. S.

*Barium Sulphide, Crystallised; The Preparation and Properties of.* A. Mourlot. Comptes Rend. 1898, **126**, 643—646.

The author has obtained the sulphides of Ba, Sr, and Ca in a crystalline form by fusing (*a*) the amorphous sulphides, or (*b*) the corresponding sulphates mixed with carbon, in an electric furnace, using a current of 500 ampères at 50 volts. The barium compound, which is now described, forms colourless crystals, transparent in thin pieces, but appearing blackish in larger fragments, owing to the presence of traces of carbon. The crystals have no action on polarised light; they exhibit two cleavage planes at right angles to each other, and appear to be built up of cubes. The density (when powdered) ranges from 4·25 to 4·30. The hardness of the crystals is not very great, being inferior to that of glass.

In chemical properties the crystalline sulphide resembles the amorphous variety; but on the whole is more resistant, especially towards atmospheric influences.—H. T. P.

*Glycerin, Oxidation of, by the Bacteria of Sorbose.* G. Bertrand. Comptes Rend. 1898, **126**, [11], 842—844.

By cultivating the bacteria of sorbose in a nutritive liquid containing glycerin, it was noticed that the liquid rapidly acquired the property of reducing Fehling's solution in the cold. On treating the reducing liquid with phenyl-hydrazine, an osazone was obtained which possessed all the properties of glycerosazone. This might have been formed either from glyceric aldehyde or dioxyacetone or from a mixture of both, but it was found that by the action of hydroxylamine an oxime was produced, which was identical with that obtained by Piloty (Ber. **30**, [12], 1656) from synthetical dioxyacetone, and hence it follows that the bacteria remove two atoms of hydrogen from glycerin, forming dioxyacetone.—J. S.

*Seeds, Note on the Influence of very Low Temperatures on the Germinative Power of.* H. T. Brown and F. Escombe. Proc. Royal Soc. 1897, **62**, 160—165.

Romanes (Proc. Royal Soc. **57**, 335) showed that neither a high vacuum, nor subsequent prolonged exposure to oxygen, hydrogen, nitrogen, carbon monoxide, carbon dioxide, hydrogen sulphide, aqueous vapour, and the vapour of ether and chloroform, exerted much, if any, effect on the subsequent germinative power of the seeds experimented on. These conditions excluded anything like respiration by ordinary gaseous exchange, but they did not preclude the possibility of chemical interactions within the protoplasm.

The authors believed that more evidence would be gained on these points if seeds could be submitted to a temperature below which ordinary chemical reactions take place, thus eliminating any interaction between the constituents of the protoplasm. To this end the seeds representative of the *Gramineæ, Cucurbitaceæ, Leguminosæ, Geraniaceæ, Compositæ, Umbelliferæ, Convolvulaceæ, and Liliaceæ*, were placed in thin glass tubes, which were slowly cooled and immersed in liquid air so as to submit the seeds for 110 consecutive hours to a temperature of −183° to −192° C. The seeds which had been previously air-dried contained from 10 to 12 per cent. of moisture. After the above treatment they were slowly and carefully thawed, and their germinative power was then compared with control experiments made on other portions of the untreated seed. Their germinative power showed no appreciable difference from that of the controls, and the resulting plants were equally healthy in the two cases. The authors consider that processes of autoxidation, and any internal change in the protoplasts are impossible at such low temperatures, and they conclude that the protoplasm of resting seeds exists in an absolutely inert state, devoid of any trace of metabolic activity, and yet conserving the potentiality of life.—J. L. B.

### *Sorbite, The Action of Mycoderma Vini on.* G. Bertrand. Comptes Rend. 1898, **126**, 653—656.

The author has formerly shown that sorbose does not pre-exist in the juice of the mountain-ash berry, but is formed by the oxidation of the sorbite originally present. This change is brought about by a distinct oxidising bacterium; and upon the accidental presence or absence of this organism, success or failure depends, in the preparation of sorbose. (As a rule the organism comes from vinegar works, and is disseminated by the agency of flies.) Excellent results may be uniformly obtained by addition of a pure culture of the oxidising bacterium to sterilised sorbite juice.

It was also shown that certain other ferments, particularly *Mycoderma vini* and moulds, *Penicillium glaucum*, which readily develop in organic liquids on exposure to air, exert a destructive action on sorbite and produce no sorbose.

Recently, however, Matrot (Comptes Rend. **125**, 874) has stated that the conversion of sorbite into sorbose may be readily effected by the aid of a *mycoderma*—which he obtained from sorbite juice, as also from wine—and regarding the identity of which with *M. vini* there is, practically, no doubt. The author has, therefore, made fresh experiments with pure cultures of *M. vini*, working under strictly aseptic conditions; and has obtained results which entirely confirm his original conclusions.

An explanation for these discrepant results is suggested by the circumstance that Matrot recommends dishes as receptacles for the cultures—obviously the risk of infection with air-borne germs would be very great.—H. T. P.

### *Organic Bases, Compounds of, with various Salts.* D. Tombeck. Comptes Rend. 1898, **126**, [13], 967—970.

When aniline in excess is added to various sulphates, nitrates, and acetates, crystalline compounds are formed which in general contain 2 mols. of aniline and 1 mol. of the salt. Cadmium, zinc, magnesium, and copper salts were investigated. Two series of compounds were formed from the sulphates of nickel and cobalt, one containing 6 mols. of aniline and the other four. The toluidines form similar compounds.—J. S.

### *Goupia Tomentosa, The Volatile Constituents of the Wood of.* W. R. Dunstan and T. A. Henry. J. Chem. Soc., April 1898, 226—229.

A sample of fine shavings from the wood of *Goupia tomentosa* was allowed to stand under water for 12 hours, and was then steam distilled. The distillate was concentrated by steam-distillation, and the process repeated until fatty particles began to separate from the distillate. The latter was then extracted with ether, and the ethereal solution dried with calcium chloride and distilled. The dark-coloured oil thus obtained became semi-solid on cooling; it was dissolved in boiling alcohol, decolorised with animal charcoal, and then allowed to cool. A colourless, fatty substance was deposited, which, on purification, was obtained in small needles, melting at 45° C. The pure substance, distilled under reduced pressure, was slowly dissolved by alkalis and reprecipitated by acids, and, on examination, was found to consist of lauric acid. The aqueous layer left after extraction with ether, was made strongly alkaline with soda, evaporated to dryness over the water-bath, taken up with dilute sulphuric acid, and the oil thus separated, removed by ether. The ethereal solution was dried with calcium chloride, the ether distilled off, and the residue fractionated several times. The resulting fractions boiled respectively at about 46°, 150°, and 206° C. The first fraction, which was very small, had a pungent odour, and its aqueous solution reduced silver nitrate and mercuric chloride. It probably contained formic acid, but there was not enough obtained for complete identification. The second fraction was found to consist of a mixture of isovaleric and caproic acids. The fraction boiling at 206° C. constituted about 60 per cent. of the whole, and consisted mainly of normal caproic acid, but also contained some succinic acid.—A. S.

### *Hydrogen and Helium, Liquefaction of.* Chem. and Druggist, May 14, 1898, 799.

On Tuesday, May 10, Prof. Dewar succeeded in producing liquefied hydrogen at the Royal Institution, and exhibited it to Lord Rayleigh. The boiling-point of the liquid is about 240° C. Some conception of the degree of cold attained, may be gathered from the fact that a tube closed at the lower end, when immersed in the liquid, was almost instantaneously filled with solid air. The density of the liquid far exceeds that arrived at by Olzewski, which is the best support that Dewar has yet given to his assertion that Olzewski's figures were theoretical. Helium was also liquefied by the use of liquid hydrogen. Its boiling-point appears to lie not very far from that of hydrogen itself.

### *Barley Straw, The Carbohydrates of.* C. F. Cross, E. J. Bevan, and C. Smith. Proc. Chem. Soc. 1898, [193], 96.

The authors have continued their investigations of growing barley, extending their observations to the plant under the artificial conditions induced by the removal of the ears at the flowering stage. The diversion of the energies of the plant from its normal seed-bearing function proved to be without influence upon the proportion of furfural-yielding carbohydrates to total carbohydrates. The constancy of this ratio has now been established under the widest possible variations of the conditions of growth, natural and artificial, confirming the conclusions that the furfuroids are assimilated with their special constitutional characters and not formed from hexoses by subsequent processes of oxidation.

By the suppression of the seminal function of the plant, the maturing of the straw was virtually arrested, and this is shown by the chemical constants determined in the straw grown up to the harvesting period after removal of the ear, compared with the straw grown under normal conditions. The cellulose and "permanent tissue" are 30 and 10 per cent. respectively in excess in the normal straw, and conversely the artificial product is much more readily hydrolysed by acids, and the resulting solutions (after neutralisation) are fermented by yeast to a much greater extent.

### *Ammonia and of Hydrogen Chloride, The Drying of.* H. Brereton Baker. Proc. Chem. Soc. 1898, [193], 99—100.

In a recent paper (this Journal, 1894, 767, and 1898, 283), Gutmann has described experiments on the action of these gases when dried. He comes to the conclusion that the drying of both gases by phosphorus pentoxide is an impossibility, because they are absorbed by the drying agent in a shorter time than is necessary to dry them. The author has repeated the experiments, and finds that this absorption of the gases, when properly purified and dried by lime and sulphuric acid respectively, only takes place when the phosphorus pentoxide is impure. When the oxide contains any quantity of metaphosphoric acid, the absorption takes place as rapidly as, and with similar results to, those described by Gutmann. From the construction of the apparatus, as well as from the similarity of the results, it is concluded that moisture was admitted whilst the phosphorus pentoxide was present, and that the difference between his results and those of the author published in 1894 is readily explained as due to the production of metaphosphoric acid.

With regard to the vapour density of ammonium chloride, it is pointed out that Gutmann's low results are probably due to his having collected the air, driven out of the Victor Meyer apparatus, over water and sulphuric acid instead of over boiled mercury, which was used by the author. A re-determination of the vapour density of dried ammonium chloride by Dumas' method is published in the paper. When the bulb was protected from the outer air by a phosphorus pentoxide tube terminated by a long capillary, the density of 28·8 was obtained from the dried ammonium chloride, confirming the author's conclusion, based on six experiments by Victor Meyer's method, that dried ammonium chloride does not dissociate at 350°.

# New Books.

Methods for the Analysis of Ores, Pig Iron, and Steel, in use at the Laboratories of Iron and Steel Works in the Region about Pittsburg, Pa. Together with an Appendix containing various special Methods of Analysis of Ores and Furnace Products. Contributed by the Chemists in Charge, and edited by a Committee of the Chemical Section, Engineers' Society of Western Pennsylvania. Chemical Publishing Co., Easton, Pa. 1898. In paper covers, price $0.75. Cloth, $1.00.

8vo volume, containing preface, table of contents, subject-matter filling 131 pages, and an alphabetical index. The text is illustrated with three engravings representing forms of apparatus used. The methods used in the laboratories of 16 different iron and steel works, representing the practices of the chief iron and steel works in the region of Pittsburg and Western Pennsylvania, are described. An appendix is given referring to :—1. Analyses of Blast-Furnace Cinders. 2. Barium Hydroxide as an Absorbent in Carbon Determinations. 3. An Improvement in the Zinc Reduction for the Determination of Iron or Phosphorus. 4. Some Evolution Methods for Sulphur in Iron and Steel. 5. The Determination of Chromium. 6. The Analysis of Mill and Puddle Cinders. 7. The Complete Analysis of Chrome Ore.

Les Nouveautés Chimiques pour 1898. Nouveaux Appareils de Laboratoires, Méthodes nouvelles de Recherches appliquées à la Science et à l'Industrie. Par Camille Poulenc, D. ès Sc. J. B. Baillière et Fils, 19, Rue Hautefeuille. Poulenc Frères, 29, Rue Vieille-du-Temple, et Boul. Saint-Germain 122. Price, 4 frs.

Small 8vo volume in paper cover, containing preface, subject-matter filling 233 pages, table of contents, and alphabetical index. There are interspersed in the text 117 woodcuts, illustrative of apparatus. The volume is devoted to descriptions, mostly illustrated, of the following branches and apparatus, &c. :—I. *Applications generally of Chemistry and Physics.*—Measuring Temperatures. Heating Apparatus. Illumination. Apparatus for the Production of Acetylene. Apparatus for Determining Densities, and also for Physical Constants. II. *Various Forms of Laboratory Apparatus.*—Miscellaneous. Extraction Apparatus. Apparatus for Distillations, Filtration, for Creating a Vacuum, and for Air Compression. Apparatus for various Mechanical Laboratory Purposes. III. *Chemical Analysis.*—General Analytical Apparatus. Apparatus for Gas Analysis, for the Analysis of Liquids and Solids. IV. Electricity. V. Bacteriology.

Technical Mycology: The Utilisation of Micro-Organisms in the Arts and Manufactures. A Practical Handbook on Fermentation and Fermentative Processes for the Use of Brewers and Distillers, Analysts, Technical and Agricultural Chemists, Pharmacists, and all interested in the Industries dependent on Fermentation. By Dr. Franz Lafar, Professor of Fermenation-Physiology and Bacteriology in the Imperial Technical High School, Vienna. With an Introduction by Dr. Emil Chr. Hansen. Translated by Charles T. C. Salter. In Two Volumes. Vol. I. Schizomycetic Fermentation. Chas. Griffin and Co., Ltd., Exeter Street, Strand, London. 1898. Price 15s.

In the Preface, Dr. Hansen points out that the Physiology of Fermentation and Technical Bacteriology has called into existence within the last few years a number of stations and laboratories on the Continent for the development of those branches of industry wherein micro-organisms play important parts. Formerly, Chemistry exercised undisputed sway over the whole of this realm, but now Biology has won for itself a co-ordinate position therein—a fact now being recognised (although not yet to an adequate extent) in the filling up of professorships at the various technical high schools. The present volume contains 405 pages of subject-matter, illustrated by 90 woodcuts. The text is subdivided as follows:—I. Theory of Spontaneous Generation. II. Theories of Fermentation. III. Organisms of Fermentation. Division I. Schizomycetic Fermentation. Section I. General Morphology and Physiology of the Schizomycetes. I. Form and Dimensions. II. Structure and Constitution of the Bacterial Cell. III. Power of Independent Movement in Bacteria. IV. Vegetative Reproduction by Fission. V. Permanent (Reproductive) Forms or Spores. VI. Germination of the Endospore. Section II. General Biology and Classification of Bacteria. VII. Bacteria under the Action of Physical Agencies. VIII. Bacteria in their relation to one another. IX. Classification of Bacteria. Section III. Principles of Sterilisation and Pure Cultivation. X. Methods of Sterilisation. XI. Methods of Pure Culture. Section IV. XII. Chromoparous Bacteria producing Red and Yellow Colouring Matters. XIII. Purple Bacteria, and their behaviour towards Light. XIV. Chromoparous Bacteria producing Blue, Green, and Violet Colouring Matters. XV. Photogenic Bacteria. XVI. Thermogenic Bacteria. Section V. Heat-resisting Bacteria: their place in Nature, and their importance in the Fermentation and Food-Stuff Industries. XVII. Bacillus Subtilis and its congeners. XVIII. Butyric Acid Fermentation and Allied Decomposition Processes XIX. Preservation of Milk. XX. Preservation of Meat, Eggs, Vegetables, and Fruit. Section VI. Lactic Fermentation and Allied Decompositions. XXI. General Characteristics. XXII. Production of Optically Active Organic Compounds by Fermentation. XXIII. Artificial Souring of Cream. XXIV. Coagulation (Curdling) of Milk. XXV. Lactic Acid Bacteria in Distilling, Brewing, and Vinification. XXVI. Lactic Acid Bacteria in the Preparation of Fodder. XXVII. Part played by Bacteria in Tanning. Section VII. Formation of Mucus, and Allied Phenomena of Decomposition. XXVIII. Importance of Bacteria in the Manufacture of Sugar. XXIX. Ropiness in Milk, Wine, Beer, and other Liquids. Section VIII. Decompositions and Transformations of Organic Nitrogenous Compounds. XXX. Phenomena of Putrefaction. XXXI. Fermentation of Cheese, and Allied Decompositions. XXXII. Fermentation of Urea, Uric Acid, and Hippuric Acid. XXXIII. Fixation of Free Nitrogen by Bacteria. Section IX. Oxidising Fermentations. XXXIV. The Iron Bacteria. XXXV. Sulphur Bacteria. XXXVI. Nitrifying Bacteria. XXXVII. Acetic Fermentation. XXXVIII. The Oxydases.

Leather Industries. Laboratory Book on Analytical and Experimental Methods. By H. R. Procter, F.I.C., F.C.S., Professor of Leather Manufacture at the Yorkshire College, Leeds, &c. E. and F. N. Spon, Ltd., 125, Strand, London. 1898. Price 9s. Spon and Chamberlain, 12, Cortlandt Street, New York.

In the introduction it is significantly stated that the "Leather manufacture has long been conducted by 'rule of thumb,' rather than by the more exact methods of modern science, not so much from any preference of manufacturers for the former, as because, from the inherent difficulties of the subject, the latter condition has been scarcely possible."

In the preface the author states that, "With scarcely an exception," the analytical methods he has described in this work "have been carefully tested" in his own laboratory, "and found satisfactory, and in many instances they have been modified to suit the special purposes for which they are employed."

The subject-matter of this work fills 276 pages, which are also illustrated by 29 woodcuts or numerous tables. The subjects treated of are as follows, and they form the themes of the various chapters of the book :—I. General Methods. II. Examination of Commercial Acids, &c. III. Water Analysis. IV. Depilitants. V. The Analysis of Used Lime Liquors. VI. Materials used in Curing, Bating, &c. VII. Chemistry of Tannins and their Derivatives. General Methods of Examining Tannins. VIII. Qualitative Recognition of Tanning Materials. IX. Analysis of Tanning Materials, Historical Notice. X. Sampling, Grinding, and Extraction. XI. Hide Powder Filter Method. XII. The Loewenthal Volumetric Method. XIII. Analysis of Used Liquors and Spent Tans. XIV. Estimation of Colour in Tanning Materials. XV. Materials used in Alum and Chrome Tannages. XVI. Detection and Estimation of

Glucose. XVII. Analysis of Soaps. XVIII. Oils and Fats. A. Determination of Total Fatty Matter and Non-Fats. B. Physical Examination of Fats. C. Quantitative Analysis of Fats. D. Special Application of Fat Analysis. XIX. The Kjeldahl Method of Nitrogen Determination. XX. The Analysis of Leather. XXI. Dyes and Dyestuffs. XXII. Use of the Microscope. XXIII. Microscopic Structure of Skin. XXIV. Bacteriology and Mycology.

The Theory and Practice of Electrolytic Methods of Analysis. By Bernhard Neumann, Ph.D., Assistant Lecturer on Metallurgy at the Technical School at Aachen. Translated by John B. C. Kershaw. Whittaker and Co., 2, White Hart Street, Paternoster Square, London; 66, Fifth Avenue, New York. 1898. Price 10*s.* 6*d.*

Small 8vo volume, containing translator's preface, author's preface, table of contents, and subject-matter filling 245 pages, an appendix giving in tabulated form the "Theoretical percentage of the Metallic Elements in certain Metallic Salts," and alphabetical indexes of authors' names and of subject-matter. The text is illustrated with 37 wood engravings, and may be classified as treating of the following subjects:—I. Theory of Electrolysis. II. Measuring and Regulating the Current. A. Current Measurement. B. Regulation of the Current. III. The Electrolytic Procedure. A. Introductory. B. Deposition of the Metals from Solutions of pure Salts. C. Separation of the Metals. D. Practical Examples. Appendix. Theoretical Percentage of the Metallic Elements in certain Metallic Salts.

Notes on Observations; being an Outline of the Methods used for Determining the Meaning and Value of Quantitative Observations and Experiments in Physics and Chemistry, and for Reducing the Results obtained. By Sydney Lupton, M.A. Macmillan and Co., Limited, London; The Macmillan Company, New York. 1898. Price 3*s.* 6*d.*

This little work contains preface, table of contents, subject-matter covering 121 pages, three tables, and the alphabetical index. An idea of the nature of the treatise may be derived from the contents, which are classified as follows:—I. Ideas. II. Reasoning. III. Fallacies. IV. Laws of Nature. V. Cause and Effect. VI. Observation and Experiment. VII. Units and Dimensions. VIII. Averages. IX. Differences. X. Interpolation. XI. Mensuration. XII. Use of Tables. XIII. Errors. XIV. Means. XV. Law of the Frequency of Errors. XVI. Weight of Observations. XVII. Method of Least Squares. XVIII. Conditioned Equations. XIX. General Formulæ. XX. The Deductive Method. XXI. Expression of Results by Graphical Methods. XXII. Expression of Results by Empirical Formulæ.

Radiography and the "X" Rays in Practice and Theory; with Constructional and Manipulatory Details. By S. R. Bottone. Whittaker and Co., 2, White Hart Street, Paternoster Square, London; 66, Fifth Avenue, New York. 1898. Price 3*s.*

Contains preface, table of contents, list of illustrations, 47 in all, and subject-matter, 172 pages. An alphabetical index of subjects concludes the work. There are ten chapters, and their several themes are as follows:—I. Action of Light, &c., on Certain Salts of Silver. II. Effects of a Partial Vacuum on Electrical Discharges. III. The Induction Coil. IV. Choice of Source of Current. V. Crookes' Tubes: Stands. VI. The Wimshurst's Machine. VII. The Holtz Machine. VIII. Fluorescent Screens. IX. General Manipulatory Details. X. Theoretical Considerations.

Practical Radiography. A Handbook of the Applications of the "X" Rays; with many Illustrations. By A. W. Isenthal and H. Snowden Ward. Second Edition. Dawbarn and Ward, Ltd., 6, Farringdon Avenue, London, E.C. 1898. Price 2*s.* 6*d.*

8vo volume containing introduction, 157 pages of subject-matter, and a bibliographic list. The text is illustrated by 51 woodcuts and numerous photographs, and treats of the following leading subjects:—I. Historical Review. II. Apparatus. III. Practical Radiography, Electrical. IV. Practical Radiography, Photographic. V. Practical Radiography, Medical. VI. Diagnostical Applications of Radiography. VII. Therapeutic Value of the "X" Rays. VIII. General Applications and Probabilities. IX. Theory of the "X" Rays.

# Trade Report.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

### France.

*Duty on Boracic Acid.*

*Board of Trade J., May* 1898, 567.

A despatch has been received from H.M. Ambassador at Paris, transmitting extract from the *Journal Officiel*, containing law, establishing a duty on boracic acid, which has hitherto entered France free of duty, as follows:—

| —— | | General Tariff. | Minimum Tariff. |
|---|---|---|---|
| Natural Tuscan boracic acid, containing 15 per cent. or more of foreign matter. | Per 100 kilos. | Free. | Free. |
| Boracic acid, other........ | „ „ | 18 francs. | 12 francs. |

It appears that since the Tariff of 1892 was passed, several manufactories of this chemical product have sprung up in France, but their acid is undersold on the French market by the German manufacturers, and it is for this reason that a duty has now been voted.

The importation of boracic acid into France has largely increased of late years. In 1895 the quantity imported was 397,977 kilos, while in 1897 it had increased by 30 per cent. to 520,900 kilos.

It will be observed that a distinction is made in the tariff between the pure boracic acid, on which a duty is imposed, and the impure, which will still be admitted free of duty being largely used for candle-making, and therefore looked upon as a raw material.

The Customs Commission state that the duty of 12 francs is only equivalent to the difference of the cost of production in France and Germany, but it is more than probable that this figure has been exaggerated in order to afford an opportunity of imposing a protective duty.

### Italy.

*Tariff Modifications.*

*Board of Trade J., May* 1898, 568.

A despatch has been received from H.M. Ambassador at Rome, transmitting copy and translation of a Royal Decree, dated the 7th April, introducing certain modifications in the General Tariff of Italy, as follows:—

| Number and Letter. | Denomination of Goods. | Import Duties. |
|---|---|---|
| | CATEGORY III. | Lire. |
| | *Chemical Products, Medicines, Resinous Objects, and Perfumery.* | |
| 31 | Acids. | |
| *e* | Boric: | |
| | 1 raw ........................... | Exempt. |
| | 2 refined ........................ | Per quintal 5·00 |
| 41 | Borax, or borate of soda .......... | „ 4·00 |

## *EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.*

### PRODUCTION OF SUGAR IN GERMANY.

*Foreign Office Annual Series, No. 2064, April 1898.*

The expectation that the cultivation of beetroot during 1896–97 would be considerably extended has been amply verified, as will be seen by the following table, showing the total production with the export and home consumption of raw sugar in Germany during the above-named years (amounts given in tons of 1,000 kilos.):—

| Year. | Quantity. | | |
|---|---|---|---|
| | Produced. | Exported. | Home Consumption. |
| | Tons. | Tons. | Tons. |
| 1895–96 | 1,637,000 | 958,128 | 678,872 |
| 1896–97 | 1,821,223 | 1,237,521 | 505,078 |
| Increase, 1896–97 | 184,223 | 279,393 | .. |
| Decrease, 1896–97 | .. | .. | 173,794 |

The decrease as shown for 1896–97 in no way proves that during this period there was a real decrease in the home consumption, because before the season of 1895–96 had expired, large quantities of raw sugar were passed into the consumption list in order that it should be on the market under the old law, and consequently under the lower tariff, and was really consumed in 1896–97, but was accounted for in the home consumption list for 1895–96, before the new tariff came into force.

The output of raw sugar in continental countries during 1896–97 has increased as compared with the previous season with one exception only, and that is in Russia. The output of the various countries is shown in the following table:—

| Country. | Production. | | |
|---|---|---|---|
| | 1896–97. | 1895–96. | 1894–95. |
| | Tons. | Tons. | Tons. |
| Germany | 1,821,223 | 1,637,000 | 1,642,027 |
| Austria | 929,200 | 781,100 | 1,043,000 |
| France | 703,300 | 624,900 | 745,000 |
| Russia | 734,400 | 773,500 | 595,000 |
| Belgium | 280,000 | 200,000 | 252,000 |
| Holland | 156,800 | 102,000 | 85,000 |
| Sweden | 106,400 | 79,400 | 140,000 |
| Other countries | 70,000 | 60,000 | .. |
| Total | 4,802,023 | 4,257,900 | 4,502,027 |

Thus the total production in 1896–97 shows an increase of 544,123 tons, or 12¾ per cent. more than in the previous season.

With respect to the sugar trade and the results of the beet-growing industry in Germany, it may be truly said that it is one of the few businesses that did not participate in the general economical progress so observable in Germany during last year (1897); no other branch of German trade had to fight for its existence so determinedly as the sugar trade had and is still doing. It may be said that it is the general prosperity of German commerce that has prevented the sugar industry from failing completely.

The new "Borsengesetz," which came into force on January 1, 1897, and the passing of the law which forbade "Terminhandel," had a very unfavourable effect on sugar business.

German capitalists withdrew in many cases their support from the markets, which therefore fell more or less into the hands of foreign purchasers, who were thus able to keep the prices down during the whole season. A further decrease in the price was caused by over-production throughout nearly the whole of the Continent, and by the uncertainty in respect to the Cuban crops, estimates for which varied between 150,000 tons and 500,000 tons (a wide discrepancy).

A further adverse influence on this market was the introduction of the United States customs tariff, which not only raised the duty, but even imposed a differential duty on sugar imported from those countries that grant an export bonus.

As France does not give such high export bonuses as Germany, French sugar would have the advantage over German in the United States market, being admitted under a lower tariff.

Previously, however, to the new customs tariff, the influence of the impending duties had a good though transitory effect on the sugar business here, as the importers bought up all the sugar at disposal in Europe for export before the new customs tariff came into force.

The total requirements in sugar for the United States of America are estimated at 2,000,000 tons per annum, while the States themselves only produce 40,000 tons a year; according to the Statistical Office at Washington the import of sugar from July 1, 1896, to June 30, 1897, amounted to 2,150,000 tons, and in the previous year only 1,660,000 tons; that gives an increase during the latter year of 490,000 tons—a condition which was doubtless abnormal.

Of these 2,150,000 tons imported into the United States of America during 1896–97, Germany sent 1,511,400 tons, against an export in 1895–96 of only 449,920 tons—an increase in 1896–97 for Germany of 1,061,480 tons.

From Stettin and Swinemunde there were exported to the United States of America within the year, from August 1, 1896, to July 31, 1897, 168,355 tons against 101,972 tons in the previous year, showing an increase of 66,383 tons.

At the commencement of the year 1897, prices for raw sugar opened at 9 marks 30 pf. (9*s.* 2*d.*) per 50 kilos., falling by April to 8 marks 57 pf. (8*s.* 5½*d.*); in May prices rose again slightly to 8 marks 83 pf. (8*s.* 8½*d.*), and then fell to 8 marks 30 pf. (8*s.* 2*d.*) in August. In September, 9 marks (8*s.* 10½*d.*) could be obtained, and in November-December, 9 marks 57½ pf. (9*s.* 5¼*d.*) could be obtained f.o.b. at Hamburg.

The statement in the Reichstag at the end of the year by the Secretary of the Imperial Exchequer, to the effect that the International Sugar Conference about to be held at Brussels would, on account of the new United States customs tariff, deal with the question of abolishing the export bonuses, had a decidedly favourable influence on the sugar market here.

### MINING INDUSTRIES OF NORTH-WEST SPAIN.

*Foreign Office Annual Series, No. 2061, April 1898.*

The north-western provinces of Spain, Galicia, Asturias, and Leon, abound in minerals of all sorts, which only want exploiting by capitalists to make a remunerative industry. If British capitalists were to make serious and exhaustive inquiries, with a determination, if satisfactory, to carry out the business, money could be made in the mining and coal industries. Foreigners are more alive to the advantages to be obtained from the minerals, &c., in these districts than the English. German, French, and Belgian firms have been making investigations, and having during the last year purchased some properties, are already working them and shipping the produce.

In the Corunna district there are good iron mines yielding from 52 per cent. to 62 per cent. of metallic iron with from 1 per cent. to 1·20 per cent. of phosphorus; asbestos, copper, iron pyrites yielding from 2½ to 8 per cent. copper, carbonate of copper, silver lead, antimony, zinc, plumbago, and auriferous arsenical pyrites also exist. Every facility exists for working these minerals, especially the iron—ample water-power, abundance of timber, and cheap labour.

The district of Corunna abounds in lodes of auriferous pyrites. These lodes are true fissure veins with well-defined walls, and measure from 5 to 12 ft. in width, and many of them can be traced from their outcrops for a distance of over half a mile. They lie as a rule in the zone of the granite or gneiss and slate formation. Assays of surface samples have given from 3 to 9 dwts. of gold to the ton, and those from pits and excavations, from 15 dwts. to 1 oz. The general composition of the ore by analysis appears to be about as follows:—Iron 30 per cent.; arsenic, 35 to 50 per cent.; sulphur, 17 per cent.; gold, 1 to 16 dwts.; silver, 1 to 11 ozs.

A syndicate has been formed in Corunna, called the Anglo-Spanish Mining Syndicate, for the purpose of opening and developing good mining properties in this and surrounding districts.

### IMPORTS OF MANURES AND CHEMICALS AT GIJON, SPAIN.

*Foreign Office Annual Series, No. 2061, April 1898.*

For beet and other roots artificial manures now come in larger quantities, but none from Great Britain, while Belgium imported in 1897 1,750 tons of superphosphate of lime, and France 32 tons, at the following prices, f.o.b., Antwerp:—

| | Per 100 Kilos. Francs. |
|---|---|
| 10 to 12 per cent. of phosphoric acid | 5·48 |
| 12 „ 14 „ „ | 5·67 |
| 13 „ 15 „ „ | 5·99 |
| 14 „ 16 „ „ | 6·30 |
| 16 „ 18 „ „ | 7·43 |

The importers are La Sociedad Azucarera Asturiana, Gijon; Don Casimiro Velasco, Gijon; Don Antonio Cabanillas, Villaviciosa.

The dynamite works imported in 1897 the following quantities:—

| —— | From | Quantity. |
|---|---|---|
| | | Tons. |
| Nitrate of soda | Belgium | 514 |
| „ | Germany | 405 |
| „ | Great Britain | .. |
| Carbonate of soda | Belgium | 158 |
| Caustic soda | „ | 12 |
| „ | Great Britain | 21 |
| „ | France | 4 |
| Bicarbonate of soda | Great Britain | 7 |
| „ | France | 1 |
| Sulphate of soda | Belgium | 260 |
| „ | Great Britain | 214 |
| Sulphur | Belgium | 64 |
| „ | Great Britain | .. |
| Salt (table) | France | 25 |
| „ | Great Britain | .. |
| Nitrate of potash | Germany | 217 |
| „ | Belgium | 60 |
| „ | Great Britain | .. |
| Starch | Belgium | 53 |
| „ | Holland | 7 |
| „ | Germany | 2 |
| „ | Great Britain | .. |

The above list, which is very unsatisfactory to British trade, applies equally to all other substances employed in pharmacy, perfumery, and chemical industries. Efforts ought to be made to recover part of this trade.

No glycerine was imported in 1897 owing to the supplies coming *viâ* Bilbao since the formation of the syndicate for the monopoly of explosives.

Mineral and vegetable wax came in smaller quantities last year in the following proportions:—

| From— | Quantity. | Value. |
|---|---|---|
| | Tons. | £ |
| Belgium | 68 | 1,053 |
| Germany | 19 | 726 |
| France | 9 | 140 |
| Great Britain | 2 | 30 |

As large quantities of the above are manufactured in Scotland for exportation, the backward position of Great Britain here is noticeable and ought to be remedied.

### METALLIC EXPORTS OF GIJON, SPAIN.

*Foreign Office Annual Series, No. 2061, April 1898.*

The copper ore mines in Villamanin are apparently worked out, as the quantities become less; 69 tons only were shipped to Swansea in 1897 against 978 tons during the year preceding, and unless new deposits are found this item of export will cease.

Manganese ore has disappeared from the export list for 1897. Some 92 tons were sent to England in 1896, and it is inexplicable why the large deposits of Asturias are not worked. They would yield, it is reported, a good return on British capital if brought into this province.

A small mine of oxide, commenced some months ago, in which an Englishman has some interest, yielded 237 tons, of which 202 tons were shipped to Great Britain and 35 to Belgium. This oxide is used for making paints. The price obtained is reported as 50 to 60 frs. per ton f.o.b. at English and Belgium ports. With good demand, therefore, our paint makers ought not to allow this small opening to pass without an effort to ascertain if larger deposits are to be found in Asturias.

Sulphate of arsenic has decreased from 88 tons in 1896 to 40 tons last year. The whole was sent to France.

Quicksilver also declined from 71 tons to 56 in 1897. France was the importing country.

A general depression appeared to have set in last year, when the Royal Asturian Zinc Company of Aviles exported only 1,621 tons of manufactured zinc against 3,454 tons in 1896. The whole found an outlet in French markets.

### WOOD PULP IN SWEDEN.

*Foreign Office Annual Series, No. 2062, April 1898.*

The export of wood-pulp was scarcely so large in 1897 as in the previous year. This was entirely caused by the destruction of the Mackmyra works by fire. These works are, however, being rebuilt on a considerably larger scale than before, and will be in full working order shortly. Otherwise, the works of the district have been fully employed at remunerative prices. It is, in point of fact, well known that this branch has been one of the most profitable in Sweden during the last five years; and although the large increase in the means of production in the north of Sweden may reduce profits in the near future, there is no doubt the possession of cheap raw material will enable the works of the district to maintain their position. The production of the district consists mostly of sulphate pulp.

The Stora Kopparbergs Bergslags Aktiebolag have commenced the erection of large paper works at Avarnsveden, near Faben. They are said to have purchased two immense American paper-making machines of 107 and 127 inches respectively, as well as 18 new American turbines, developing together 12,000 horse-power, for these works. British papermakers will find it hard to compete in price with a powerful company like this, that manufacture paper from raw material grown in their own forests, unless they are able to acquire control of their own wood-pulp supply; and it is surprising that they have not more largely invested in wood-pulp works.

### SODA AT LULEA, SWEDEN.

*Foreign Office Annual Series, No. 2062, April 1898.*

The importation of soda during the year 1897 was 1,898 tons. It is the first time that this article has been imported to this port in such quantities, and there is no doubt that this import will increase, as the article is used for a new manufacture recently commenced.

### NEW INDUSTRY IN SWEDEN.

*Foreign Office Annual Series, No. 2062, April 1898.*

A new industry for mechanical magnetic separation of iron ore has been developed during the year. The magnetic ore, containing phosphorus, is crushed into sand, then separated by an American invention called the "Monarch system." The ore being thus concentrated has become almost non-phosphoric. By a new invention and patent process, the tailings, which consist of apatite, are made into a manure called "Wiborgh's phosphate." The works are calculated for an output of 100,000 tons concentrated iron ore; the output of the manure is entirely dependent upon the percentage of apatite contained in the ore, which percentage varies considerably. The new works promise to give work for a considerable number of labourers when fully developed.

### RUSSIAN BEET SUGAR IN AFGHANISTAN.

*Foreign Office Annual Series, No. 2067, April 1898.*

Owing to permission having been granted by the Russian Government for the exportation of beet sugar to Afghanistan through the custom-house at Kelif, where the excise duty is returned to the exporter, a marked animation has taken place in the Russian trade with that country, and it is stated that about 150,000 poods, or 2,410 tons, of sugar crossed the frontier into Afghanistan during the past year.

### MANGANESE FROM BATOUM.

*Foreign Office Annual Series, No. 2067, April 1898.*

During the past five years the export of Caucasian manganese from Poti and Batoum were as follows:—

| To | Quantity. | | | | |
|---|---|---|---|---|---|
| | 1893. | 1894. | 1895. | 1896. | 1897. |
| | Tons. | Tons. | Tons. | Tons. | Tons. |
| Great Britain | 42,930 | 65,110 | 60,616 | 77,754 | 68,650 |
| France | 4,100 | .. | 150 | 5,650 | .. |
| Russia | .. | 9,890 | 9,600 | 20,175 | 28,446 |
| Belgium | 3,125 | 2,520 | .. | 220 | .. |
| Germany | 40,405 | 51,455 | 59,565 | 58,925 | 70,810 |
| United States of America. | 36,070 | 28,300 | 55,787 | 3,600 | 42,200 |
| Total | 126,630 | 157,275 | 185,718 | 166,324 | 210,106 |

This table shows a considerable increase in favour of the past year, but at an enormous sacrifice to some of the dealers. At the present moment the f.o.b. price of manganese at Poti is about 2 cents per pood less than the actual cost of production including the cost of transport to that port.

Further deposits of manganese ore are to be found in the Trans-Caucasus, among which is an extensive seam that exists at a distance of about 50 miles from Batoum along the valley of the river Tchorokh. A good vehicle road leads up to the spot where the mines are situated, and the quality of the ore has been found to be superior to that procured at Tchiatur. English capital has been placed in these deposits, which are now being exploited, some of the ore having already found its way to Batoum, which will in future be the shipping port for all ore coming from that district.

### MINING AND SMELTING WORKS AT LAURIUM, GREECE.

*Foreign Office Annual Series, No. 2068, May 1898.*

The returns of the several companies show that, in spite of the war, the mining works have steadily increased. New sources have been discovered of some importance, which have repaid for the explorations made in the north and south districts, good veins of manganese iron and hematite ore having been found, whilst calamine or zinc ore becomes more and more scarce, and is now reduced to less than half the production of former years. As seen by the following table, the total output of marketable ores reached in 1897, 376,597 tons, as against 323,041, or 53,556 tons more than in 1896.

| | Quantity. |
|---|---|
| | Tons. |
| Manganese iron ore, raw | 147,789 |
| " " roasted | 15,000 |
| Hematite | 136,811 |
| Calamine or zinc ore, roasted | 21,830 |
| Silver lead ore | 9,660 |
| Rich galena, dressed | 6,915 |
| Blend of zinc and lead ore | 3,084 |
| Lead smokes | 2,508 |
| Total | 376,597 |

The above total, consisting of purely marketable ores, does not include the annual production of poor lead ore grounds and carbonates, which amounted to 409,085 tons, containing 3 to 5 per cent. of lead, which, when dressed by mechanical washing, was reduced to 287,693 tons, and out of which 16,074 tons of pig lead were obtained, containing from 2,100 to 2,915 grms. of silver to the ton.

Smelting works are carried on with 26 furnaces in all, which are continually at work in both the French and Greek foundries.

### U.S. CHEMICALS IN CHINA.

*Foreign Office Misc. Series, No. 455, May 1898.*

The export of chemicals, dyes, &c. (including ginseng) from the United States to China amounted to over 1,000,000 dols. (200,000*l.*) in 1897.

### CHEMICAL TRADE OF LEGHORN.

*Foreign Office Annual Series, No. 2075, May 1898.*

A good and increasing business is done in carbonate of soda, and there is room for further development. Importers here, however, have known how to excite competition and secure the article at a much lower price than used formerly to be paid. It is needless perhaps to insist on care and trouble being taken to secure a suitable agent.

The carbonate of soda imported here is used in the large soap and glass works of the city, and, in its crystallised form, in laundries, and also for the bleaching of all manner of piece-goods.

An excellent and rapidly increasing business is being done in sulphate of copper and zinc. The following are the statistics for the last seven years.

TABLE showing the Quantities and Values of Sulphate of Copper and Zinc imported from 1891–97.

| — | Quantity. | Value. |
|---|---|---|
| | Tons. | £ |
| 1891 | 808 | 14,757 |
| 1892 | 640 | 7,673 |
| 1893 | 704 | 11,427 |
| 1894 | 1,716 | 27,867 |
| 1895 | 1,835 | 29,809 |
| 1896 | 2,931 | 47,613 |
| 1897 | 3,667 | 62,537 |

Wine-growers are loud in their praise of this article, and now use nothing else, after the first application of sulphur, for the preservation of their vines. The import in 1897 is nearly three times greater than the average import for the years 1891–96. There is a very good opening here for business in this article, and a first-class agent with extensive connections among the Tuscan wine-growers, would be sure to obtain satisfactory results for his principals.

After two prosperous years (1895 and 1896) the export of boracic acid has again suffered a severe drop, owing to inability to make headway against the competition of Californian, South American, and other borates. As is always the case when the demand for the raw material declines, a little more business is done in its conversion into borax, and the export of borax is slightly in increase of last year's figure, though far enough below the average.

The fame of the Tuscan mercury of Monte Amiata suffers no abatement, and is always in brisk demand. The export has risen in the present year, and reached a figure nearer the average. Prices have ruled low, and mercury can now be secured for one-third less than the cost in years gone by. But the famous mines of Almaden in Spain are too potent a rival for Tuscany, and it may almost be said that Almaden dictates the price of cinnabar for the whole world.

### SIENNA EARTHS, OCHRES, AND UMBERS AT LEGHORN.

*Foreign Office Annual Series, No. 2075, May 1898.*

In the storing and manipulation of Sienna earths, Leghorn still enjoys the possession of an industry which may in a sense be described as peculiarly her own. The writer has recently visited the extensive works and warehouses of Messrs. Enrico Gianni and Co., in whose hands is the greater part of the trade of the place, and has inspected the stocks and the processes of burning and grinding. The principal articles stored and manipulated are Sienna earths, ochres, and Turkey umbers. The earths and ochres all come from a district lying close to Monte Amiata, in the province of Grosseto; but little of the product is found over the close lying border of the province of Sienna, and none at all near the city of Sienna, which has given its name to the famous earths. The yellow ochre is a surface earth, the Sienna earths lying beneath it in three strata as follows:—Upper stratum or "Bolo di Terza," *i.e.*, 3rd quality; middle stratum or "Bolo di Seconda," *i.e.*, 2nd quality; lower stratum or "Bolo di Prima," *i.e.*, 1st quality. There is yet another quality superior to first quality, being a careful selection made by hand of the finer and brighter pieces, chosen more particularly from the Bolo di Prima. Locally this selected earth is called "spurgo di bolo," or "giallone"; in the German trade it is known as "Electa." In their natural state the earths are of very varied shades of yellow according to quality; after being burnt they turn red. At the place of excavation the raw material, which contains perhaps 70 to 80 per cent. of water, is banked in heaps and left to dry in the sun. The ochres are washed in tanks through which running water flows, carrying off all impurities. Excavation is carried on only in the summer months, when the earths and ochres can be dried by natural heat, and a rainy summer gravely interferes with operations. Burning is also extensively carried on at the place of excavation at all times of the year, and charcoal being the fuel employed, the cost is very trifling, but the earths and ochres are also burnt in the Leghorn works. Here, however, the principal operation is to reduce the material, whether raw or burnt, to a fine impalpable powder by a process of grinding. It may be said that the pigments leave Leghorn in four different forms, namely, raw for burning and for grinding at place of destination, burnt for grinding at place of destination, raw already ground here, and burnt already ground here.

The Turkey umbers come from Cyprus in large Italian sailing ships at low rates of freight, and are warehoused at a very cheap rate in the vaulted warehouses, which date from the days when Leghorn was the emporium of the Mediterranean. Turkey umbers are of three principal kinds: light colour or limasol; dark colour or larnaca; very dark, commonly called black. It is always the raw or unburnt material which is imported. The umber is subjected to very careful overhauling before being burnt or ground, and all extraneous matter is removed. There is an import duty of 4 lire the quintal on this class of goods, but by special regulation the umbers may be taken out of bond for manipulation, and if exported, the duty is waived. The process of burning is somewhat different to that generally adopted for earths and ochres. The umber is kneaded into rounded masses about the size of a melon, and placed in a different kiln upon iron grids. This method is said to improve the colour. Sometimes, however, the umber is merely kiln-burnt like the earths and ochres. 721 tons of Turkey umber were imported in 1897, and 555 tons exported, nearly all after manipulation.

Sardinian ochres, both striped and plain yellow, are also imported and manipulated, but the quality is far inferior to the Tuscan. A certain amount of Sardinian manganese is also reduced to pigments in their works.

It is difficult to convey an idea of the price of these different articles, owing to the great variety of quality and shades of quality. During the past year raw siennas ranging over the three boli have fetched from 4*l.* to 12*l.* per ton; raw ochres (unwashed), 3*l.* 10*s.* to 5*l.* 10*s.*; washed, from 8*l.* to 10*l.*; raw umbers, 2*l.* per ton. Burnt siennas of the three principal qualities (know in Italy as "Chicco," "Gripolo," and "Polvere"), 4*l.* to 20*l.* per ton; "Spurgo di Bolo," or superior quality, a small and special business, 60*l.* per ton; ground siennas (both raw and burnt), including all qualities, 6*l.* to 30*l.* per ton; and ground "Spurgo di Bolo," 70*l.* per ton. Burnt ochres, 4*l.* to 10*l.*, and ground ochres (raw and burnt), 10*l.* and more per ton. Burnt umbers, 3*l.*, and ground umbers, 5*l.* 10*s.* per ton. The reason why there is little difference between the raw ground material and the burnt ground material, though the latter has had to bear the cost of burning, is that the dearer qualities of raw ochres and siennas are usually reserved for grinding. As no previous full report on this industry appears to have been sent in, some typical samples of earths, ochres, and umbers have been forwarded to the Association of Chambers of Commerce for the inspection of the English colour trade.

Italian siennas easily take the lead of all others for body, colour, strength, and durability. The absolute purity and genuineness of their character, still legible in the pictures of Italy's old masters, may be safely guaranteed in all acquisitions made in Leghorn. Local traders have to compete in some markets with a German article which appears to be a compound of German earth with the genuine sienna impalpable pigment, but nothing can well hope to dislodge Italian siennas from the famous English colour works.

### PAINTS AND CHEMICALS IN BILBAO.

*Foreign Office Annual Series, No.* 2078, *May* 1898.

Red lead and white lead are made in the country and are not imported. Oxide of iron is imported from England, but endeavours are being made to start a factory in Spain, with the object of securing this branch of the trade. Colours in powders are at present entirely supplied by Germany, with which England cannot compete. Zinc white and preparations are mostly procured from Belgium, but the national production is being developed. Earths used in painting used to be chiefly supplied from France, but for some years Spain has been preparing them, and has defeated not only France, but Germany, which also used to send small quantities. Prepared colours come but little from England, indeed only special qualities. They are admittedly good, but are unable to compete in price.

Chemical and pharmaceutical products hardly come at all from Great Britain. The market is supplied partly from France and much more by Germany. Surgical instruments, syringes, and similar articles sold by druggists are very largely supplied from Germany. Chemical products used in industries are mostly supplied from the United Kingdom. Belgium appears to compete in caustic soda and carbonate of soda. France, Germany, and recently Belgium have been competing with Great Britain in the supply of chloride of lime. France seems to send the most.

### EXPLOSIVES MONOPOLY IN SPAIN.

*Foreign Office Annual Series, No.* 2078, *May* 1898.

In the month of June, 1897, a law was passed authorising a monopoly in explosives for a term of 20 years in return for an annual payment of 3,000,000 pesetas. The monopolists soon closed all establishments where fuse, dynamite, powder, &c., were being made, pending a settlement of the terms of transfer, and raised the price of dynamite No. 3 from 42 pesetas 50 c. for a case of 25 kilos. delivered at the mines, accounts paid monthly, to 75 pesetas per case delivered at the depôt for cash. The monopoly began by supplying old and inferior dynamite, thus rousing the mining interest to take united action in order to obtain relief by the annulment of the contract.

The matter was submitted to first class legal opinion, and defects in the contract were at once discovered. The contract gave no specification; it only mentioned dynamite Nos. 1, 2, and 3. Also the monopolists failed to deposit samples. They should have done so at once, but did not do so until three months after they had begun operations, and not until the mining interest had already taken action.

The miners have been successful in the Tribunal de lo Contencioso, and the matter will now come before the Council of State.

If the monopoly is annulled the miners as a corporation offer to pay the 3,000,000 pesetas at present obtained by the Government, leaving the manufacture free. They propose to raise the money by having the right to require a stamp to be attached to every packet of dynamite.

### PETROLEUM IN THE DUTCH EAST INDIES.

*Board of Trade J., May* 1898, 601.

According to a recent report from the United States Consul at Batavia, the imports of oil into Java for two years have been, in gallons:—

| — | 1896. | 1897. |
|---|---|---|
| American | 16,786,820 | 20,092,900 |
| Russian | 7,319,510 | 4,951,060 |
| Langkat (Sumatra) | 3,306,520 | 5,295,690 |
| Total | 27,412,285 | 30,439,985 |

The current prices are 5*s.* 3*d.* per case of 10 gallons for American oil, 5*s.* 1*d.* for Russian, and 4*s.* 11*d.* for Langkat. The duty on foreign oil is about 2*d.* per gallon. The Consul says that Russian oil, though it undersells American, is being driven out of the market. The Sumatra oil shows a larger percentage of increase than the American oil, but this is due to the lower price of the local article. It must be taken into consideration that a most determined fight is being made against the Standard Oil Company, and that everything is done, officially and commercially, to hinder and annoy it. Locally, it is commonly reported that the Standard Oil Company is doing business at a loss, whereas the Sumatra oil companies are supposed to be making money, one of them having last year paid a dividend amounting to 100 per cent. There is a successful refinery in Java, near Sœrabaya, and it is claimed that Borneo will prove to be a great oil country.

### Belgian Experiments in Beer-making.

*Board of Trade J., May* 1898, 596.

The United States Consul at Brussels reports that the general association of the Belgian brewers has in view the creation in the district of Brussels of an experimental bureau for the trial of products for beer-making. The object of this bureau is to study all questions of a technical nature concerning the brewing industry, such as the use of brewers' yeast, research as to the different methods of production, analysis of all secondary products sold to brewers, &c. There will also be a study of all technical questions submitted yearly to the brewers' congress.

A capital of at least 25,000 francs (1,000*l.*) will be subscribed by different professional syndicates and by individual brewers. The annual budget of expenses, comprising the acquisition of products and apparatus, and the salaries of the employees, will be covered by the public funds.

### Aniline Colours in Germany.

*U.S. Cons. Reps., April* 1898, 564.

In response to a request for information regarding the manufacture of coal-tar colours in Germany, a department instruction was sent on Sept. 29th, 1897, to several consulates. The following report has been received from Consul-General Mason at Frankfort:—

The utmost that can be done in a report of this character will be to show some of the more important and obvious conditions which underlie the aniline industry and the methods by which Germany has attained her undisputed supremacy in that branch of manufacture.

Germany now manufactures nine-tenths of all the coal-tar dyes produced in all countries, and this to a considerable extent from raw materials—benzol and anthracene—imported to this country from Great Britain.

Coal tar colours may be divided commercially into two categories: first, the old, standard dyes, such as eosines, safranines, the soluble alkaline blues, and others, the patents on which have expired and which can therefore be made at will by anyone having the requisite facilities; second, the large and important class of dyes of all shades and colours that have been discovered during the past 15 years by European chemists, and patented.

The development of the aniline industry in the United States meets, therefore, at the outset two difficulties: the standard colours, open to competitive manufacture everywhere, are produced so abundantly and cheaply that their production yields little profit; while the rarer dyes, which yield large profits, are patented monopolies in which competition is impossible. As an example of the decline in the market value of a coal-tar colour, the manufacture of which is open to free competition, may be cited the group of scarlets, which a few years ago were protected by patents, and sold for 10 marks per kilo. Since the patents on these colours expired, a special establishment has been built in Germany for their production, with the result that they are now to be had in any quantity for from 1·70 to 1·80 marks per kilo. Even an *ad valorem* duty of 35 per cent. adds but little to the market value of such products in the United States, and their original inventors no longer produce them, but, when an order is received, purchase them from the laboratory expressly devoted to their manufacture. When it is remembered that all the leading aniline laboratories in Germany pay large dividends and have their entire plants "written off" on their books—that is paid for—it will be seen how difficult it would be for a new plant, with originally invested capital, to start in competition with them in the common and cheaper class of dyes.

The manufacture of coal-tar colours in the United States is no new or untried enterprise. Many years ago, a laboratory for that purpose was established in Albany, another at Brooklyn, and a partially successful effort was made at Cleveland in the eighties to produce aniline colours from the refuse of petroleum distillation. The great chemical works of Friedrich Beyer, at Elberfeld, where 100 skilled chemists and 25 educated engineers are now employed, have in operation at or near New York, a special laboratory for the manufacture of certain standard dyes, the materials of which can be imported or otherwise cheaply obtained in the United States. The firm of Heller and Merz, of New York, produces eosines and magentas; another first manufactures successfully Bismarck browns and nigrosines, all of which colours are of moderate price and not protected by patents. The manufacture of aniline colours involves the use, not only of crude coal-tar derivatives, but of sulphuric, nitric, and hydrochloric acids, besides alkalis and other chemicals which are mainly imported, and therefore more costly in our country than in Europe, although some progress has been made in recent years toward their production in the United States.

But the great advantage possessed by Germany in this competition lies in her command of abundant and cheap chemical skill and specially educated operatives. The universities, the polytechnic, and technical high schools of this country turn out a constant throng of scientific chemists and educated working-men, specially trained and prepared in the processes of chemical manufacture, and willing to work for wages that college graduates and the better class of operatives in America would refuse. As has been stated in a former report of this series (Commercial Relations, 1895–96, Vol. II., p. 166):—

"The great laboratories of Hoechst, Mainkur, Ludwigshafen, Elberfeld, and Berlin employ a large staff of chemists—from 50 to 70 at each establishment—whose sole function is that of research, whether it be a new colour or pharmaceutical product, or a cheaper, more direct method of producing some known product. They are mostly young graduates, and work for small salaries in laboratories perfectly equipped and supplied by their employers, under contracts which provide that whatever valuable discovery they make shall be patented and the patents transferred to the company, the inventor receiving a specified percentage of the profit accruing thereafter from its manufacture and sale. A single fortunate discovery may thus make the fortune of the inventor, and many of the most important chemical discoveries of recent years have been made by men under 35 years of age, working for less than 500 dols. a year for great corporations that pay annual dividends of 15 to 28 per cent. to their stockholders."

In respect to the cost of labour in this industry, it will be sufficient for the present purpose to give the wages of the different classes of employees in the Farbwerke at Hoechst, which may be taken as in all respects a standard establishment of its class.

Wages, per week of 60 hours: skilled operative in laboratory, 4·28 dols.; workman in laboratory, 4·21 dols.; mechanic, 4·05 dols.; yard labourer, 3·58 dols. As a partial offset to these low wages, the employees of the Farbwerke live at cheap rents in good houses provided by the company, have the services of a physician, and are assisted by wholesale purchasers of groceries and other articles of household use through a well-managed Consum-Verein. The company is also obliged by law to furnish its employees with bathing facilities at the works, and to make a certain provision for them in old age or in case of disability.

It should not be understood that Germany is to any serious extent dependent upon Great Britain or other foreign countries for aniline materials. English cannel coal is far richer in anthracene than either German or American coals, and, being largely used in gas manufacture, there is produced in the United Kingdom a surplus of anthracene, of which a considerable share comes to Germany. But for benzol, which is produced directly from the roasting of bituminous coal, Germany depends upon her own resources, the great coking plants of Westphalia and Silesia. So abundant has this material become in Germany, that much less benzol is imported from England than hitherto; and aniline salt is exported to the United States by not less than four different shippers in the consular district of Frankfort. The rate at which this export is increasing indicates that the manufacture in the United States of certain coal-tar colours is steadily gaining in importance, although the United States paid for imported aniline colours in 1896 not less than 3,072,915 dols.

One of the works most likely to be useful to the inquirer is the systematic survey of the organic colouring matters, by Schultz and Julius, edition of 1897 (this Journal, 1896, 926).

The use of such colours is steadily growing, and the activity of American textile manufactures has caused a decided increase in the importation of dyestuffs. When American ironmasters save the by-products of coke manufacture which they now waste; when education is specialised and young men are trained in colleges and universities to know thoroughly and practically the application of advanced science to productive industry; when Americans have learned to produce from the abundant materials at home the "heavy chemicals" which are now so largely imported; when they learn to economise materials as they now economise labour, and to sell the higher products of scientific manufacture in foreign markets as skilfully as they are now marketed at home—then the foundations of a permanent emancipation will have been laid.

### Coal Industry [Coke Ovens, with Recovery of By-Products] of the Rhenish-Westphalian Provinces.

*Foreign Office Misc. Series*, 1898, [454], 51—55.

In a report by Her Majesty's Consul at Düsseldorf, Germany, it is stated that the old beehive coke ovens are being abandoned in favour of the so-called "tar ovens," as the repairs are excessive, whilst the output is considerably less (20 to 25 per cent.) than in the new ovens. Although the coke produced in the tar ovens has not the same brilliant, silvery appearance as of old, its quality is said to be quite as good; in fact, a large amount of the carbon is lost in cooling the coke in round ovens. During the period 1885 to 1895 the number of round ovens in the Mining Board District of Dortmund fell from 762 to 142. In the same time the number of all coke ovens increased from 6,404 to 8,063, whilst the number of ovens with by-product systems increased from 306 to 1,864. The annual production of these ovens is about 1,860,000 tons of coke. Most of the new ovens are of the Hofmann type. In 1895 there were 1,262 such ovens in the Dortmund district. Each oven works separately, though the gases in the partition walls heat neighbouring ovens jointly. The ovens have double flues under the floor level. The air is drawn into the regenerators by fans. Each oven is 32·8 feet long, 5·9 feet high, and 1·74 feet wide. The charge of dry coal weighs about 6½ tons; the time for coking is 30 hours, and the production per oven and day is 4 tons, or in the year 1,440 tons per oven. The yield of coke is 79 per cent. Experimental batteries of coke ovens have been erected by Otto Hofmann at Dahlhausen, to ascertain under what conditions it is possible to abandon the use of regenerators. It is of the greatest importance to profit by the high calorific power of the gases. They should be introduced into the ovens in such a way as to secure the most perfect combustion, so that the coking is effected at a very high temperature. By this means the production of ammonia is greatly increased. According to Wehler, only 0·22 per cent. of the total nitrogen in coal is obtained in the form of ammonia, amounting to 11 kilos. of ammonia per ton of coal. In 1896 the sale of coke by the Rhenish-Westphalian Coke Syndicate was 7,000,000 tons; in addition to this, 30,000 tons of ammonia, at a value of 5,000,000 marks, were sold.

In coking, 280 to 300 cb. m. of gas escape per ton of coal, according to quality. The gas ascends through pipes in the retort. There is a stop-valve in the pipe, and this is closed when the coking process is complete, to prevent the introduction of air into the retort whilst the oven doors are open and the coke is being pressed out by mechanical means. The gases are exhausted by a fan, and go at first through air-coolers, which retain a large amount of tar and ammonia, and reduce the temperature from 600°—700° down to about 120°. They are then introduced into vertical iron cylinders placed at a higher level, in the bottom of which are 88 pipes, 4 ins. in diameter, through which cold water flows in a downward course, the gas ascending around being cooled to 17° or 18°. Leaving the fans, the gas is forced into cylindrical washers provided with perforated horizontal plates 4 ins. apart. Ammonia water descending extracts further quantities from the ascending gas. The same water is used for washing until it has reached a density of 3·5° B., when it is worked up into ammonium sulphate, utilising the apparatus designed by Feldmann. The gas leaving the washers is led through pipes into vessels, in which it is scrubbed with heavy tar oils. When the latter have been saturated with benzene, they are subjected to distillation, to recover the same, the gas yielding about 0·6 per cent. of benzene.

Sixty coke ovens produce in a month, according to the quality of the coal, from 160 to 180 tons of tar and 40 to 100 tons of ammonium sulphate.—D. B.

## *GENERAL TRADE NOTES.*

### Mineral Production of Spain in 1897.

*Board of Trade J., May* 1898, 597.

The following table, showing the production of certain minerals in Spain in the years 1896 and 1897, is taken from statistics published in the *Epoca* of Madrid:—

| — | | 1896. | 1897. |
|---|---|---|---|
| Lead .............................. | Tons | 167,017 | 176,000 |
| Silver .............................. | Kilos. | 64,554 | 131,000 |
| Quicksilver.............................. | Flasks | Not stated | 49,540 |
| Copper (of all kinds).............. | Tons | 1,500,954 | 1,458,191 |
| Zinc ore .............................. | " | 64,828 | 60,000 |

### The Sewage Commission.

*Chem. Trade J., May* 14, 1898, 333.

The Royal Commission on the Treatment and Disposal of Sewage has been constituted as follows:—Earl of Iddesleigh (chairman), Sir Richard Thorne-Thorne (Medical Officer of the Local Government Board), Lieut.-Col. Constantine Phipps Carey, Mr. Charles Philip Cotton, Professor Michael Foster (Cambridge), Lieut.-Col. Thomas Walter Harding, Mr. Thomas William Killick (Mersey and Irwell Joint Committee), Professor William Ramsay (University College, London), and Dr. James Burn Russell.

The following is the reference:—To inquire and report: 1 (*a*) What method or methods of treating and disposing of sewage (including any liquid from any factory or manufacturing process) may properly be adopted, consistently with due regard for the requirements of the existing law for the protection of the public health and for the economical and efficient discharge of the duties of local authorities; and (*b*) if more than one method may be so adopted, by what rules in relation to the nature or volume of sewage or the population to be served, or other varying circumstances or requirements, should the particular method of treatment and disposal to be adopted be determined; and (2) to make any recommendations which may be deemed desirable with reference to the treatment and disposal of sewage. Mr. Frederick James Willis is the secretary.

### Petroleum in Burma.

*Eng. and Mining J., May* 2, 1898, 555.

A monograph by Dr. F. Noetling, recently published by the Geological Survey of India, gives many interesting particulars about petroleum production in Burma. The present yearly output is about 250,000 barrels. So far the anticipations as regards the petroleum productions of Upper Burma have not been realised. The most important field has been thoroughly tested, and Dr. Noetling doubts whether the future will show any considerable increase. The localities where petroleum is known to occur spread over a considerable area, all the localities being situated on the top of anticlinal arches. The oil occurs in beds of miocene age, and its occurrence seems to be limited to a zone of about 10 miles in breadth, which runs along the eastern side of the Arrakan Yoma. As regards the chemical composition of the oil, much remains to be investigated, because no detailed analysis has yet been made. From a commercial point of view, the approximate average composition of Burma petroleum is as follows: illuminating oil, 50 per cent.; lubricating oil, 40 per cent.; paraffin, 10 per cent.

Since 1888 there has been a great increase in the number of productive wells, in their depth and in their output. Although this seems to indicate a prosperous condition of the native oil fields, it is not really so, because the increase has been the result of the sinking of a number of new wells in a district hitherto but little worked. On the other hand, those wells situated in the older parts of the field, which formerly supplied the production, show a distinct decline in yield, notwithstanding their increased depth.

### Canadian Trade with Great Britain.

*Chem. and Druggist, May* 14, 1898, 797.

The Canadian Government now admit imports from Great Britain on a tariff 12½ per cent. lower than those from nations which do not reciprocate or which have not treaty rights, and from and after August 1, 1898, this preferential advantage will be increased to 25 per cent., while at the same time the treaties will expire which gave other nations the right to share in this benefit. That these circumstances offer a great opening for the development of British trade with the Dominion will be evident from the following statistics:—

In the year ending June 30, 1897, Canada imported merchandise to the value of 114,512,415 dols. (nearly 23,000,000*l*. worth), and exported goods to the value of 134,157,703 dols. (nearly 27,000,000*l*.). Of these total imports Great Britain furnished 29,101,188 dols. worth, while the United States supplied 57,023,312 dols. worth. Germany supplied goods to the amount of 6,493,368 dols., France to the value of 2,601,351 dols., and Belgium, China, Japan, Spanish countries, and the West Indies contributed merchandise in each case of values between one and two million dollars.

Although Canada buys from the United States almost twice as much as she buys from the United Kingdom, she sells to the United Kingdom her products to the value of 77,227,502 dols., and to the United States products to the value of 41,933,792 dols.

To give an idea of the possibilities of British trade with Canada, we take the item of "drugs, dyes, chemicals, and medicines" imported into Canada during the year ending June 30, 1897. The imports of these goods from Great Britain were valued at 868,691 dols., those from the United States at 1,429,038 dols., and those from all other countries at 620,024 dols. This shows that in this particular branch of trade alone Great Britain, with the tariff advantage in her favour, has a good opportunity of adding anything between a quarter of a million and half a million sterling to her trade.

### Paraffin.

*Eng. and Mining J., May* 7, 1898, 552.

The export trade of the United States in paraffin and paraffin wax has grown up very rapidly, says the *New York Times*. Twenty-five years ago this material was unknown to commerce, and only 15 years ago it began to be exported. In 1881 the exports of the product were more than 17,000,000 lb., of which 16,000,000 lb. went to Great Britain. In the fiscal year 1897 the American exports of paraffin and paraffin wax reached about 126,000,000 lb., of which the United Kingdom took 82,000,000 lb. In the States the consumption of this article has increased at still more rapid rate, but the production has more than kept pace with the demand. The marked chemical indifference of the substance has led to its introduction in connection with a large number of industries. It is employed for lining wooden and metal vessels for acids, and voltaic batteries, in electric insulation, in coating splints and other appliances subject to septic influences, as a vehicle for fulminate in the manufacture of matches, as a covering for cartridges, for preserving fruits and vegetables by forming a coating over their surface, and for an almost endless variety of similar purposes. It is also used extensively in the manufacture of candles, for securing a high polish on clothes in laundries, and for waterproofing paper and fabrics.

### Corundum in North Carolina.

*A. M. Stone. Eng. and Mining J., April* 23, 1898, 490.

In preparing corundum for market, the raw material is first washed thoroughly in sluice boxes and shallow tanks in order to remove the lighter alluvial matter, chlorites, &c. If no other impurities are present, a clean, marketable product is the result; if any heavier crystalline matter is also present, after washing the material, it is passed through screw conveyers, and from thence to mullers, wherein the impurities are ground up by the abrasive action of the corundum, and afterwards eliminated by a further washing.

On the whole, this wet process is not satisfactory, and a large percentage of the product marketed does not fully meet the wants of the abrasive trade, because of neglect or inability to remove the foreign crystalline matter. It seems probable that the best results in cleaning corundum can be obtained through some form of dry process by attrition. As corundum is 2½ times harder than any of the impure matters associated with it, and can be depended upon to cut those impurities into powder, it is necessary to have a process wherein a high degree of friction is obtained. Heat generated by such a process would not be injurious, but, on the contrary, beneficial in promoting the disintegration of the refuse. This process would be more rapid and cleaner, while a more satisfactory product would be secured. The corundum would receive no injury whatever in thus being temporarily used as a pulveriser, but a slight loss in the shape of flower corundum would ensue.

### Congo Trade.

*Chem. and Druggist, May* 7, 1898, 777.

The annual report of the Congo Free State to the King of the Belgians, which has just been published, contains full particulars of the exports and imports of the colony. Among the exports are the following:—India-rubber shows a total export amounting to 1,785,378 kilos., valued at 8,926,890 frs.; copal exports amounted to 53,825 kilos. of the white variety and 45 kilos. of the red, the value being 91,610 frs. Palm oil reached 1,334,870 kilos., value 694,132 frs. The Portuguese possessions bought two-thirds of this, and Great Britain 216,339 kilos. The exports of kola-nuts were 4,791 kilos., valued at 7,186 frs. No less than 3,446 kilos. of the kola were sent to Germany. Among the imports we observe:—Colours, varnishes, and paints, valued at 48,878 frs.; drugs, 63,722 frs.; chemicals, 10,063 frs.; pharmaceutical products, 142,072 frs.; soaps, 41,818 frs. Naturally, Belgium does the bulk of the trade with the Congo, but in many cases Great Britain comes in a respectable second; for example, it supplied 10 per cent. of the colours, &c., and 972 frs. worth of pharmaceutical products, this being the next highest figure to Belgium's 113,684 frs. In soaps this country leads the way with 13,571 frs., compared with Belgium, 12,860 frs. Our trade in chemicals with the colony is small, *viz.*, 697 frs. worth, compared with 9,239 frs. from Belgium.

### The Mineral Production of Germany.

*Eng. and Mining J., April* 23, 1898, 488.

From advance sheets of the official returns, we are able to present the following table of the mineral production of Germany in 1897, comparisons being made with 1896. The figures are in metric tons, and the average prices are reduced to United States currency;—

*Mineral Production of Germany.*

| Substance. | Measure. | Quantity. | | Average Value. | |
|---|---|---|---|---|---|
| | | 1896. | 1897. | 1896. | 1897. |
| Products of mines: | | | | Dols. | Dols. |
| Coal | Metric tons | 85,690,233 | 91,007,624 | 1.65 | 1.76 |
| Brown coal | " | 26,780,873 | 29,423,432 | 0.54 | 0.54 |
| Petroleum | " | 20,395 | 23.303 | 13.87 | 14.26 |
| Rock salt | " | 758,867 | 763,412 | 1.01 | 1.00 |
| Kainit | " | 877,885 | 995,821 | 3.61 | 3.34 |
| Other potash salts | " | 902,707 | 950,367 | 3.13 | 3.02 |
| Iron ore | " | 14,162,335 | 15,448,212 | 0.86 | 0.92 |
| Zinc ore | " | 729,942 | 663,850 | 5.55 | 6.05 |
| Lead ore | " | 157,504 | 150,179 | 19.64 | 20.63 |
| Copper ore | " | 717,346 | 700,619 | 5.63 | 6.16 |
| Silver and gold ore | " | 11,320 | 9,708 | 36.00 | 35.62 |
| Cobalt, nickel, and bismuth ore | " | 4,087 | 3,356 | 36.68 | 39.65 |
| Pyrites | " | 129,168 | 133,302 | 1.80 | 2.08 |
| Products of metallurgical works: | | | | | |
| Pig iron | " | 6,372,575 | 6,879,541 | 11.19 | 12.10 |
| Zinc | " | 153,100 | 150,739 | 73.23 | 79.70 |
| Lead | " | 117,723 | 122,222 | 52.51 | 57.44 |
| Copper | " | 29,917 | 29,723 | 234.37 | 242.13 |
| Silver | Kilos. | 428,429 | 448,068 | 21.59 | 19.32 |
| Gold | " | 2,487 | 2,781 | 661.92 | 662.19 |
| Nickel and bismuth products | Metric tons | 1,391 | 1,464 | 931.45 | 970.31 |
| Quicksilver | " | .. | 4,980 | .. | 0.99 |
| Tin (plates and wares) | " | 826 | 929 | 271.29 | 294.90 |
| Antimony and manganese | " | 1,326 | 1,665 | 112.12 | 120.50 |
| Arsenic products | " | 2,637 | 2,989 | 79.93 | 86.30 |
| Sulphuric acid | " | 587,524 | 608,767 | 6.70 | 5.81 |
| Sulphate of copper | " | 6,046 | 5,519 | 73.45 | 80.64 |
| Products of salt works: | | | | | |
| Salt | " | 547,486 | 543,332 | 6.37 | 5.31 |
| Chlorate of potash | " | 174,515 | 168,001 | 31.19 | 32.67 |
| Glauber salt | " | 66,661 | 68,822 | 6.04 | 6.01 |
| Potassium sulphide | " | 19,538 | 13,774 | 39.33 | 39.10 |
| Magnesium chloride | " | 27,161 | 35,072 | 3.78 | 4.22 |
| Aluminium sulphate | " | 34,370 | 37,053 | 16.49 | 15.77 |

The total value of the products given in the table for 1897 was 1,430,571,597 marks; and to this is to be added an estimated sum of 500,000 marks for minor products not enumerated, which include graphite, asphalt, kieserit, tin ore, uranium ore, manganese ore, and arsenical pyrites among the minerals; cadmium among the metals; sulphur, certain mineral paints, and a few chlorides. This would bring the total to 1,431,071,597 marks, or 240,595,040 dols. It is to be noted that there is some duplication in the table, both ores and the metals obtained from them being given. Making allowances for this, the total net value was a little over 200,000,000 dols., or somewhat over one-quarter of that of the United States.

The chief iron ore producing districts in 1897 were Elsass-Lothringen, which furnished 5,360,840 tons; Luxemburg, 5,349,010 tons; and Bonn, 2,736,071 tons.

Of the copper production, 29,408 tons was in fine copper and 315 tons in black copper. The lead included 118,881 tons pig lead and 3,341 tons oxide. Of the sulphuric acid, 604,082 tons were ordinary commercial acid and 4,685 tons concentrated acid. The other products are sufficiently shown in the table.

### The Corundum Deposits of Ontario.

*Imp. Inst. J., May* 1898, 138.

In the autumn of 1896, Mr. Ferrier, of the Geological Survey, came across a deposit of corundum in Hastings county, which, now that the general results of the preliminary tests are announced, bids fair to rank amongst the most valuable discoveries of recent years in Ontario.

Corundum had not previously been found in the Dominion. Dr. Dawson, upon examination of the samples brought back to Ottawa, was impressed with the probable importance of the occurrence, and upon his report to the Ontario Bureau of Mines a large block of Crown lands in the vicinity was withdrawn from sale.

In the spring, Mr. W. G. Miller, of the Kingston School of Mining, was instructed to make a thorough exploration of the district. Ten weeks were spent in Northern Hastings and Southern Renfrew, and the corundum belt was traced for a distance of 30 miles across country from Carlow, the site of the first find, eastwards to Sebastopol. The mineral was found in seven different townships over an area of about 100 square miles. There are numerous indications that the belt stretches still further towards the north-east, whilst it is also believed that a second and probably parallel belt exists towards the south, as a discovery of corundum has been made in Peterborough county, about 40 miles south-west from the original location of the mineral. The distribution was unequal, and the presumption is that the workable quantities will be found in isolated bodies.

Whilst corundum is chiefly known as an abrasive material, the importance of which recently has sustained somewhat of a check from the adoption of an artificial substance, carborundum, the main value of the present discovery is based upon its being an ore of aluminium, and therefore probably a producer of that metal. In Europe and the United States, aluminium is chiefly produced from kaolin, bauxite, and cryolite, all of which are low in aluminium compared with corundum. The only known valuable mine of cryolite is in Greenland, where mining operations are attended with many difficulties. In Ontario, on the contrary, the corundum deposits are easily accessible and can be worked throughout the year, whilst the cost of production, owing to the water-power available, will be relatively cheap. The Madawaska river and other streams almost in the centre of the belt, provide power for driving machinery or producing electricity, and at Palmer's Rapids there is a water privilege of several thousand horse-power capacity still in the right of the Crown. Above this point the river is navigable to the head of Barry's Bay, close to the Parry Sound line of railway. Towards the south there will also shortly be means of transit.

At present everything hinges on the results of the tests now in progress. During the month of December, several tons of ore were taken out and conveyed to Kingston, where a series of tests is being made by Mr. De Kalb, of the School of Mining. The preliminary report has just appeared. The tests which have so far been made are merely sufficient to indicate that successful concentration of the corundum is feasible upon a large scale. By means of additional machinery now being erected, an exhaustive investigation of the problem of the economical milling of corundum will be undertaken, after which Mr. De Kalb will submit a full report.

So far, the ore has been crushed and then passed through variously meshed screens. The resultant sized siftings were weighed, and each lot tested to ascertain the percentage of corundum and of magnetite which it contained. The quantity of corundum is relatively greater in the coarser and finer grades, whilst the magnetite reaches its maximum in the grades of medium fineness. This, Prof. De Kalb considers a fortunate circumstance, since it admits of a better separation of the corundum, and will also yield grades of concentrates rich in magnetite which may be economically separated as a by-product. Although the experiments in practical concentration were incomplete, enough was done to demonstrate that this work can be accomplished economically. Prof. De Kalb adduced from his tests that the ore contained 27·91 per cent. of corundum and 1·64 per cent. of magnetite. As an example, 45 lb. of the ore screened, yielded concentrates weighing 5·06 lb., which contained 71·56 per cent. of corundum and 22 per cent. of magnetite. After the elimination of the magnetite by the magnet, the resultant concentrates contained 91·71 per cent. of corundum.

"The commercial standard of purity for corundum concentrates," says Prof. De Kalb, "requires only 80 per cent. of corundum, so that it appears that this limit can be easily passed. I may venture the opinion, from the preliminary work done, that 80 per cent. of all the corundum in the ore can be obtained in the form of concentrates averaging 90 per cent. of corundum." The report is regarded as very satisfactory. It assures the value of the corundum deposits of North Hastings, and it is confidently expected that an important mining industry will spring up in this section of Ontario. It is also worth noticing that, during his examination, Mr. Miller came upon signs indicating the presence of tin, and, as the geological formation is favourable to the presence of this metal, a further investigation will be conducted with the advent of spring.

### The Indigo Trade of India.

*Imp. Inst. J., May* 1898, 137.

There has recently been issued, by Messrs. Cresswell and Co., an indigo report dealing with the question of shipping produce direct to London, instead of allowing it to be sold in Calcutta—a course which, in their opinion, has had a most disorganising effect upon the market, and contributed in no small measure to the existing dulness in trade.

During the present depression in the indigo trade, the questions of improving quality and cheapening production are especially emphasised by the appearance of an artificial substitute brought out by the Badische Anilin und Soda Fabrik, under the name of "indigo pure" (this Journal, 1898, p. 41). It appears both in powder and paste, and, compared with the natural product, is said to offer the following advantages:—

(1.) Owing to the extremely fine state of division, it enables the tedious and expensive operation of grinding, necessary in the preparation of indigo for use, to be dispensed with.

(2.) The product being pure, a more effective control of the vats can be maintained than was formerly possible, and the amount of indigotin consumed from time to time may be determined with exactitude.

(3.) It is easily tested and valued.

The product is already offered at about the same *pro ratâ* price as ordinary indigo of good quality, *viz.*, 17·5 marks per kilo. (about 8*s.* per lb.) for 98 per cent. of colour—Oudhs, giving about 50 per cent. of indigotin, is about 3*s.* per lb.—and it is considered cheaper than the refined indigo of commerce. It has been found that whilst the new product behaves towards wool, in dyeing, as natural indigo, it does not dye cotton so well as the latter, owing to the presence, in commercial indigo, of other constituents, which act as mordants on the vegetable fibre. Natural indigo contains, besides indigotin, the substances known as indirubin, indigo-brown, and indigo-gluten, together with traces of mineral salts which make the "ash." The proportions of these constituents vary considerably:—

| | Per Cent. |
|---|---|
| Indigotin ........................ | 20 to 80 |
| Indirubin ........................ | 2 to 10 |
| Indigo-brown........................ | 1 to 6 |
| Indigo-gluten ........................ | 2 to 5 |
| Ash ........................ | 3 to 20 |

The general experience among dyers is that indirubin plays an important part as a dyeing agency, and it is said that without this constituent there is no thoroughly fast indigo-blue in the fibre. On this account the redder qualities of indigo (containing the larger proportion of indirubin) command a higher price, and Java indigo (containing a very small proportion of indirubin) cannot be used for yarndyeing. The importance of the presence of this constituent is recognised by the Badische Anilin und Soda Fabrik, who propose the addition of certain compounds, notably albuminoids, glue, and fatty matters, amongst which Turkey-red oil may be particularly mentioned, as acting even more powerfully than the substances contained in natural indigo. These materials may be added to the dye vats, or the fibres may be impregnated with them before dyeing. The cotton thus prepared not only dyes in a satisfactory manner, but also exhibits, when dyed, a fine purple shade contrasting greatly with the shade ordinarily obtained, and especially with the weak greenish shade resulting from the use of the artificial product in the absence of such mordanting compounds.

In addition to the use of this synthetical product as a substitute for natural indigo in dyeing, it has been advantageously employed in the manufacture of indigo-extract and indigo-carmine.

The indigo plant has hitherto been an important factor in the commercial prosperity of the Indian Empire, the total export for the last few years being as follows:—

| | | Crores of Rupees. |
|---|---|---|
| 1893—1894, 131,407 cwt. | ................ valued at | 4 |
| 1894—1895, 166,308 .. | ................ .. | 4½ |
| 1895—1896, 187,368 .. | ................ .. | 5½ |

Hence, the appearance of a substitute which can be sold at a lower price than would cover the cost of production of the natural product should receive the serious consideration of the planters.

### The Artificial Silk Industry.

*Imp. Inst. J., May* 1898, 142.

The artificial silk industry is now reaching a period of heavy production in France. According to a recent article in *La Nature*, a wealthy company has established extensive works at Besançon, the products of which are becoming more and more widely known. Silk dealers and the manufacturers of fabrics are, however, the only persons who are acquainted with artificial silk; the public being unable to perceive any difference between it and the natural product, which it equals in texture and brilliance. It is simply not quite so strong as natural silk, and is therefore specially employed for fabrics of which the warp is made of ordinary silk of inferior quality, for ribbons with a cotton warp, and for all kinds of fancy goods, for dress materials, theatrical costumes, &c.

With proper precautions, and by using special processes, dyers give this artificial product all the richest shades of colour. The fundamental principle and general processes of its manufacture from nitro-cellulose (gun-cotton) were discovered by Count de Chardonnet. Nitro-cellulose is first manufactured by the ordinary processes. Instead of cotton, wood-pulp, such as that used for the making of paper, may be employed; but there is no advantage in this at the present price of cotton. The gun-cotton is dissolved in a mixture of alcohol and ether under pressure (40 or 50 atmospheres) in special apparatus. In this way there is obtained a viscid collodion thicker than that used in photography. This has to be filtered under pressure with the greatest care, for it must not retain the smallest fibre of undissolved guncotton. The thoroughly clarified collodion is led by a large pipe to the spinning room, and, always under a pressure of from 40 to 50 atmospheres, is there distributed by smaller pipes to all the spinning frames. Upon the pipes that occupy the length of each frame are fixed the spinning nozzles, each of which consists of a small reservoir communicating with the feed pipe, and terminating in a glass tube drawn out to a point provided with an orifice one hundredth of a millimetre in diameter.

The thread of cotton that issues from this tube (an imitation of the spinneret of a silk worm) was, in the first place, plunged into water, which solidified it immediately by removing the alcohol of the collodion; but much better results are now obtained by dry spinning, in which the solvent (alcohol and ether) rapidly evaporates, so that the threads have no tendency to adhere to each other when the machine winds them around the bobbins.

It requires three or four cocoon threads to constitute a warp thread, and more to form thrown silk. The threads of the artificial silk are, like those of the ordinary article, submitted to "throwing" and "twisting," but, before working it up into fabrics, the new silk has to be denitrated. In fact, the product being in the first instance still nitro-cellulose, and therefore very inflammable and even explosive, the finished skeins are immersed in a solution of ammonium sulphide, which removes the nitrogen oxide and restores the material to cellulose or cotton. The Chardonnet silk, thus denitrated, loses none of its qualities and remains but slightly combustible; no more so, in fact, than is spun cotton in the same condition of manufacture.

Since this industry can now furnish unlimited quantities of the product at 50 per cent. less than cost of silk, the applications of it have become very numerous. It is used in the manufacture of the "silk straw" that is so much in favour for women's bonnets, the threads being juxtaposed and held by an elastic sizing in such a way as to constitute flat ribbons a few millimetres in width. This silk enables milliners to obtain entirely new and striking effects.

### The Mineral Production of British Columbia.

*Imp. Inst. J., May* 1898, 142.

From the annual report of the Minister of Mines for British Columbia, a copy of which has recently been received, it appears that the value of the minerals produced in the province during 1897 was 2,091,054*l.*, against 1,501,589*l.* in 1896. In gold alone the increase is equal to nearly 200 000*l.*, the total value of last year's output of placer and lode mines being 527,268*l.*, against 337,641*l.* in the preceding year. Silver shows a still greater increase, the value of the 1897 output being 654,567*l.*, compared with 420,138*l.* in 1896. Copper rose from 38,185*l.* in 1896 to 53,252*l.* last year, and lead from 144,277*l.* to 278,102*l.* Coal fell off a little, there being produced 882,854 tons, compared with 894,882 tons in 1896; but coke rose from 615 tons in 1896 to 17,832 tons. Of other minerals the total value was 30,320*l.* in 1897 and 3,000*l.* in 1896. The increase in coke was due to the demand for the Kootenay smelters, the Kootenay goldfields, and the Comox (Vancouver Island) coke ovens, the only available source of supply at present, reacting on each other.

It should be observed that cheap coke is the chief condition requisite to make the treatment of the low grade ores of the Rossland division profitable. Now that the Crow's Nest Pass railway is being pushed through, the Kootenay district will soon have an alternative source of supply from an opposite quarter, the Crow's Nest coalfields. The road is to be built through to Nelson by the end of August. It will undoubtedly give new life to the gold and silver mining camps of Southern British Columbia. At the same time the British Columbia Government is endeavouring to have the Victoria, Vancouver, and Eastern railway built from the coast into this district. Moreover, the Canadian Pacific Railway Company promises to carry on its Crow's Nest Pass line 100 miles beyond the Rossland region and into the Boundary Creek country, where it would meet the line of the Victoria, Vancouver, and Eastern. According to present expectations it will reach Boundary Creek this year.

The report shows a steady growth of the mining industry in British Columbia during the year 1897, and it is said that the present year will see a substantial increase; this appears to be indicated by the Customs returns for shipments of ore for January, 1898, which amounted to 1,193,158 dols., as compared with 675,506 dols. in January 1897.

### American Quinine.

*Oil Paint and Drug Reporter; through British and Colonial Druggist, May* 13, 1898, 607.

The tariff duties on quinine sulphate entering the United States have varied very much The duty was removed in 1879, and that is what the home manufacturers mean by speaking of that year as one of adverse legislation to themselves and advantage-giving for the Germans. In speculating on future developments, the author of this paper suggests that with free bark, and a protective duty of 5 cents per oz. on quinine, the United States manufacturer may probably be able to battle effectually with his foreign competitor. A duty of 2½*d.* per oz., with the present low price of quinine, should certainly give any home manufacturer a decided advantage. In connection with this question of imports of, and duties on, bark and quinine, it is interesting to summarise the statistics of the former. From these we see that the United States reached her highest year in the importation of quinine-yielding bark in 1879, when she imported 6,389,378 lb. of bark. The duty-repeal must have been the cause for the subsequent decline, for two years after the imports had dropped to rather over four million pounds. For the last ten years they have varied between two millions, and three and a half millions. The imports of quinine from 1875 up to 1891, had a general upward tendency, reaching in the latter year over 3,000,000 oz. Since then they have fluctuated, being as low as 1,380,959 oz. in 1895, and amounting to 2,714,147 oz. in 1897. It is curious to note that the imports of cinchonidine into the United States during the last 13 years were highest in 1888, when they amounted to over 609,000 oz., and the lowest in 1892, when they were only 11,183 oz. Last year they were 450,000 oz.

### Some Metal Statistics.

*Ironmonger, May* 21, 1898, 399.

A useful compilation of comparative statistics of lead, copper, spelter, tin, silver, nickel, aluminium, and quicksilver has just been issued by the Metallgesellschaft of Frankfort-on-Maine. Not all this information is new, but some of it has not been published before; and the tables of statistics are presented with a lucidity deserving of all praise. Lead is the first of the metals dealt with. The world's production in 1897 is given as 709,400 metric tons, which is the largest annual output on record. The United States were the largest producers of this metal (176,500 metric tons). This output is also a "record," although it was run close by the U.S.A. output of 1892, which reached 166,200 metric tons. After that year the figures fell back considerably until 1897. Spain comes next with 169,000 metric tons, against 165,000 and 154,000 in the two preceding years. Our own production is estimated at 60,000 metric tons, which is an improvement upon the average of the last decade, but only equal to that of 1884 and 1885. Turning to consumption, the United States again hold first place, followed closely by Great Britain, the respective totals being 205,839 and 202,331 metric tons, while Germany is third with 129,898 metric tons. Last year's total consumption of lead in the world is given as 726,000 metric tons, against 677,100 metric tons in 1896, so that stocks must have been drawn upon to the extent of about 17,000 tons. The average price for the year reflects the increased demand for the metal, and stands at 12*l.* 8*s.* 9*d.*, compared with 11*l.* 5*s.* in 1896. An interesting table giving the average price of English lead since 1771 shows that the highest price recorded (35*l.* 15*s.* per ton) was reached in 1806; but the price all round the beginning of the century—in fact, from 1801 to 1811 (the period of the Napoleonic wars)—was never below 24*l.* per ton. The reverse side of the picture is shown by the 9*l.* 11*s.* 6*d.* quotation, the lowest average yearly price. This was reached in 1894.

The copper production in 1897 (metric tons being used in all cases) is given as 401,000 tons, and the consumption as 401,000 tons. Great Britain heads the list of consumers with 105,263 tons, the United States coming next with 96,713 tons, and Germany third with 89,798 tons, while France takes fourth place with 49,868 tons. A point worth noticing in connection with the consumption of the four leading countries is that whilst Great Britain's total in 1897, compared with that of 1889, shows an increase of only about 18½ per cent., and that of the United States of 25½ per cent., that of Germany has nearly doubled and France has more than quintupled her figures. In the matter of price the past year witnessed a considerable improvement, the average working out at 49*l.* 2*s.* 6*d.*, compared with 46*l.* 15*s.* in 1896; and when it is remembered that 40*l.* 7*s.* 6*d* was the average in 1894, it will be understood how great a harvest is being reaped by the copper-producing companies throughout the world.

Spelter is next dealt with, the world's production for 1897 being returned as 436,000 English tons (443,000 metric tons), to which Western Germany, Belgium, and Holland contributed 184,000 tons, Silesia, 94,000 tons, and the United States, 88,000 tons. As a consumer Germany is easily first with 119,000 metric tons, Great Britain and the United States being next with 86,000 and 77,000 metric tons; but production out-stripped consumption to the extent of about 2,000 metric tons. Nevertheless, producers make a good thing of it, the average price (17*l.* 10*s.*) being higher than since 1892, when 20*l.* 15*s.* was recorded.

The tin-production table is a copy of that of a well-known London house, which gives the yield as 69,972 English, equal to 71,092 metric tons. Here also consumption has made inroads on the stock, the total quantity used in 1897 being given as 75,900 metric tons. The United States were easily first with 24,000, whilst Great Britain is second with 15,000 tons, and Germany follows with 2,500 tons less. The average price, 61*l.* 7*s.* 6*d.*, although more satisfactory to producers than the 59*l.* 10*s.* quotation of 1896, cuts but a pitiful figure beside the 93*l.* 7*s.* 6*d.* of 1892 and the 117*l.* 10*s.* of 1888.

The nickel returns for 1897 are not complete, but the figures available show increases upon those of the previous year. The production of antimony nearly doubled itself last year, rising to 3,414 metric tons from 1,789 in 1896, and the average price is the same as that of nickel. The returns show that the production of quicksilver varies very little, but, as with nickel, the statistics for 1897 are not completed. The falling-off in the United States is compensated by the increase in Spain, and the average price of the year shows an improvement upon that of 1896. The aluminium statistics (see below) bring home the fact that aluminium is now 400 times cheaper than when it first became a marketable article. The North American output of nickel has grown from 409 metric tons in 1889 to 1,800 metric tons in 1897, and the total production in the same period from 1,830 to about 5,400 metric tons. The only great producer besides Canada nowadays is New Caledonia. Some nickel is found in the Saxon mines, but the quantity is not published, and the output of Scandinavian nickel, after steadily dwindling from 125 metric tons in 1891 to 20 metric tons in 1896, ceased altogether in 1897.

PRODUCTION OF ALUMINIUM.

*Ber. der Metall Gesellschaft Act.-Ges., Frankfurt a/M.*, 1898.

| Year. | England. | France. | | | Germany. | | Switzerland. | | U.S.A. | | Total. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Production. | Production. | Imports. | Exports. | Production | Imports. | Production. | Exports. | Production. | Imports. | Production. |
| | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. | Kilos. |
| 1885 | 1,000 | 1,982 | .. | 1,114 | 10,000 | .. | .. | .. | 310 | 199 | 13,292 |
| 1886 | 1,000 | 2,430 | .. | 152 | 10,000 | .. | .. | . | 2,950 | 204 | 16,380 |
| 1887 | 1,000 | 2,042 | .. | 542 | 15,000 | .. | .. | .. | 8,090 | 571 | 26,132 |
| 1888 | 11,500 | 4,155 | .. | 1,807 | 15,000 | .. | .. | .. | 8,640 | 641 | 39,295 |
| 1889 | 34,500 | 11,840 | .. | 762 | .. | .. | .. | .. | 21,580 | 452 | 70,920 |
| 1890 | 70,000 | 37,000 | 109 | 187 | .. | .. | 40,548 | 137,200 | 27,850 | 929 | 175,388 |
| 1891 | 52,500 | 36,000 | 594 | 430 | .. | .. | 168,669 | 201,900 | 76,138 | 1,777 | 333,307 |
| 1892 | 41,000 | 75,000 | 2,007 | 12,287 | .. | .. | 237,395 | 277,900 | 133,635 | 19 | 487,030 |
| 1893 | .. | 137,000 | 1,524 | 33,367 | .. | .. | 137,476 | 404,100 | 441,336 | 3,511 | 715,812 |
| 1894 | .. | 270,000 | 2,958 | 30,787 | .. | .. | 600,000 | 520,200 | 370,372 | 2,493 | 1,240,372 |
| 1895 | .. | 360,000 | 3,806 | 109,954 | .. | .. | 650,000 | 490,900 | 416,760 | 11,458 | 1,426,760 |
| 1896 | .. | 500,000 | 7,200 | 192,000 | .. | 591,500 | 650,000 | 661,100 | 588,900 | ? | 1,738,900 |

With the increase in production there has been a fall in price to 100 m. in 1886, to 12 m. in 1891, to 3 m. in 1895, and to 2·6 in 1896, whilst the price in 1898 is expected to be 2·34 m. per kilo.—W. G. M.

ARSENIC EXPORTS FROM BELGIUM.

*Eng. and Mining J.*, *May* 14, 583.

United States Consul Gilbert, at Liege, Belgium, says that in the year 1895 arsenic was exported to the United States to the value of 19,998 dols. In 1896 the aggregate of such exports was 18,051 dols. In the first two quarters of the year 1897 no arsenic was invoiced at this consulate, but during the last two quarters 29,225 dols. worth of arsenic was forwarded to the United States. During the first quarter of 1898 the declared value of white arsenic at this consulate was 15,961 dols.

ZINC DUST FROM BELGIUM.

*Eng. and Mining J.*, *May* 14, 584.

United States Consul Gilbert, at Liege, Belgium, says: "The exports of zinc dust collected at the several zinc works in Liege have largely increased. During the year 1896 the declared value of zinc dust for the American market was 35,826 dols. In 1897 it amounted to 105,000 dols. For the first quarter of 1897 such exports were 14,621 dols.; for the same quarter of 1898 the invoiced value was 28,156 dols. Zinc dust is employed in the United States for the manufacture of paints."

GERMAN PATENTS.

*Eng. and Mining J.*, *May* 14, 584.

In 1897 the German Patent Office showed great activity, the number of applications for patents having increased from 16,486 in 1896 to 18,347. In connection with mining, 43 patents were applied for and 26 granted; in ore dressing 46 were applied for and 23 granted. In connection with the metallurgy of iron, 36 patents were applied for and 13 granted; in connection with the metallurgy of other metals 104 were applied for and 36 granted.

### Gold Mining in Korea.

*Eng. and Mining J.* 1898, **65**, [9], 249.

A report by the United States Consul-General at Seoul upon the work in connection with the gold-mining concession granted to J. R. Morse by the Korean Government, states that the district is about 25 miles in width, and includes the country reputed to be the richest in gold of any in Korea. The concession is for a period of 25 years, and includes the right to mine any other minerals found in the district. A modern mill of 40 stamps is to be erected shortly, and although nothing has yet been done beyond prospecting on a large scale, veins of medium-grade ore have been opened, which give good prospects. The placers have been well worked over upon the surface by natives in the past, but the bedrock has not been reached. In working the quartz veins, the natives chip out the gold-bearing rock as much as possible with their soft iron tools, then fill the hole with fuel and set fire to the latter. The surface is then cracked by pouring in cold water, and the ore chipped off and crushed by means of large stone rollers. Water is the greatest obstacle, as the native miners do not possess pumps.

A mining concession has been granted to the Germans for a portion of the district adjoining the American concession, and the French also have the right to open certain mines.—A. S.

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 30th April. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,701,612 | 1,750,809 |
| Chemicals and dyestuffs | 608,029 | 535,301 |
| Oils | 522,568 | 614,103 |
| Raw materials for non-textile industries. | 3,307,117 | 3,441,017 |
| Total value of all imports | 35,136,555 | 40,246,716 |

### Summary of Exports.

| Articles. | Month ending 30th April. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 2,958,212 | 2,670,392 |
| Chemicals and medicines | 856,276 | 765,130 |
| Miscellaneous articles | 3,057,121 | 2,778,247 |
| Total value of all exports | 19,700,122 | 17,496,011 |

### Imports of Oils for Month ending 30th April.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Cocoa-nut | Cwt. | 17,802 | 25,773 | 18,949 | 28,531 |
| Olive | Tuns | 898 | 2,922 | 30,625 | 97,781 |
| Palm | Cwt. | 63,713 | 72,513 | 65,288 | 78,160 |
| Petroleum | Gall. | 12,990,838 | 13,972,963 | 236,699 | 228,434 |
| Seed | Tons | 3,230 | 2,867 | 63,626 | 52,779 |
| Train, &c. | Tuns | 1,095 | 706 | 19,997 | 12,460 |
| Turpentine | Cwt. | 12,342 | 33,566 | 11,849 | 41,575 |
| Other articles | Value £ | .. | .. | 75,540 | 74,383 |
| Total value of oils | | .. | .. | 522,568 | 614,103 |

### Imports of Metals for Month ending 30th April.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| Copper:— | | | | £ | £ |
| Ore | Tons | 2,432 | 5,346 | 24,923 | 66,901 |
| Regulus | „ | 6,480 | 5,954 | 177,901 | 156,921 |
| Unwrought | „ | 3,794 | 5,816 | 189,586 | 303,654 |
| Iron:— | | | | | |
| Ore | „ | 480,675 | 497,747 | 344,593 | 364,109 |
| Pig | „ | 11,906 | 6,822 | 34,157 | 19,525 |
| Bolt, bar, &c. | „ | 4,737 | 2,302 | 34,487 | 18,566 |
| Steel, unwrought | „ | 6,681 | 4,056 | 31,876 | 22,570 |
| Lead, pig and sheet | „ | 15,249 | 16,376 | 178,457 | 206,656 |
| Pyrites | „ | 49,512 | 64,320 | 81,877 | 102,162 |
| Quicksilver | Lb. | 1,179,708 | 400,070 | 113,813 | 37,373 |
| Silver ore | Value £ | .. | .. | 92,272 | 118,899 |
| Tin | Cwt. | 41,188 | 16,816 | 123,235 | 53,140 |
| Zinc | Tons | 6,262 | 6,376 | 109,187 | 107,778 |
| Other articles | Value £ | .. | .. | 165,239 | 172,555 |
| Total value of metals | | .. | .. | 1,701,612 | 1,750,809 |

### Imports of Chemicals and Dyestuffs for Month ending 30th April.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Alkali | Cwt. | 11,581 | 15,517 | 6,372 | 10,979 |
| Bark (tanners', &c.) | „ | 27,000 | 17,436 | 9,032 | 5,633 |
| Brimstone | „ | 29,015 | 36,386 | 5,519 | 8,205 |
| Borax | „ | .. | 416 | .. | 360 |
| Chemicals | Value £ | .. | . | 103,439 | 118,355 |
| Cochineal | Cwt. | 163 | 562 | 959 | 3,618 |
| Cutch and gambier | Tons | 2,158 | 1,361 | 39,440 | 24,802 |
| Dyes:— | | | | | |
| Alizarin | Value £ | .. | .. | 16,437 | 13,800 |
| Anilin and other | „ | .. | .. | 39,697 | 42,153 |
| Indigo | Cwt. | 11,575 | 6,053 | 210,746 | 90,878 |
| Nitrate of potash | „ | 35,258 | 31,123 | 24,779 | 23,687 |
| Valonia | Tons | 3,876 | 3,355 | 37,680 | 34,912 |
| Other articles | Value £ | .. | .. | 113,929 | 157,919 |
| Total value of chemicals | | .. | .. | 608,029 | 535,301 |

### Imports of Raw Material for Non-Textile Industries for Month ending 30th April.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Bark, Peruvian | Cwt. | 2,165 | 6,739 | 4,581 | 12,726 |
| Bristles | Lb. | 361,103 | 225,789 | 55,928 | 30,643 |
| Caoutchouc | Cwt. | 30,363 | 37,563 | 338,561 | 471,145 |
| Gum:— | | | | | |
| Arabic | „ | 5,506 | 2,594 | 10,379 | 5,263 |
| Lac, &c. | „ | 20,538 | 12,752 | 97,327 | 42,810 |
| Gutta-percha | „ | 3,442 | 2,953 | 30,306 | 29,835 |
| Hides, raw:— | | | | | |
| Dry | „ | 68,423 | 67,262 | 180,106 | 183,930 |
| Wet | „ | 42,237 | 41,371 | 94,602 | 87,503 |
| Ivory | „ | 1,093 | 1,122 | 44,463 | 38,862 |
| Manure:— | | | | | |
| Guano | Tons | 350 | 795 | 2,650 | 4,734 |
| Bones | „ | 6,111 | 5,643 | 22,586 | 25,065 |
| Nitrate of soda | „ | 12,958 | 23,983 | .. | .. |
| Phosphate of lime | „ | 32,930 | 28,569 | .. | .. |
| Paraffin | Cwt. | 66,617 | 98,285 | 55,608 | 80,975 |
| Linen rags | Tons | 1,873 | 1,639 | 19,801 | 16,832 |
| Esparto | „ | 10,025 | 23,751 | 40,688 | 84,689 |
| Pulp of wood:— | | | | | |
| Chemical | „ | 25,610 | 14,619 | 131,091 | 97,240 |
| Mechanical | „ | | 18,440 | | 54,155 |
| Rosin | Cwt. | 67,541 | 77,700 | 16,518 | 16,243 |
| Tallow and stearin | „ | 102,008 | 207,796 | 99,162 | 203,622 |
| Tar | Barrels | 2,839 | 2,422 | 1,972 | 1,494 |
| Wood:— | | | | | |
| Hewn | Loads | 231,547 | 164,964 | 461,351 | 388,425 |
| Sawn | „ | 222,569 | 166,485 | 551,452 | 417,309 |
| Staves | „ | 8,573 | 6,874 | 53,633 | 43,644 |
| Mahogany | Tons | 5,281 | 3,740 | 52,853 | 33,683 |
| Other articles | Value £ | .. | .. | 791,516 | 852,486 |
| Total value | | .. | .. | 3,307,117 | 3,441,017 |

Besides the above, drugs to the value of 87,950*l*. were imported, as against 67,317*l*. in April 1897.

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 30TH APRIL.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 7,771 | 8,829 | 35,520 | 39,259 |
| Copper :— | | | | |
| Unwrought.... „ | 28,087 | 42,135 | 73,199 | 110,783 |
| Wrought ...... „ | 24,686 | 20,440 | 76,414 | 62,092 |
| Mixed metal... „ | 18,775 | 18,725 | 46,108 | 44,599 |
| Hardware and cutlery. Value £ | .. | .. | 186,425 | 157,840 |
| Implements ..... „ | .. | .. | 113,532 | 102,968 |
| Iron and steel ... Tons | 331,038 | 282,046 | 2,170,121 | 1,903,462 |
| Lead ............ „ | 2,683 | 2,545 | 36,801 | 36,397 |
| Plated wares ... Value £ | .. | .. | 30,328 | 27,477 |
| Telegraph wires . „ | .. | .. | 75,688 | 64,883 |
| Tin ............ Cwt. | 8,628 | 9,168 | 27,650 | 30,988 |
| Zinc ............ „ | 12,092 | 13,235 | 9,123 | 11,660 |
| Other articles .. Value £ | .. | .. | 77,297 | 77,384 |
| Total value ....... | .. | .. | 2,958,212 | 2,670,392 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 30TH APRIL.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 477,566 | 297,958 | 125,849 | 75,017 |
| Bleaching materials „ | 88,184 | 97,809 | 28,076 | 27,950 |
| Sulphate of copper Tons | 10,410 | 11,081 | 176,699 | 177,890 |
| Chemical manures „ | 34,239 | 320,108 | 169,683 | 165,076 |
| Medicines....... Value £ | .. | .. | 91,615 | 86,882 |
| Other articles... „ | .. | .. | 264,354 | 232,315 |
| Total value .......... | .. | .. | 856,276 | 765,130 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH APRIL.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 633,200 | 831,100 | 11,966 | 21,195 |
| Military stores.. Value £ | .. | .. | 154,840 | 230,208 |
| Candles.......... Lb. | 1,902,100 | 2,080,700 | 26,354 | 27,318 |
| Caoutchouc ..... Value £ | .. | .. | 95,063 | 105,193 |
| Cement.......... Tons | 25,908 | 29,783 | 41,817 | 56,491 |
| Products of coal Value £ | .. | .. | 179,135 | 130,861 |
| Earthenware ... „ | .. | .. | 171,591 | 132,818 |
| Stoneware ...... „ | .. | .. | 20,184 | 13,516 |
| Glass :— | | | | |
| Plate........... Sq. Ft. | 114,296 | 105,717 | 7,770 | 5,667 |
| Flint........... Cwt. | 9,304 | 5,712 | 19,650 | 14,866 |
| Bottles......... „ | 85,338 | 68,485 | 40,511 | 32,218 |
| Other kinds.... „ | 25,690 | 16,535 | 21,652 | 13,869 |
| Leather :— | | | | |
| Unwrought .... „ | 13,325 | 12,987 | 117,719 | 124,490 |
| Wrought ..... Value £ | .. | .. | 30,487 | 29,046 |
| Seed oil.......... Tons | 5,644 | 4,195 | 92,849 | 70,596 |
| Floorcloth ..... Sq. Yds. | 2,672,500 | 2,086,100 | 105,435 | 88,782 |
| Painters' materials Val. £ | .. | .. | 138,322 | 135,130 |
| Paper ........... Cwt. | 96,524 | 76,490 | 149,227 | 119,553 |
| Rags............ Tons | 4,432 | 4,557 | 25,988 | 21,221 |
| Soap ............. Cwt. | 65,989 | 63,187 | 70,791 | 61,830 |
| Total value .......... | .. | .. | 3,057,121 | 2,778,247 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

8942. H. Todd and R. Cotton. An improved method of firing ovens and kilns. April 18.

9005. H. Rockling. Improvements in or relating to apparatus for compressing coal or similar material. Complete Specification. April 18.

9029. A. Goddard. Improvements in mechanical digesters. April 19.

9143. W. Pilkington. Improvements relating to metallic cylinders or bottles for containing gas under high pressure, and for other purposes. Complete Specification. April 20.

9206. G. W. Holmes and J. Fieldhouse. Improvements in filter press plates. April 21.

9580. G. H. Jones. A combined water-purifier and filter. April 26.

9582. W. H. Wheatley.—From C. Mendelson, United States. Apparatus for carbonating liquids. Complete Specification. April 26.

9585. W. Flecken. An improved process of and apparatus for drying superphosphate. April 26.

9605. I. Fokchaner. Improvements in filters for water and other liquids. Complete Specification. April 26.

9606. I. Fokchaner. Improvements in filters. Complete Specification. April 26.

9874. W. Bryant. Improvements in kilns or furnaces. April 29.

9926. C. Chester and F. A. Keil. Improvements in apparatus for drying and pulverising colouring matters, starch, and other materials and substances. April 30.

10,038. O. W. Ackermann. Improvements in apparatus for carbonating liquids. May 2.

10,100. A. J. Boult.—From The Nitrogen Processing Company, United States. Improvements in or relating to drying and pulverising apparatus. May 3.

10,132. P. A. Newton.—From The Baker and Adamson Chemical Company, United States. An improved apparatus for distillation of acids and other corrosive substances. Complete Specification. May 3.

10,257. G. Daimler. Improvements in water-cooling apparatus. May 5.

10,410. J. Week. Improvements in sterilising apparatus. May 6.

10,509. C. W. Ramsay. Improvements in or relating to the distillation or treatment of liquids. May 7.

10,517. B. Watson. Improvements in centrifugal separators. May 9.

10,648. J. M. Tavernor and W. A Thornton. Improvements in or relating to governors for controlling the flow and pressure of acetylene and other gas. May 10.

10,673. W. J. Napier, C. Sealy, and B. Malcolm. Improvements in or relating to refrigerating machines or apparatus. May 10.

10,693. E. Theisen. Improvements relating to the distillation, evaporation, and extraction of liquids. May 10.

10,759. L. Lewis. An improved composition for preventing incrustation in steam boilers. May 11.

10,764. A. R. Bennett. Improvements in brick cement and like kilns. May 11.

10,774. A. Skassa. Improvements in and relating to distilling and rectifying apparatus. Complete Specification. May 11.

10,775. H. A. Desrumaux. Improvements in filters. May 11.

10,812. F. W. Golby.—From E. Oulmiére, France. Improvements in apparatus for purifying water and other liquids. May 11.

10,882. L. Seeger. Improved freezing or refrigerating apparatus. May 12.

10,892. C. Mallinson. Improvements in or relating to apparatus for conditioning or drying grain berries, sugar, seeds, crystals, and granular or pelverulent material generally. May 12.

11,062. R. L. Mond. Improvements in the heating of fluids. May 14.

### Complete Specifications Accepted.*

1897.

11,450. H. McPhail. Method of and means for evaporating or drying viscous substances, semi liquids, and pasty materials. April 27.

11,451. H. McPhail. Method of boiling and evaporating liquids, and of digesting materials in liquids. April 27.

13,353. W. N. Twelvetrees. Cleansing, drying, and disinfecting apparatus. May 4.

13,983. W. Schmidt. Construction of tubes for cooling and heating purposes, specially adapted for brewers, dyers, dairies, and other trade uses, also for condensing steam and vapours. April 27.

14,387. F. C. Roberts. Furnace tuyères. April 27.

14,778. H. Mawhood. A new system of reverberatory furnaces, with retorts for calcining divers products. May 11.

14,896. F. H. Addis. Apparatus for cooling air. April 27.

14,933. R. Kurka. Filters. April 27.

15,106. S. C. Davidson. Apparatus for the drying, or partially drying, or other treatment of tea leaf, tobacco leaf, coffee, grain, malt, fruits, and other substances. May 18.

16,836. J. Johnson. Worm liqueur cooler and refrigerator. May 4.

21,311. G. T. Holloway. New or improved laboratory gas furnace. May 11.

1898.

3926. E. C. Segundo. Apparatus for sterilising milk and other nutritious substances. May 4.

4459. L. Rümelin and H. Auer. Improved receptacle for the storage of calcium carbide. April 27.

6837. B. H. Thwaite and F. L. Gardner. Method and apparatus for separating dust from blast-furnace gases. April 27.

7024. F. Hugershoff. Centrifugal liquid precipitating or separating apparatus. April 27.

7590. J. M. Thèvenet-le-Boul. Metallic capsules for storing compressed or liquefied gases. May 4.

8238. J. Mittwich. New or improved filtering apparatus. May 18.

## II.—FUEL, GAS, AND LIGHT.

### Applications.

9023. W. H. Dargue. Improvements in the generation of acetylene gas. April 19.

9075. J. Büeb. Improvements in the extraction of cyanogen from coal-gas. April 19.

9104. F. S. Thorn and C. Hoddle. A new and improved apparatus for acetylene purifying. April 20.

9240. S. Chandler, jun., and J. Chandler. Improvements in apparatus used in the manufacture of gas from oil or other fluids. April 21.

9269. W. P. Thompson.—From J. Grand, France. An improved means for producing acetylene gas. April 21.

9319. A. Javal. Improvements in apparatus for the production of acetylene gas. April 22.

9384. J. G. A. Kitchen. Improvements in generators for producing acetylene from calcium carbide and water. April 23.

9462. P. Naef. Process and apparatus for producing ammonia, cyanides, coke, and gas. Filed 25 April. Date applied for 25 Sept., being date of application in United States.

9545. A. Williamson. Improvements in acetylene gas generators. Complete Specification. April 26.

9594. M. J. Silver. Improvements in or relating to mantles for incandescent gas burners. April 26.

9596. N. Paquet. Improved manufacture of blocks or briquettes of coal. April 26.

9619. W. Westlake. Improvements in or in connection with feeding and burning pulverised fuel. April 26.

9718. M. Strakosch and F. Schmid. Improvements in or relating to apparatus for producing and burning acetylene gas. April 27.

9750. W. A. P. Werner. Improvements in and relating to acetylene generators. April 28.

9772. W. Blackie. Improvements in apparatus for the generation of oxygen gas. April 28.

9920. W. Ireland. Improvements in acetylene gas generators. April 30.

9954. M. Taylor. Improvements in gas producing apparatus. April 30.

10,008. I. Loeser and A. Lecomte. Improved gas burner for incandescent lighting. Complete Specification. May 2.

10,056. I. Saxl. Improvements in controlling apparatus for acetylene generators. May 2.

10,114. C. Barnett. Apparatus for the automatic production of acetylene gas. May 3.

10,119. A. E. Alexander.—From J. R. Hatmaker, United States. Method of and means for utilising fuel. May 3.

10,141. R. Haddan.—From F. Vörös, Hungary. Improvements in apparatus for producing acetylene gas. May 3.

10,188. P. Barré. Acetylene gas producing apparatus. May 4.

10,305. J. H. Schülke. Improved acetylene gas generator. Complete Specification. May 5.

10,502. P. R. de F. d'Humy. Improvements in the manufacture of oil and peat fuel, and in apparatus therefor. May 7.

10,522. D. A. Casalonga. Improvements in or relating to the treatment of wood for the manufacture of charcoal. Complete Specification. Filed May 7. Date applied for Oct. 8, 1897, being date of application in France.

10,571. L. Maurice. Accumulators of heat. May 9.

10,692. J. W. Dawson.—From J. Lounge, United States. Improvements in and relating to apparatus for the generation and storage of acetylene gas. May 10.

10,746. H. Simon. Improvements in and connected with valves used in plant for the manufacture of gas. Complete Specification. May 11.

10,763. J. Sartig. Treating calcium carbide for use in acetylene generators. Complete Specification. May 11.

10,815. F. Windham. Improvements in or relating to the burning of acetylene gas. May 11.

10,906. G. Rothgiesser. Improved process for mixing combustible gases with air. Complete Specification. May 12.

10,938. J. Varon. Improved acetylene gas producing apparatus. May 13.

10,984. F. Murray.—From Sir C. S. Forbes, Bart., France. Improved apparatus for the generation and supply of acetylene gas. May 13.

### Complete Specifications Accepted.

1897.

10,508. E. Evans. Apparatus for generating acetylene or other gas generated by the addition of a liquid to a solid. April 27.

10,686. J. J. Patterson. Apparatus for generating acetylene. May 4.

11,644. H. Sutcliffe. Gas generators. May 11.

13,191. F. G. Bartlett. Burners employed in incandescent gas lighting. April 27.

14,259. H. Kayser. Manufacture of incandescent bodies for gas and other lighting. May 11.

14,313. G. W. Gaskill and R. F. Reeve. Apparatus for generating gas by the addition of a solid to a liquid. May 11.

14,594, J. B. I. Julhe. Means or apparatus for supplying and burning petroleum or other inflammable liquids for illuminating purposes. May 11.

14,835. G. W. Gaskell and R. F. Reeve. Acetylene gas-light apparatus applicable for use in photographic studios for photographing at night. May 11.

15,060. C. Kaestner. Acetylene gas burners. April 27.

16,793. O. V. Sigurdsson. Means employed in the manufacture of acetylene gas. May 11.

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* *See* Note (*) on previous page.

21,464. J. Windmüller, of the firm of Windmüller and Co. Improved device for producing and burning acetylene gas. May 11.

28,258. L. J. Keck. Lamp for generating and burning acetylene gas. May 11.

29,571. H. de Thiersant and W. A. Coulson. Acetylene gas producer. April 27.

1898.

4529. J. B. Bomsean. A cartouche or receiver to store gas under pressure. April 27.

4619. C. W. Smith. Apparatus for producing artificial fuel. May 4.

5498. L. P. Hvüd. Apparatus for generating and utilising acetylene. April 27.

8723. J. W. Turner and C. Jousset. Block fuel. May 4.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Complete Specifications Accepted.

1897.

13,665. W. Young and J. Fyfe. Retorts for the destructive distillation of shale. April 27.

## IV.—COLOURING MATTERS and DYES.

### Applications.

9338. B. Willcox.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of colouring matters for cotton. April 22.

9435. M. Prud'homme. A process for preparing diazotisable and non-diazotisable compounds by means of primary aromatic monoamines and aromatic diamines, and the manufacture of colouring matters derived therefrom. April 23.

9506. O. Imray.—From Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of colouring matters from tetranitro-anthrachrysone. April 25.

9690. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. A process for producing ethereal salts of indoxyl-carboxylic acid. April 30.

9792. C. Goldschmidt. The manufacture of methenyldi-para- and ortho-phenetidine and anisidine. April 28.

10,315. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis Albin Haller, and Alfred Guyot, France. Processes for converting substitution derivatives of amidobenzoylbenzoic acids and substitution derivatives of oxy-amidobenzoylbenzoic acids into corresponding anthracene derivatives. May 5.

10,516. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of o- and *p*-amidobenzylideneaniline their homologues and their sulphonic acids and of amidobenzaldehydes therefrom. May 7.

10,590. H. E. Newton.—From The Farbenfabriken vormals Friedrich Bayer and Co., Germany. A new process for producing hydroxy compounds of the aromatic series. May 9.

10,597. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new mordant-dyeing colouring matters. May 9.

11,003. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of ortho- and para-nitrobenzeaniline sulphonic acids and their homologues. May 13.

11,077. F. Hauff. Process for the production of orthotoluol sulpho acid from orthothuocresol by oxidation with the aid of concentrated nitric acid. May 14.

11,078. F. Hauff. Process for the production of orthotoluol sulpho chloride by the action of chlorine on orthotoluol sulphonic acid salts in presence of phosphorous, and a suitable dissolvent for the last-named, for example, bisulphide of carbon. May 14.

### Complete Specifications Accepted.

1897.

12,011. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co. The manufacture or production of new alizarine dyestuffs. May 4.

13,999. R. Holliday and Sons, Ltd., J. Turner, and H. Dean. Manufacture of naphthylamine derivatives and colouring matters therefrom. May 11.

14,132. H. R. Vidal. Manufacture of colouring matters. April 27.

14,917. M. Otto. Manufacturing of colouring or carbinol matters. May 18.

14,918. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Manufacture and production of black colouring matters of the anthra quinone series. May 18.

14,919. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of yellow colouring matters from benzoin. May 18.

14,920. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Manufacture and production of asyonmetric diamidophenyl acridines and their alkyl substitution products.

14,921. J. Y. Johnson. From The Badische Anilin and Soda Fabrik, Germany. Manufacture and production of green mordant-dyeing colouring matters from halogen substituted fluoresceines. May 18.

1898.

5456. F. Valentiner. Production of acetophenonphenetidine. April 27.

5949. G. W. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or production of scanthines and their derivatives. April 27.

6055. J. Imray.—From La Société L. Durand, Huguenin et Cie., France. New colouring matters from oxazines. May 4.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

10,592. H. Ferguson. Improvements in the process and machinery for breaking and scutching fibrous plants and stems such as brown rhea, ramie hemp, jute and the like. Complete Specification. May 9.

10,631. J. Newton. An improved method of and means for mercerising cotton cloth. May 10.

10,708. W. Hall. Improvements in heating or mercerising cotton and other fibres to impart a silky or lustrous appearance thereto. May 10.

10,736. J. Hunter. Improved device and process for mercerising cotton yarns. May 11.

10,757. J. Miller, sen., and J. Miller, jun. Improvements in the method of and apparatus for rendering textile fabrics waterproof by one treatment or process. May 11.

### Complete Specifications Accepted.

1897.

15,642. C. W. Radeke. Treatment of textile vegetable fibres. May 18.

19,256. W. Baldwin. Process of and means for sizing yarn. May 11.

22,477. F. Holtschmidt. Manufacture of fabrics. May 18.

29,766. J. V. Esop. Process for rendering fabrics and other materials acid proof. May 11.

1898.

881. C. Geige. Process for the manufacture of chemically prepared peat fibres. May 11.

3898. H. C. Woltereck. Manufacture of artificial silk. May 11.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

9255. A. G. Green and the Clayton Aniline Company, Ltd. Improvements in dyeing vegetable fibres. April 21.

9484. F. Haber. Improvements in mordanting. Complete Specification. April 25.

9885. J. R. Hope, W. T. Galey, and T. McConnell. Improvements in machines for treating yarns for mercerising, dyeing, and the like. Complete Specification. April 29.

10,205. R. Brandts. Apparatus for dyeing and similarly treating textile rovings. Complete Specification. May 4.

10,264. C. Hartley. Improvements in and connected with apparatus for dyeing, scouring, bleaching, and otherwise treating yarn in cop or other compact form. May 5.

10,265. C. Hartley. Improvements in apparatus for dyeing, scouring, bleaching, and otherwise treating yarn in cop or other compact form. May 5.

10,550. S. Wardleworth. Improvements in colouring paper. May 9.

10,596. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. A method of dyeing half wool with basic azo dyestuffs in an acid bath. May 9.

### Complete Specifications Accepted.

1897.

12,940. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik. Dyeing vegetable fibre with indigo. May 18.

13,916. B. Willcox.—From S. Wallach and Co. and C. Schoer Fixing insoluble colouring matters on fibres or fabrics. May 18.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

9003. D. A. Peniakoff. Improvements in the manufacture of caustic alkalies by means of alkaline aluminates. April 18.

9073. E. G. Scott. Improvements in the manufacture or production of acetic acid. April 19.

9211. J. Plummer. Improved process for the manufacture of chlorine and caustic soda. April 21.

9464. P. Naef. Process and apparatus for calcining limestones and other substances, and recovering valuable by-products. Filed April 25. Date applied for, Sept. 25, 1897, being date of application in United States.

9577. A. E. Alexander.—From J. R. Hatmaker, United States. Process for obtaining pure sodium chloride from crude or natural brines. April 26.

9730. F. Henkel. Process for the manufacture of solid but easily soluble alkaline silicate. Complete Specification. April 27.

9831. J. Armstrong. Improvements in the manufacture of caustic alkali, and in obtaining of by-products. April 28

9904. T. C. Lowden. Improvements in or relating to the manufacture of salt. April 29.

10,021. J. E. T. Woods and W. A. Byrom. Improvements in the manufacture of calcium carbide and other compounds by hydrogen. May 2.

10,290. C. S. Bradley and C. B. Jacobs. Improvements in process of manufacturing phosphorus and other materials. Complete Specification. May 5.

10,823. W. A. Harper and J. L. F. Vogel. Improvements in the production of zinc and chlorine. May 12.

### Complete Specifications Accepted.

1897.

12,274. S. Rosenblum, S. Rideal, and The Commercial Ozone Syndicate, Ltd. Manufacture of hydrogen peroxide, and apparatus therefor. May 18.

12,817. F. Blum. Process for production of divisional products of halogen-albumen by means of alkalis. May 11.

12,952. E. G. Scott. Manufacture of acetic acid. May 4.

13,519. W. Feld. Process and apparatus for obtaining barium oxide and barium hydroxide from barium carbonate. May 11.

1898.

4033. J. Landin. Method of producing carbides. May 11.

4298. P. Jensen.—From J. von Orlowsky, Russia. Process for rendering carbide non-hygroscopic. May 11.

6037. H. H. Scow. Valves for regulating the flow of gases or liquids. April 27.

## VIII.—GLASS, POTTERY, and ENAMELS.

### Applications.

10,128. G. W. Tooker. Improvements relating to the gilding of articles of china, earthenware, porcelain, and the like. Complete Specification. May 3.

10,807. J. T. Newell. Improved treatment of glass for coating purposes as a backing for slabs, tiles, facing plates, and the like, and for light reflection and other purposes. Complete Specification. May 11.

### Complete Specifications Accepted.

1897.

12,759. W. Wachter. Method of transferring metachrome types upon ceramic objects before glazing the latter. April 27.

1898.

6203. F. Vitte, senr. Processes of apparatus for manufacturing plate or sheet glass. May 18.

6632. H. Leek. Means of supporting pottery ware in kilns or ovens while being baked or fired. May 11.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, and CEMENTS.

### Applications.

8966. H. Porter and R. Wyatt. A combined new system of rendering buildings fire-proof, of extinguishing fire in the same, and also of ventilating the same. April 18.

9181. H. L. Cunnah and H. A. Hughes. Improvements in and connected with fire-proof and other floors and ceilings. Complete Specification. April 20.

9265. G. C. Levey and C. L. O'Brien. Improvements in the manufacture of artificial stone, tiles, or the like. April 21.

9476. E. Graham. Improvements in the process of manufacturing wood-panels, fancy wood-work boards, and all kinds of materials suitable for the purposes of building and decorating. April 25.

9632. G. C. Marks.—From L. Marchand and A. M. L. Perin, France. An improved process and apparatus for converting gypsum or like materials into plaster of Paris used for modelling and other purposes. April 26.

9695. B. J. B. Mills.—From J. Morel, France. A new or improved artificial marble and other translucent artificial products, and processes employed in the production thereof. April 27.

9934. T. Holden and C. Brooke. Improvements in plastic compositions for decorating walls, ceilings, and other surfaces in relief. April 30.

10,046. G. Lilienthal. Improvements in the construction of fire-proof floors. May 2.

10,425. M. Bachert and D. W. O'Neil. Improvements in impregnating wood. Complete Specification. Filed May 6. Date applied for March 1, 1898, being date of application in United States.

10,527. P. Wernicke. Process of and apparatus for depositing colours for the patterns of cement and other slabs. Complete Specification. May 7.

10,658. W. R. Johnson and G. Nichols. Improvements relating to gypsum or plaster of Paris. May 10.

10,917. W. Thomlinson. The utilisation of "flue-dust" by incorporating it into briquettes either alone in its natural state, or in a concentrated state, or in conjunction with finely divided iron ores or similar substances, such as pyrites, residues, purple ore, and the like, for use in the blast furnace. May 13.

### Complete Specifications Accepted.

1897.

10,317. W. F. Dyson. Artificial stone. May 4.

14,299. A. S. Elbers. Treating blast-furnace slag for use in cement, mortars, or the like. May 18.

1898.

1769. P. Kleber. Process for the manufacture of fireproof building stones, and for the production of cement from the same. May 18.

## X.—METALLURGY, MINING, Etc.

### Applications.

8965. J. B. de Alzugaray. Improvements in and connected with the refining and manufacture of iron, steel, and other metals and alloys. April 18.

9027. J. Powell, D. Colville, jun., J. Jardine, and T. B. Mackenzie. Improvements in apparatus for charging materials into open-hearth steel-melting furnaces. April 19.

9153. A. Cochn and E. Salomon. Improvements relating to the separation of cobalt from nickel and other metals. April 20.

9212. J. Plummer. Process for producing aluminium. April 21.

9367. W. J. Foster. Improvements in and in means or apparatus for use in the manufacture of iron and steel. April 23.

9463. P. Naef. Smelting ores and recovering valuable by-products. Filed 25 April. Date applied for 25 Sept. 1897, being date of application in United States.

9664. Mrs. L. Bloume, *née* M. Humbert. Amalgamating process and apparatus. Filed April 27. Date applied for Oct. 30, 1897, being date of application in Belgium.

9717. F. W. Minck. Improvements in or connected with smelting furnaces for metallic alloys. Complete Specification. April 27.

9956. T. T. Draper and W. Ryley. An improved process and apparatus for extracting the liquid obtained by the treatment of metalliferous slime, pulp, or sand with solvents. April 30.

10,639. A. Homer. An improved solder. May 10.

10,671. A. K. Huntingdon and R. T. Preston. A process for preventing solution of zinc in alloys containing it. May 10.

10,823. W. A. Harper and J. L. F. Vogel. *See* Class VII.

10,864. J. Armstrong. Improvements in extracting metals from sulphide, carbonate, refractory, or other ores or matter, and apparatus therefor. May 12.

11,001. H. L. Sulman. Improvements in obtaining gold from certain of its ores. May 13.

11,002. C. H. Pead. Improvement in separating liquids from fine slimes of gold-bearing and other ores, and apparatus therefor. Complete Specification. May 13.

11,004. H. L. Sulman. Improvements in the precipitation of gold and silver from their cyanide solutions. May 13.

11,011. F. A. Edwardes. Improvements in apparatus for use in the treatment of metallic ores. May 14.

11,051. The Cowper-Coles Galvanising Syndicate, Ltd., and S. O. Cowper-Coles. Improvements in apparatus for use in the electro-deposition of zinc or other metals. May 14.

### Complete Specifications Accepted.

1897.

10,403. J. Colley. Linings of metallurgical vessels. May 4.

11,151. G. H. Blenkinsop. Roasting and calcining of minerals and the like substances. April 27.

12,325. J. C. Bull. Alloys. April 27.

13,308. J. Parry and G. H. Llewellyn. Manufacture of steel. May 11.

14,007. B. K. Jamison. Apparatus employed in the face-hardening of armour-plates or other articles of steel or of steel alloyed with other metals. May 11.

29,574. W. Wood. Production or formation of hollow metal castings. May 11.

30,934. J. T. Wainwright. Art of manufacturing steel armour plate. May 18.

1898.

1027. T. Odlum. Welding compounds.

2293. J. T. Wainwright. Apparatus for exhausting molten metals. May 11.

3296. C. V. Burton. Process for tinning and soldering aluminium. April 27.

3807. G. F. Smith. Apparatus for extracting precious metals by chlorination. May 11.

4296. J. Jones. Process for the recovery of zinc from refractory zinc-bearing ores. May 18.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

8988. C. Alker and P. Mennessier. Improvements in electrodes for accumulators. April 18.

9002. J. Brock and The United Alkali Company, Ltd. Improvements in or connected with apparatus for the manufacture of potassium chlorate or sodium chlorate by electrolysis. April 18.

9105. F. Greenfield. Improvements in apparatus to be used in connection with the electro-deposition of metals. April 20.

9218. G. E. B. Pritchett, T. W. Pritchett, and A. G. Gold. Improvements in secondary batteries. April 21.

2637. J. W. Richards and C. W. Roepper. Improvements in process for the production of chemical compounds by electrolysis. Complete Specification. April 26.

9638. J. W. Richards and C. W. Roepper. Process for manufacturing metallic sulphides electrolytically. Complete Specification. April 26.

9739. F. H. Bowman and F. E. Bowman. Improved means and apparatus for electrolytically decomposing salts of the alkaline and earthy metals, or other substances containing them, for the manufacture of caustic bleaching powder, chlorine, or other products contained in them. April 28.

9778. M. Bernstein, Proprietor of the Firm of J. F. Wallmann and Co. Improvements in plates for electric accumulators. Complete Specification. April 28.

10,034. A. Fraser and G. A. Smith. A new or improved primary electrical battery. May 2.

10,094. A. C. Croftan. Improvements in the art of making plates for electric battery purposes. Complete Specification. May 3.

10,457. E. Habermann. Improvements in galvanic batteries. May 7.

10,475. W. Peck. Improvement in electrical accumulators. May 7.

10,710. J. Weiss. An improved dry battery. May 10.

10,805. F. H. Perry. Improvements in primary batteries. May 11.

10,960. J. B. Scammell. Improvements in electrical storage batteries or accumulators. May 13.

11,068. H. H. Lake.—From A. Lotti, Italy. Improvements in the production of zinc and sulphate of copper by electrolysis. May 14.

### Complete Specifications Accepted.

1897.

10,484. J. L. Dobell. Electric batteries. April 27.

10,719. W. Rowbotham. Primary batteries. May 11.

12,675. R. Krayn and C. Koenig. Galvanic batteries. May 11.

15,047. A. Heinemann and W. Schäfer. Electric accumulators or storage batteries. May 11.

20,145. A. Lehmann and A. Mann. Accumulator plates. May 11.

23,329. G. Platner. Substances to prevent polarisation in electric batteries. May 4.

1898.

2967. H. L. Haas. Electro-depositing anodes. May 4.

5912. G. Laura. Electrical galvanic batteries. May 4.

## XII.—FATS, OILS, AND SOAP.

### APPLICATIONS.

9252. J. N. Harris and The Globe Trading Company, Ltd. Improvements relating to the treatment of edible oils and fats of various kinds. April 21.

9599. Countess R. de Diesbach (*née* J. de B. Souvagny). Improvements in the production or extraction of glycerin from waste products. Complete Specification. April 26.

9824. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in or relating to the refining and decolorising of cotton-seed oil and other vegetable oil. April 28.

9883. T. Pate, T. H. Thompson, G. Duxbury, and J. H. Whittaker, trading as The Thoreclean Soapworks Company. A preparation or composition for cleansing or scouring wool, cotton, linen, silk, and the like, or goods made therefrom. April 29.

10,875. L. O. Helmers. Improvements in and relating to sulphur compounds of mineral oils and similar hydrocarbons. May 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

9529. S. Rosenblum, S. Rideal, and The Commercial Ozone Syndicate, Ltd. Oxidising of oils and apparatus for the purpose.

12,456. R. T. Cardell. Improvements in soaps and all toilet preparations. April 27.

30,684. C. G. Hepburn. Apparatus for treating fats and oils. April 27.

1898.

3585. G. Pommerhanz, C. Ristic, and V. Noback. Production of fats suitable for lubricating purposes. May 4.

4741. H. H. Lake.—From E. Twitchell, United States. Separation of fats or oils into glycerin and fatty acids, and compounds for use therein. May 4.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, ETC.

### APPLICATIONS.

10,263. T. Turnbull. Anti-corrosive damp-resisting paint. May 5.

10,491. J. Nead. An improvement in the manufacture of white lead. May 7.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

8510. C. A. Hakanson, J. H. Hakanson, and A. R. Hakanson. Paint or composition to be used as a coating, more especially in the manufacture of artificial slates, and for coating tiles, plates, and other articles. May 11.

14,439. H. Edmunds. Manufacture or production of an insulating material, and the application thereof to the insulation of electrical conductors.

1898.

6344. W. Ramsay. Treatment of gutta-percha and like substances. April 27.

6968. P. L. Clark. Reclaiming rubber. April 27.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### APPLICATIONS.

8952. J. Muir. Improvements in the treatment of hides or skins for use in the manufacture of leather tires and other purposes. April 18.

10,814. F. Hepburn. Improvements in vegetable glues, sizes, or gelatines. May 11.

10,999. J. Hall. Improvements in and in connection fish with the manufacture and production of materials to be employed as substitutes for wood, leather, and other substances. May 13.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

14,052. J. E. Chevallot. New iron compounds and their application to the preparation of leather. April 27.

14,076. C. J. Dunn. Tanning of leather. May 18.

## XV.—AGRICULTURE AND MANURES.

### APPLICATION.

9033. C. G. T. Dixon, J. Crone, D. C. Taylor, and F. Williams. Improvements in and in the manufacture of chemical manure. April 19.

## XVI.—SUGARS, STARCHES, GUMS, ETC.

### APPLICATIONS.

9079. A. Classen. Manufacture of non-coagulable albuminous bodies from coagulable albuminous bodies, and from fibrin, and processes therefor. April 19.

9325. A. B. Greig and J. Wilson. Improved method of and means or apparatus for treating bagasse. April 22.

9979. E. Casper. Improvements in refining raw sugar. April 30.

10,595. C. D. Abel. The Actien-Gesellschaft für Anilin Fabrikation, Germany. The manufacture of soluble albuminous preparations. May 9.

### COMPLETE SPECIFICATION ACCEPTED.

1898.

4532. W. Painter. Industrial compounds involving the use of gluten. May 4.

## XVII.—BREWING, WINES, SPIRITS, ETC.

### APPLICATIONS.

9615. J. Effront. Improved process for fermenting amylaceous substances. April 26.

9927. C. Chester and F. A. Keil. An improvement in the process of malting grain. April 30.

10,374. M. A. Russell. An improved method of treating malt and cereals for the preparation of malted and other meal, and the like. May 6.

10,509. C. W. Ramsay. *See* Class I.

10,633. J. F. Adams. An improved method of manufacturing beer. May 10.

10,809. H. G. Turner. Compounds for use as ferments for the production of alcohol from farinaceous matter. May 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

11,515. J. H. Folkerts. Production of yeast and apparatus therefor. April 27.

16,836. J. Johnson. *See* Class I. May 4.

25,140. C. Ameye. Process and apparatus for sterilising and saturating with carbonic acid gas liquids contained in casks or like vessels. May 18.

27,858. E. B. Ludlow. Appliance for aërating, rousing, and mixing, fermenting, and other liquids, or for lifting the same. May 11.

## XVIII.—FOODS, SANITATION, ETC., AND DISINFECTANTS.

### APPLICATIONS.

#### *A.—Foods.*

10,487. T. G. Whitehouse. Improvements in and relating to foodstuffs. May 7.

10,903. A. Bernstein. Improvements in the manufacture of food products from milk. May 12.

#### *B.—Sanitation.*

8982. W. Shedlock. Improvements in the treatment of fish, fish-offal, and similar substances, and in apparatus therefor. April 18.

9201. C. Chester and F. A. Keil. Improvements in the method of and apparatus for the destructive distillation of sewage sludge and other waste matter and refuse. April 21.

10,157. C. Schierholz. Automatic apparatus for purifying water. May 3.

10,427. W. M. Ducat. Improvements in or connected with filters for the purification of sewage and other foul liquids by bacterial action. May 6.

10,471. J. Y. Johnson.—From G. C. Purvis, Cape Colony. Improvements in the filtration of sewage or impure waters. May 7.

11,015. J. Jones. Improvements relating to the treatment of night soils, town refuse, or the like material, for the production of cement, papier-mâché, artificial stone, and the like, and for the extraction of grease, glass, metals, and alloys therefrom. May 14.

### *C.—Disinfectants.*

9658. J. W. Dougal and J. Nimmo. Improved manufacture of nitrogenous charcoal. April 27.

9659. J. W. Dougal and J. Nimmo. Improved manufacture of filtering charcoal. April 27.

### Complete Specifications Accepted.

### *A.—Foods.*

1897.

12,731. R. G. Nash. Aeration and preservation of milk, and apparatus therefor. April 27.

26,628. A. Schmidt. Process for the production of food for cattle. April 27.

### *B.—Sanitation.*

1897.

14,058. W. J. Lomax and T. Aspinall. Treatment and purification of sewage and other polluted waters and liquids. May 11.

26,625. H. G. Downton and W. H. Nicholls. Process and apparatus for the destruction of night soil and noxious matters. May 11.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

9682. H. Sanguinetti and P. H. Sanguinetti. Improvements in apparatus for straining paper pulp and like materials. April 27.

9704. E. H. Joynson and J. S. Fearnley. An improvement in or applicable to the manufacture of paper. April 27.

11,005. G. Leske. Improvements in machinery for corrugating paper. May 13.

### Complete Specifications Accepted.

1897.

12,208. W. A. Hall. Manufacture or treatment of paper. April 27.

15,797. K. A. Zschorner. Fibrous material suitable for making paper from peat. May 11.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

9165. J. C. W. F. Tiemann. Manufacture of derivatives of ionone and pseudo-ionone and β-ionone therefrom. April 20.

9668. G. Bauwens. New method of preparing the nitro-derivative of a hydro-carbon obtained by dehydration of a suitable mixture of acetones of the fatty class and serviceable in perfumery. April 27.

9690. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Bruning, Germany. A process for producing ethereal salts of indoxylcarboxylic acid. April 27.

9713. G. W. Johnson.—From Kalle and Co., Germany. The manufacture and production of cinnamyl-meta-cresol. April 27.

9966. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new chemical compounds containing phosphorus. April 30.

10,433. G. W. Johnson.—From Kalle and Co., Germany. The manufacture and production of camphoric and citric ethers of the pyrocatechin-monoalkylethers. May 6.

10,434. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the manufacture and production of homologues of the monalkyl ethers of pyro catechol. May 6.

10,740. J. Roberts. Camphor-terebene toilet preparations for the skin and hair. May 11.

11,061. C. D. Abel.—From The Actien-Gesellschaft für Anilin Fabrikation, Germany. New pharmaceutical compound of gelatin and tannin. May 14.

### Complete Specifications Accepted.

1897.

11,314. W. L. Wise.—From Knoll and Co. Manufacture of compounds of ichthyol. May 11.

13,214. J. B. Cohen. Method of preparing aldehydes and ketones from alcohols. May 4.

14,305. M. Otto. Apparatus for extracting the essential matters contained in aromatic plants, and analogous plants. May 11.

1898.

1989. J. U. Lloyd. Method of obtaining the salts of nicotine or kindred alkaloids in a concentrated state from the vegetable substances which contain them. April 27.

## XXI.—PHOTOGRAPHY.

### Application.

10,589. M. Petzold. An improved process for making silver halogen emulsions sensitive to light. May 9.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

9190. U. Alvisi and E. Pulifici. Improvements in the manufacture of explosives. Complete Specification. Filed April 21. Date applied for Sept. 21, 1897, being date of application in Italy.

9546. L. Davies and C. W. Curtis. Improved explosive. April 26.

10,430. C. A. McEvoy. Improvements in percussion fuses for projectiles. May 6.

10,489. J. G. Dixon. Improvements in detonators for railway fog signalling. Complete Specification. May 7.

### Complete Specifications Accepted.

1897.

14,271. F. J. Maubeuge. Combined time and percussion fuses. April 27.

1898.

7100. H. P. Hurst. Improvements in explosive shells, and in fuses or fuse fittings for detonating the explosive. April 27.

7579. W. Weiffenbach. Firework and process of making same. May 4.

8041. L. Muller. Process for increasing the resistance of explosives to atmospheric influences, and for increasing their explosive power. May 11.

## XXIII.—ANALYTICAL CHEMISTRY.

### Applications.

9518. P. Girard. Improvements in or relating to the treatment of cellulose for the purpose of obtaining sheets or blocks for dialysis and other purposes. Filed April 25. Date applied for April 1, 1897, being date of application in France.

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# THE JOURNAL
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| 3 subscriptions received in 1895 | 3 15 0 | |
| 76 subscriptions received in 1896 | 95 0 9 | |
| 2,704 subscriptions received in 1897 | 3,380 3 2 | |
| 35 subscriptions received in 1898 | 43 15 0 | |
| (Sundry balances of subscriptions) | 1 19 0 | 3,524 12 11 |
| 2,818 | | |
| 226 Entrance Fees | 237 12 0 | |
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| Interest on Metropolitan 3 per cent. Consolidated Stock | 129 16 4 | |
| Interest on Gas Light and Coke Company's 6 per cent. Debenture Stock | 17 8 0 | |
| Interest on North British Railway 3 per cent. Consolidated Lien Stock | 31 9 2 | |
| Interest on Midland Railway 4 per cent. Preference Stock | 17 15 8 | |
| Interest on Great Eastern Railway 4 per cent. Guaranteed Stock | 25 5 8 | |
| Interest on New Zealand 3 per cent. 1945 Stock (half-year's interest) | 14 10 0 | |
| Interest on New South Wales 3 per cent. 1935 Stock (half-year's interest) | 7 0 8 | |
| Interest on Deposit Account at Bank | 19 7 1 | 262 12 7 |
| Journal:— | | |
| Advertisements | 576 0 0 | |
| Sales | 368 10 8 | 944 10 8 |
| | | £4,738 16 8 |

### EXPENDITURE.

| | | £ s. d. | £ s. d. |
|---|---|---|---|
| Journal Expenses:— | | | |
| Publishing | | 1,589 2 2 | |
| Editorial:— | | | |
| Editor's Salary | 500 0 0 | | |
| Editor's Expenses | 44 12 0 | | |
| Abstractors' Accounts | 418 1 7 | | |
| Indexing Journal | 66 18 0 | | |
| Patent Lists | 67 11 0 | | |
| Foreign Journals | 8 18 0 | | |
| Sundry Journals | 3 7 3 | | |
| Portion of Secretary's salary | 100 0 0 | 1,209 7 10 | |
| Insurance of Stock | | 3 15 0 | 2,802 5 0 |
| Printing Sundries | | | 68 4 6 |
| Sectional Expenses | | | 306 12 2 |
| Secretary's Salary (Portion not included in Journal Editorial Expenses) | | | 337 10 0 |
| Expenses in connection with "Annual Meeting" | | | 89 6 10 |
| Honorary Treasurer's Assistant | | | 52 10 0 |
| Stationery | | | 46 15 4 |
| Office Expenses | | | 113 5 4 |
| Library (Binding Books) | | | 5 15 10 |
| Auditors' Fee | | | 10 11 6 |
| "Collective Index" (Amount paid on Account) | | | 100 10 0 |
| Bank Charges | | | 4 3 8 |
| Treasurer's Petty Cash | | | 14 3 6 |
| Secretary's Petty Cash | | | 49 10 7 |
| Balance of Revenue over Expenditure | | | 737 12 5 |
| | | | £4,738 16 8 |

## THE TREASURER—IN ACCOUNT WITH THE SOCIETY OF CHEMICAL INDUSTRY FOR THE YEAR 1897.

Dr.

| | £ s. d. | £ s. d. | £ s. d. |
|---|---|---|---|
| To Cash on Deposit (1st January 1897) | | 750 0 0 | |
| Balance at Bank (1st January 1897) | | 162 17 5 | |
| Balance in Secretary's hands (1st January 1897) | | 7 10 10 | 920 8 3 |
| Annual Subscriptions:— | | | |
| 3 subscriptions for the year 1895 | | 3 15 0 | |
| 53 subscriptions for the year 1896 | | 66 5 0 | |
| 2,704 subscriptions for the year 1897 | | 3,380 3 2 | |
| 66 subscriptions for the year 1898 | | 82 9 0 | |
| 2,826 | | | |
| Sundry Balances on account of 1897 and 1898 | | 2 19 0 | 3,535 11 2 |
| Entrance Fees (226 at 1*l*. 1*s*., and 6*s*. balance) | | .. | 237 12 0 |
| Life Composition Fees (2 at 20*l*.) | | .. | 40 0 0 |
| Subscriptions on account of the "Collective Index" | | .. | 7 0 6 |
| Interest on Investments:— | | | |
| Metropolitan 3 per cent. Consolidated Stock | (4,476 9 2) | 129 16 4 | |
| Gas Light and Coke Company's 6 per cent. Debenture Stock | (300 0 0) | 17 8 0 | |
| North British Railway 3 per cent. Consolidated Lien Stock) | (1,084 13 4) | 31 9 2 | |
| Midland Railway 4 per cent. Preference Stock. | (460 0 0) | 17 15 8 | |
| Great Eastern Railway 4 per cent. Guaranteed Stock | (654 0 0) | 25 5 8 | |
| New Zealand 3 per cent. 1945 Stock (half-year) | (1,000 0 0) | 14 10 0 | |
| New South Wales 3 per cent. 1935 Stock (half-year) | (485 4 8) | 7 0 8 | |
| | (8,460 7 2) | | |
| Deposit Account at Bank | | 19 7 1 | 262 12 7 |
| Journal:— | | | |
| Advertisements | | 480 0 0 | |
| Sales | | 360 14 0 | 840 14 0 |
| | | | £5,843 18 6 |

Cr.

| | | £ s. d. | £ s. d. |
|---|---|---|---|
| By Journal Expenses:— | | | |
| Publishing | | 1,374 12 6 | |
| Editorial:— | | | |
| Editor's Salary | 500 0 0 | | |
| Editor's Expenses | 40 0 9 | | |
| Abstractors | 396 8 1 | | |
| Portion of Secretary's Salary | 100 0 0 | | |
| Indexing Journal | 77 16 0 | | |
| Patent Lists | 68 0 8 | | |
| Foreign Journals | 8 18 0 | | |
| Sundry Journals | 3 0 9 | 1,194 4 3 | |
| Insurance of Stock | | 3 15 0 | 2,572 11 9 |
| Printing Sundries | | | 52 10 6 |
| Sectional Expenses: | | | |
| Liverpool Section | | 34 6 2 | |
| London Section | | 102 8 7 | |
| Manchester Section | | 55 5 6 | |
| Newcastle Section | | 8 19 11 | |
| New York (U.S.A.) Section | | 51 2 6 | |
| Nottingham Section | | 12 4 7 | |
| Scottish Section | | 20 11 0 | |
| Yorkshire Section | | 18 13 11 | 306 12 2 |
| Secretary's Salary (Portion not included in Journal Editorial Expenses) | | | 337 10 0 |
| Expenses in connection with "Annual Meeting" | | | 89 6 10 |
| Honorary Treasurer's Assistant (for work in 1896) | | | 52 10 0 |
| Stationery | | | 26 18 9 |
| Office Expenses:— | | | |
| Rent | | 95 0 0 | |
| Cleaning, Light, and Sundries | | 18 5 4 | 113 5 4 |
| Library (Binding Books) | | | 5 15 10 |
| Auditors' Fee | | | 10 11 0 |
| Purchase of 1,000*l*. 3 per cent. New Zealand 1945 Stock | | 1,003 16 0 | |
| Purchase of 485*l*. 4*s*. 8*d*. 3 per cent. New South Wales 1935 Stock | | 496 4 0 | 1,500 0 0 |
| Bank Charges | | | 4 3 8 |
| Treasurer's Petty Cash | | | 14 3 6 |
| Secretary's Petty Cash | | | 49 10 7 |
| Amount paid on account of the "Collective Index" | | | 100 10 |
| Cash on Deposit (31st December 1897) | | 500 0 0 | |
| Balance at Bank (31st December 1897) | | 97 8 3 | |
| Balance in Secretary's hands (31st December 1897) | | 10 10 4 | 607 18 7 |
| | | | £5,843 18 6 |

We have compared the above Statement with the Vouchers, Counterfoils of the Receipts issued, and the Books of the Society, and certify it to be correct. The amount of the Metropolitan 3 per cent. Consolidated, 3 per cent. New Zealand, and 3 per cent. New South Wales Stocks has been confirmed by the Chief Accountant to the Bank of England, Certificates for the remaining investments have been inspected, and the Bankers have verified the Balances upon the Deposit and Current Accounts.

(Signed) MIALL, WILKINS, RANDALL, & Co.,
Chartered Accountants.

23, St. Swithin's Lane, London, E.C.,
22nd March 1898.

# NOTICES.

### ANNUAL GENERAL MEETING.

The Annual General Meeting will be held in Nottingham on the 13th, 14th, and 15th July next. For Programme and Application Forms for Tickets, which should be sent in to the Local Secretary at once, see May Number of the Journal.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those Members whose names are placed in italics in the list of Council will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. George Beilby has been nominated to the office of President; Prof. F. Clowes, D.Sc., has been nominated Vice-President under Rule 11; and Mr. George E. Davis, Mr. John Heron, and Dr. Chas. A. Kohn have been nominated Vice-Presidents under Rule 8.

Sir Robert Pullar has been nominated an Ordinary Member of Council under Rule 24, and the Hon. Treasurer and Hon. Foreign Secretary have been nominated for re-election to their respective offices.

Sir John Evans, K.C.B., F.R.S., Mr. W. Winwood Gossage, Dr. Rudolph Messel, and Mr. E. C. C. Stanford have been nominated under Rule 18 to fill four vacancies among the Ordinary Members of Council.

A Ballot List and Member's Ticket are enclosed herewith or have been already forwarded to members abroad under separate cover.

### ALTERATION OF BYELAWS.

Notice is hereby given, in accordance with Rule 54 of the Bye-laws, that the Council will propose to the forthcoming Annual General Meeting the amendment of the Society's Bye-laws as follows:—

1. Rule 7. To add rule 19 to the list of exceptions.
2. Rule 19. To add "and no ballot shall be taken. If four persons only be nominated, no ballot shall be taken."
3. Rule 20. To add at beginning "If the number of persons nominated, as provided by Bye-law 18, be more than four—"
4. Rule 56. To replace the words "be at the discretion" by the words "subject to the sanction."

### COLLECTIVE INDEX.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows:—

| | |
|---|---|
| To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List | Each copy 10*s.* |
| To Subscribers ........................ | " 12*s.* 6*d.* |
| To others........................ | " 15*s.* |

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. EYRE and SPOTTISWOODE, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

### TELEGRAMS.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

## LIST OF MEMBERS ELECTED 23rd JUNE 1898.

Baldwin, Abram T., Solvay Process Co., Syracuse, N.Y., U.S.A., Alkali Manufacturer.

Davis, Geo. Keville, Sandilands, Knutsford, Cheshire, Chemical Engineer.

Dow, Herbert H., Midland, Mich., U.S.A., Manufacturing Chemist.

Evans, S. Lavington, Sittingbourne, Kent, Tanner.

Eveleigh, Bertie C., 2, Cotham Vale End, Hampton Road, Bristol, Chemical Traveller.

Gabain, Chas. E., Messrs. Gabain Frères, Havre, France, Manufacturer of Edible Fats.

Garnett, Chas. R., 226, Branston Road, Burton-on-Trent, Supervisor Inland Revenue.

Garry, H. Stanley, St. John Street, Mansfield, Notts, General Manager (Chemical Manure Works).

Haigh, Fred., 677, East 24th Street, Paterson, N.J., U.S.A., Chemist (Flax Spinning Co.).

Jackson, Alf. Greville, Adelaide Street, Brisbane, Queensland, Electrical Engineer.

Leman, Wm. T., Manhattan Oil Co., Galatea, Wood Co. Ohio, U.S.A., Oilworks Manager.

Moody, Herbert R., Gilbert School, Winsted, Conn., U.S.A., Science Instructor.

Raschen, Dr. Julius, The Glen, Runcorn, Cheshire, Consulting Chemist (United Alkali Co.).

Royle, Thos. H., c/o Messrs. Carew and Co., Rosa, N.W.P., India, Chemist.

Sanderson, T. Crisp, Brooklyn, Humber Road, Westcombe Park, S.E., Chemical Engineer.

Thurnauer, Gustav, c/o Chicago and Aurora Smelting Co., Aurora, Ill., U.S.A., Chemist.

Tripp, Dr. E. Howard, Granville House, Dalby Square, Margate, Science Master.

Weeple, Lawrence, Pinchin's Wharf, Broad Street, Ratcliff, E., Colour Works Chemist.

Youl, John, Herold's Institute, Drummond Road, Bermondsey, S.E., Leather Trades Chemist.

## CHANGES OF ADDRESS.

Affleck, Dr. J., l/o Garston; Highfield, Woodend Park, Grassendale, near Liverpool.

Benfey, Dr. Hans, l/o 128; 274, Upper Brook Street, Manchester.

Bird, Arthur W., l/o Westbourne Terrace Road; 10, Norfolk Square, London, W.

Bird, Henry, l/o Plymouth; Springfield, Bude, North Cornwall.

Briggs, Jno. F., l/o St. Ann's Hill; Brooklyn, Wimbledon Park Road, Wandsworth, S.W.

Butterfield, J. A., l/o Redhill; Overdale, Bletchingley, Surrey.

Coward, Percy, l/o Manchester; Sewage Disposal Works, Deighton, Huddersfield.

Curphey, W. S., l/o Glasgow; Borva, Lenzie, N.B.

Duguid, Jas., l/o 2; 46, Sotheby Road, Highbury, N

Dunn, Fred., l/o Flinders Lane; 193, Collins Street, Melbourne, Victoria, Australia.

Elworthy, H. S., l/o London; 2, Peel Terrace, Havre-des-Pas, Jersey.

Fawcett, Jas. H., l/o Melbourne; St. George's Club, Hanover Square, London, W.

Foden, Alf., l/o Everton Valley; 4, Goodison Road, Walton, Liverpool.
Garner, D. C., l/o Syston; 40, Nichols Street, Leicester.
Glendinning, T. A., l/o Leeds; 136, Montague Street, Blackburn.
Glover, Wm., l/o Newcastle; Rio Tinto Mines, Huelva, Spain.
Gow, R. J., l/o The Cedars; Ivy Lea, Hough Green, near Widnes.
Gray, Jno., l/o Loanhead; 13, Queen's Road, Rock Ferry, near Birkenhead.
Hambly, F. J., l/o Aberdeen; c/o The Electric Reduction Co., Buckingham, Quebec, Canada, Chemist.
Hamilton, D. R., l/o Graham Street; 25, Robertson Street, Glasgow.
Harris, W. T. A., l/o London; Ideal Soapery, East Street, Brompton, Adelaide, South Australia.
Hartley, Arthur, l/o Gosport; The Brewery, Emsworth, Hants.
Hersey, Milton L.; Journals to 16, St. Sacrament Street, Montreal, Canada.
Johnstoun-Coombes, Dr. W., l/o Cambridge Place; Pilmuir, Falmouth, Cornwall.
Kebler, Lyman F., l/o Cherry Street; 35, Poplar Street, Philadelphia, Pa., U.S.A.
Lange, Dr. M., l/o Van Baerlestraat; Nieuwe Witsenkade 35, Amsterdam, Manufacturing Chemist.
Lynn, A. H., l/o Zürich; c/o Mrs. Holt, Beeches Road, West Bromwich.
Macdonald, G. W., l/o Dartford; c/o W. Macnab, 14, Great Smith Street, Westminster, S.W.
Mansbridge, Wm., l/o Rotterdam; 22, St. Stephen's Road, Lee, S.E.
Moale, Dr. Philip R., l/o Chestnut Street; 100, Merrimon Avenue, Asheville, N.C., U.S.A.
Morris, Herbert N., l/o Manley Road; 48, Lansdowne Road, Crumpsall, Manchester.
Muir-Smith, W., l/o Dundee; c/o A. B. Fleming and Co., Ltd., Caroline Park, Edinburgh.
Owen, Thos., l/o Bristol; 5, Whitehall Gardens, London, S.W.
Oxland, Robt., l/o Plymouth; 32, Ravenswood Road, Redland, Bristol.
Picard, Hugh K.; Journals to c/o C. Vantin, 66, Old Broad Street, E.C.
Playfair, D. J., l/o Kelvinside; 7, Victoria Crescent, Dowanhill, Glasgow.
Scrutton, W. J., l/o Australia; 18, Billiter Street, London, E.C.
Shepard, Dr. Chas. U., l/o Meeting Street; 56–58, Broad Street, Charleston, S.C., U.S.A.
Smith, R. Greig, l/o Newcastle; Linnean Society's House, Elizabeth Bay, Sydney, N.S.W.
Smith, H. Wilson, l/o Bow Common; c/o Wm. Docker, Varnish Works, Lower Edmonton, N., Varnish Works Manager.
Spoor, J. L.; Journals (temp.) to Culver House, Gravesend, Kent.
Townsend, C. F., l/o Wrotham; Basted Cottage, Ightham, near Sevenoaks.
Ullman, Jas. A., l/o Pearl Street; c/o Sigmund Ullman Co., 146th Street and Park Avenue, New York, U.S.A.
Weigall, A. R., l/o Queensland; Totok, *viâ* Menado, Celebes, Netherlands India.
Welch, J. Cuthbert; Journals to P.O. Box 196, Trail, British Columbia.

## CHANGES OF ADDRESS REQUIRED.

Claus, Carl F., jun., l/o 94, Grove Park Terrace, Chiswick, W.
Gendall, W. H. S., l/o Gas Works, Elton, Bury.
Passmore, Dr. F. W., l/o 29, Wolfington Road, West Norwood.

## Deaths.

Stanley, Charles L., Messrs. C. Stanley and Son, Ltd., Wath-on-Dearne. June 18.
Vaughan, J. Ivens, Harold Road, Leytonstone, Essex. April 18.

## Liverpool Section.

*Meeting held on Wednesday, May 25th, 1898.*

DR. C. A. KOHN IN THE CHAIR.

### SOME NOTES ON THE DISINFECTION AND DEODORIZATION OF SEWAGE WITH CHLORINE.

BY MAX MUSPRATT AND E. SHRAPNELL SMITH.

The subject of Chlorine Disinfection has during the last year attracted afresh considerable attention, notably at the Sanitary Congress of 1897, when Prof. Delépine gave convincing testimony of its enormous germicidal value, based upon both laboratory and practical experience, and was supported in his views by Dr. Sims Woodhead, Dr. Porter, and others.

It will also be remembered that Dr. Clayton read a paper upon the same subject before this section, in which he summarised the work done upon chlorine disinfection, and contributed some important researches of his own in conjunction with Prof. Boyce upon the action of sodium hypochlorite on pathogenic germs.

His work was later confirmed by further researches made by Dr. Klein and published in the *Lancet*.

Though the results obtained by various workers differ somewhat owing to variations in method, the mass of evidence points to the following conclusions:—

1. Chlorine, whether as hypochlorite of soda, hypochlorite of lime, or as chlorine gas, has, the same germicidal and deodorant value, though this in practice is modified by the question of contact.

2. Chlorine in the proportion of 1 to 2,000 is a safe germicide for all ordinary pathogenic germs within 15 minutes.

3. Chlorine is a *deodorant* at a dilution of 1 in 150,000. These figures are not approached by any known disinfecting bodies except sublimate and perhaps formaldehyde, both expensive articles; and, when germicidal value and price are jointly considered, chlorine stands as undoubtedly the best disinfectant for practical use.

At the same time it must be remembered that in the case of chlorine we have a body which attacks organic matter, and hence, though bacteriological investigation has given such excellent results, it may be contended that in practice the chlorine exhausts itself upon the organic matter present, in the sewage to be disinfected, and ceases to have the desired disinfecting value.

The present communication aims at fixing the limits of the exhaustion of chlorine by organic matter in sewage, and also by ammonia, the most sensitive of the bodies found in excreta:

In these experiments the chlorine was employed in the form of. hypochlorite of soda ("Chloros") containing 10 per cent. av. Cl.

To classify the sewages, the oxygen absorption test was employed, and carried out upon the lines laid down by Mr. J. Carter Bell in a paper read recently before the Manchester Section of this Society. Briefly, the method is as follows:—

Solutions used—Permanganate = 0·001 gr. av. O per c.c., sulphuric acid 1·4; thiosulphate of soda to correspond with permanganate solution; strong potassium iodide solution and starch solution.

To 25 c.c. sewage were added 50 c.c. water, 10 c.c. sulphuric acid, and 50 c.c. permanganate.

After an exposure of four hours, potassium iodide was added and the liberated iodine titrated back with thiosulphate.

The difference between this result and that obtained in the absence of sewage gave the oxygen absorption. For the purposes of this paper the absolute correctness of the test is unimportant, but the results are comparative, and are sufficiently accurate for the classification of the sewages dealt with.

The sewages were, at the same time, treated with chlorine in the form of hypochlorite of soda, and, after varying exposure, potassium iodide was added and then acid; all available chlorine thus liberating free iodine, which was titrated with the same thiosulphate solution as in the oxygen absorption test. The oxygen absorption and chlorine exhaustion are thus obtained in an easily comparable form, and the results in the following experiments are expressed as oxygen.

*Experiment I.*—Absorption test, four hours' contact at 50° F. gave 7·9 grs. absorbed 0 per 100,000 of sewage.

Hypochlorite of soda containing 10 per cent. av. Cl. was added in the proportion of 1 to 100, that is 1 in 1,000 chlorine.

| | | | | | |
|---|---|---|---|---|---|
| After 30 mins. the loss of chlorine equalled | | | | 4·2 | grs. 0 per 100,000 |
| „ | 4 hrs. | „ | „ „ | 7·1 | „ |
| „ | 24 „ | „ | „ „ | 9·2 | „ |
| „ | 7 days | „ | „ „ | 11·8 | „ |

A solution of 1 in 100 hypochlorite in pure water corresponds to 26 grs. 0 per 100,000. It will thus be seen that, in this case, the loss of chlorine is only 16 per cent. in 30 minutes, and in four hours it is 27 per cent; and approximately the same as the permanganate absorption.

The rapidity with which the chlorine was lost at the commencement, however, led us to think that it was perhaps due to the strong hypochlorite acting rapidly before it had become mixed with all the sewage, and accordingly the next experiment was modified so that strong hypochlorite and sewage did not come immediately in contact with one another, as follows:—

*Experiment II.*—Equal volumes of sewage and dilute hypochlorite (2 in 100) were mixed, the results being:—

Oxygen absorption 8·2 grains per 100,000. Sewage and hypochlorite treated as described above gave—

| | | | |
|---|---|---|---|
| An immediate loss of Cl..... | = 2·5 | grs. 0 | per 100,000 |
| After 30 mins................ | = 4·2 | „ | „ |
| „ 1 hour.................... | = 7·5 | „ | „ |
| „ 4 hours.................. | = 9·5 | „ | „ |

Seeing that these results are on only half the quantity of sewage, they are distinctly worse, but they dispose of the question of the rapid action at the beginning being due to the concentrated liquor.

After settling this point, a number of experiments were made upon the lines indicated, the results of which vary considerably with the varying nature of the sewages; the following is a table of them:—

| — | 1. | 2. | 3. | 4. | 5. | — |
|---|---|---|---|---|---|---|
| Oxygen absorption, 4 hrs. | 7·9 | 8·9 | 14·0 | 5·2 | 6·6 | grs. 0 per 100,000 |
| Loss of Cl after 30 mins | 4·2 | 4·8 | (7·1 | 2·4) | 6·0 | „ |
| Loss after 4 hrs | 7·4 | .. | {8·7 | 3·0} | 10·0 | „ |
| 1 in 100 hypochlorite. | 25·0 | 26·0 | 26·0 | 26·0 | 26·0 | „ |

(Figures in brackets are on equal volumes of sewage and dilute hypochlorite.)

It will be noticed that, with the possible exception of No. 3, which is an abnormally bad sewage, the loss of chlorine, though quite appreciable, in no case exceeds 50 per cent. of the quantity originally present, or, in other words, the sewage remains for fully four hours at a strength of over 1 in 2,000 of chlorine, which is the figure at which, according to the various authorities cited, chlorine is a certain disinfectant in 15 minutes at the most.

It may be of interest to point out the quantity of organic matter present which is not attacked by the chlorine within the time limit and which, therefore, does not stultify the chlorine as a disinfectant. The following instances will suffice. Sewages:—

| | | | |
|---|---|---|---|
| Oxygen test ............ | 6·6 | 14·0 | grs. per 100,000 |
| Total organic matter.... | 48·8 | 131·6 | „ „ |
| Loss of chlorine......... | 6·0 | 7·1 | (On half the sewage.) |

It is thus seen that in sewage the quantity of matter which is attacked by hypochlorite in weak solution is exceedingly small, and, if a strength of 1 in 100 is used of hypochlorite of soda containing 10 per cent. av. Cl. an exposure of 15 minutes will kill all pathogenic germs present.

Or, if other forms of chlorine are applied, if the chlorine added is 1 in 1,000 of total sewage, the same result will be obtained.

Our next experiment was directed to the question of deodorization of sewage and whether the manurial value is destroyed by the action of chlorine.

It has been proved both on the large scale and in the laboratory that ordinary sewage is deodorized by 1 part of chlorine to 150,000 of sewage.

Ordinary sewage contains about 5 parts of ammonia per 100,000, and in order to have the most adverse conditions, we treated a solution containing 10 parts of ammonia per 100,000 with hypochlorite of soda in the proportion of 1 to 15,000.

There was no evolution of nitrogen, and after 24 hours' standing the following was the condition of the solution: the chlorine though reduced in quantity was still present in spite of the excess of ammonia; ammonia was still present; and, most important of all, after destroying the chlorine by boiling, the presence of nitrous acid in appreciable quantities was proved with potassium iodide and starch in acid solution, leaving little doubt that in such low dilution chlorine does not destroy but merely nitrifies ammonia, and this does not affect its manurial value.

Reverting once more to the question of disinfection, we made the following experiments:—

Equal volumes of dilute ammonium hydrate (2·00 per cent. $NH_3$) and hypochlorite (2 in 100) were mixed and kept at various temperatures by immersion. The losses of available chlorine during different periods, expressed as grains 0 per 100,000, were as follows:—

| Temperature. (F.) | 57° | 60-65° | 70-75 | 80-85 | 90 95 | 100-105 |
|---|---|---|---|---|---|---|
| After 1 hour | 2·8 | 3·4 | 6·4 | 11·0 | 15·9 | 21·0 |
| „ 2 hours | 6·0 | 7·2 | 13·0 | 17·4 | 22·1 | 25·8 |
| „ 4 „ | 11·9 | 13·4 | 19·4 | 23·6 | 25·9 | 27·4 |
| 1 in 100 hypochlorite | 27·4 | 27·4 | 27·4 | 27·4 | 27·4 | 27·4 |

This series was undertaken to approximate to the disinfection of mixed excreta and urine—as met with in the conservancy or pail system used in a great many towns—where the total ammonia (free and albuminoid) is about 1 per cent. The use of 1 per cent. free ammonia in the experiment is admittedly too severe, but it only emphasises the certainty of chlorine in its action as a germicide. The results go to prove that hypochlorite may be used 1 in 100 with a margin of safety, for the chlorine left exceeds 1 in 2,000 at the end of one hour in the case of temperatures up to 80° to 85° F., and at the end of four hours in the case of temperatures of 60° to 65° F. It will be noticed that there is a retardation of the reaction with ammonia, as the chlorine becomes exhausted.

An interesting point bearing upon the facts mentioned in connection with deodorization is that here too, part of the ammonia is oxidised to nitrous acid. At ordinary temperatures the percentage is considerable, and the decomposition with evolution of nitrogen is not of moment with the dilutions named at temperatures below 90° F.

We regret that we have not had time to go into this question of nitrification in very great detail, but trust, however, that by next session we shall have a further communication to make, in which we can give fuller details on this part of the subject.

### Discussion.

Mr. Alfred Smetham remarked that the investigation of the authors did not seem to quite meet the requirements of the case, inasmuch as the manurial value of sewage was due, not merely to the actual ammonia which it contained, but also to the considerable proportion of nitrogenous matters which had not been converted into the condition of ammonia. Before sewage could be of any value in the field the nitrogenous compounds should be converted by the organisms in the soil into nitric acid, and from experiments which he had himself carried out, he was afraid that the treatment with chlorine compounds would prevent the nitrification from taking place. There would thus be no benefit from the nitrogen other than ammonia contained in the sewage, and in addition there would be the risk of injury to the crops grown upon the land to which sewage so treated was applied.

Mr. **Herbert E. Davies** held that to disinfect sewage was a mistake both economically and biologically, because not only pathogenic organisms were destroyed, but also useful ones. The only satisfactory system of sewage treatment was the bacteriological system which had been adopted at Sutton in Surrey, Exeter, &c. With regard to the proposals of the authors, he did not see how they could be adopted on a large scale, as in a city like Liverpool, over 480 tons of chlorine per day would be required, according to Koch's researches, and this meant an expenditure of at least 2,000*l.* a day.

Dr. **C. A. Hill** said that during the last few months he had been making experiments with various disinfectants in connection with the disinfection of typhoid excreta. The plan pursued was to mix the excreta with a definite quantity of the disinfectant to be tested, leaving it for half an hour, a reasonable working time for use in a sick room, and then making plate cultivations from the resulting mixture, incubating them and seeing whether any organisms grew. Amongst the disinfectants tested were solutions of chloride of lime containing 15 and 35 per cent. of available chlorine, and the solution of hypochlorite of sodium known as "chloros." In no case did disinfection take place under the conditions employed. An important point determined by this investigation was that unless the excreta were thoroughly broken up and well mixed with the disinfectant, the interior of the fœcal mass was never sterilised in the slightest degree. Any pathogenic organisms contained in the interior of these lumps grew luxuriantly afterwards. He thought that the disinfection of sewage generally with chemicals was a mistaken idea—the rational method being to let it treat itself, and this view was daily growing in favour amongst sanitarians, owing to the marked success of the "septic tank" system. As regards pathogenic organisms in sewage there were very few which would live any length of time in that medium. He had made many observations on the bacteria of sewage, and in no single instance were any pathogenic organisms detected; typhoid bacilli very rapidly died out in sewage, they lasted practically only two or three days, or perhaps a little longer, and during that time became greatly attenuated. The deodorizing effect of chlorine solutions was well marked, but at the same time the smell of chlorine was objectionable ot many people—a drawback to its use in private houses. Other substances were found to deodorize just as well without having any objectionable odour of their own.

Mr. **Arthur Carey**, replying for the authors, said it seemed to him the discussion had become one rather on the general question of the treatment of sewage than on the points raised by the paper. To attempt to disinfect the whole sewage of a town by chemical means was of course entirely out of the question. The chemicals were valuable to sanitary authorities, and were likely to be so for many years to come, but only for restricted application, such, for instance, as deodorization, which on a small scale was frequently required, especially during warm weather. Chemicals were also required for the disinfection of infected sewage, such as that from infectious diseases hospitals or from private houses in which there was infectious disease, before it enters the main sewers. The figures quoted by Mr. Davies were Koch's figures for the sterilisation of liquid sewage, and the figures in the paper with which he compared them were for the deodorization of sewage, two very different things. Under no conceivable circumstances would it be necessary to sterilise the sewage of a whole town as considered in Mr. Davies' calculation. Disinfection was required only when there was infection to kill, and unless the whole population of a town were down at once with some infectious disease there would be no advantage in treating all the sewage. Mr. Davies had observed that the only satisfactory system of sewage treatment on a large scale was the bacteriological treatment advocated by Mr. Dibdin, and now working successfully. This was no doubt the direction in which success would ultimately be achieved, but was it not rather a question whether Mr. Dibdin had yet succeeded? Dr. Hill's remarks on the action of disinfectants, and especially hypochlorite of soda, on germs embedded in solid excreta were open to this criticism—that no disinfectant could be expected to act unless brought into contact with the germs to be killed. In all cases mechanical disintegration was required to destroy germs inside the solid mass of excreta. Dr. Hill's remarks were directed chiefly against hypochlorite of soda, but the same arguments and objections applied to all other disinfectants. With regard to Mr. Smetham's remarks, undoubtedly the manurial value of the sewage depended on the other nitrogenous matter as well as on the ammonia contained in it. Mr. Smetham appeared to have an exaggerated idea of the effect, the possible effect, of the quantities of chlorine it was ever suggested to mix with the sewage. The chlorine in this case could only have been used as a deodorant, and never would be at a dilution of about 1 in 150,000 of sewage; not a quantity the effect of which could ever be noticed.

## MINERALS OF LIVERPOOL AND DISTRICT.

BY CHARLES C. MOORE, F.I.C.

The local rocks consist almost entirely of sandstones, the composition of which varies within very narrow limits. Recently, however, I examined some small black deposits occurring in the sandstone in the form of fine threads, occasionally enlarging into tiny pockets $\frac{1}{4}$ inch in diameter; the black grains analysed consisted of silica with a coating of metallic oxides, and possessed the following composition:—

| | | | |
|---|---|---|---|
| $SiO_2$ | 81·40 | $MgO$ | 1·00 |
| $Fe_2O_3$ | 3·25 | $TiO_2$ | 0·12 |
| $Al_2O_3$ | 2·32 | $NiO$ | 1·72 |
| $Mn_2O_3$ | 2·78 | $CoO$ | 2·18 |
| $CaO$ | Trace | Combined Water | 4·56 |

More extensive examination of the rocks in various parts of the district shows that this occurrence of nickel and cobalt is pretty general. At Moel Hiraddug near Prestatyn in North Wales a considerable nickel and cobalt deposit is worked commercially. It would seem that such deposits are not part of the original rock but are due to subsequent infiltration, and this is supported by a specimen of sandstone from Bidston Hill near Birkenhead; this specimen contains 15 per cent. of barium sulphate. Its porosity is 17·34 while the volume per cent. of barytes is 7·88, so that in the absence of barytes the porosity would be 25·22; a specimen of a sandstone, similar in every way except that it contained no barytes, gave a porosity of 24·8, a figure very close to what might be termed the original porosity of the other specimen. This seems to show very clearly that the barytes has simply filled up the spaces already existing in the rock, and it is obvious that such would be the effect of a genuine infiltration.

It may be interesting to mention that small quantities of zirconia have been found in the sand of the Mersey Bar, the zirconia being present in the form of the mineral zircon. Its source is most probably the Millstone Grit, which abounds in the district where the Mersey rises.

# London Section.

*Meeting held on Monday, April 4th, 1898.*

DR. R. MESSEL IN THE CHAIR.

## THE BACTERIAL TREATMENT OF SEWAGE CONTAINING MANUFACTURING REFUSE.

BY W. J. DIBDIN, F.I.C., F.C.S., AND G. THUDICHUM, F.C.S.

(*This Journal*, 1898, 315.)

### Discussion.

The following remarks have been accidentally delayed:—

Dr. A. Dupré felt that the meeting would join with him in regretting that Mr. Dibdin could not be with them, and especially at the cause of his absence. At the same time they must acknowledge that the cause had been left in good hands and therefore had not suffered. A few months ago he would have claimed the idea as one of his own, but Mr. Koechlin had recently shown that the discovery, like many other good and bad things, had been made in Germany, and the only credit they could now take was that of having first brought it to a practical conclusion, owing chiefly to the labours of Messrs. Dibdin and Thudichum. This was a matter which interested a very large number of manufacturers; and in reply to their frequent inquiries as to whether the process was applicable or not to their residues, he had always answered that it would deal with any manufacturing refuse that could be treated on a sewage farm, and as was now shown in the present paper, he might have gone further and said with many others that could not be so treated. Some years ago he had inspected the Wolverhampton Sewage Farm, where the whole of the land was sterilised and the sewage went through it without purification. The present process would deal with these pickling residues successfully, and he hoped that the Royal Commission which was about to sit would recognise this fact and take into account that it was no longer necessary to acquire land for the treatment of industrial residues. He was sure that every chemist in England would second him in expressing the hope that Sir Edward Frankland might be the chemist on that Commission, for he was a fair and large-minded man who would not be hampered by any considerations for older processes and methods simply because they were older. He did not quite agree with Dr. Rideal that the bacteriological argument might be disregarded. It had been proved by Parry Laws and Andrews that typhoid bacilli die in sewage in a very short time; and Dr. Picquart, working with a very small experimental tank and a relatively large number of typhoid bacilli, had shown that, even under these favourable conditions to their survival, very few escaped destruction. They might therefore safely conclude that if the process were worked on a large scale with the mixed sewage of a town, which at no time contained any appreciable number of typhoid bacilli, none at all would escape destruction; they would be like foxes trying to get through a pack of fox-hounds.

*Meeting held on Monday, June 6th, 1898.*

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DR. R. MESSEL IN THE CHAIR.

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Before proceeding to the formal business of the evening, the Chairman observed that Prof. Dewar's remarks in the discussion on Dr. Hampson's paper read at the last meeting seemed to have come at a psychological moment, for they immediately preceded his communication to the Royal Society respecting his brilliant achievement in producing the liquefaction of hydrogen in a stable condition, with which doubtless all the members were now familiar.

The Chairman also referred in feeling terms to the great loss which science had suffered by the death of Lord Playfair.

## ON THE CONDITIONS EXISTING IN ACETYLENE GENERATORS.

BY VIVIAN B. LEWES.

With the introduction of calcic carbide on a commercial scale and at a price which enables it to be used for the generation of acetylene for lighting purposes, there has arisen a prolific crop of generators in which the operation of decomposing the carbide by contact with water is carried on.

When in January 1895 I first introduced the subject of commercial carbide and acetylene illumination to an English audience, I experimentally showed that there were two ways in which contact between the interacting compounds could be conveniently brought about.

1. By allowing water to drip on to carbide,
2. By plunging carbide into water,

and that these two main operations could be so modified as to give forms of generators which should be more or less automatic, *i.e.*, should hasten or decrease the rate of interaction and generation of the gas as might be required.

In the early generators produced by the inventive genius of those anxious to get a good start in this novel field of industry, it was to be expected that but little attention would be paid to scientific principles, or to the details of the actions going on between the water and the carbide, but although three years have now passed and a considerable amount of desultory work has been done on the subject, many of the generators on the market still show so amazing a disregard to the principles which should be embodied, and, in some cases, to the safety of the user, that I thought it would be of use to the constructors of such forms of apparatus to have the conditions existing during the decomposition of calcic carbide clearly defined by experiment, so that they may have a basis to go upon in knowing what to aim at and what to avoid, and it is this task that I have attempted in the work described in the following paper.

When water and calcic carbide are brought in contact, the completed re-action is—

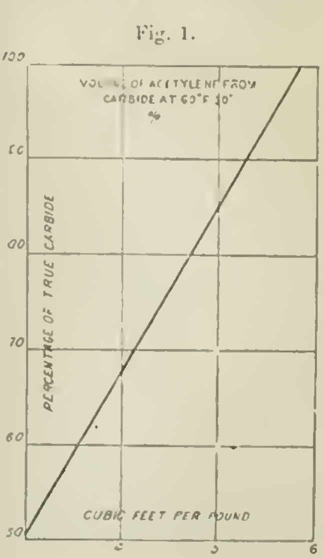
$$CaC_2 + 2\,H_2O = CaH_2O_2 + C_2H_2,$$

and a kilo. of pure carbide would yield 348·9 litres of acetylene at 0 C. and 760 mm., or, in English measures, 1 lb. of carbide would yield 5·58 cb. ft., a volume increased to 5·89 cb. ft. under ordinary atmospheric conditions.

In practice, however, the average yield rarely, if ever, exceeds 5 cb. ft. per lb., this being partly due to commercial carbide never being quite pure, and partly to the excessive heating of the gas during generation bringing about polymerisation of some of the acetylene with consequent loss in volume.

In England, the calcic carbide made by the Acetylene Illuminating Company at their works at Foyers is of a considerably higher degree of purity than the ordinary run of Continental carbide, this being due to the care exercised in the selection of the raw material from which the carbide is made, as with a good lime practically free from magnesia, phosphates, and sulphates, and a dense coke of low ash, a very pure carbide is obtained. The reason of the Foyers carbide containing as a rule only 85 to 90 per cent. of true carbide is due to the presence of a certain proportion of uncombined original material present with it and not to any undue proportion of deleterious impurity.

Fig. 1.



In making carbide in the form of electric furnace usually employed, only a part of the original charge of raw material is used up, and at the end of the "make" there is left the ingot of practically pure carbide surrounded by a crust containing from 50 to 70 per cent. of carbide, whilst outside this again is the unacted-upon mixture of lime and powdered coke.

It is manifest that unless a demand for this crust on a large scale sprang up, it would be impossible for the manufacturers to supply pure ingot alone, and ingot and crust being broken to standard size are in practice blended to give carbide yielding 5 cb. ft. per lb.

The yield of acetylene from carbide of varying purity is a matter of considerable interest to the generator maker, and the diagram (Fig. 1), which illustrates the yield of acetylene per lb. of carbide of varying purity given off under ordinary atmospheric conditions, will be found of use.

Great stress has been laid upon the dangers of acetylene being largely dependent upon impurities in the gas derived from foreign matters in the carbide, and in conjunction with Mr. Boverton Redwood I have devoted considerable attention to trying to elucidate how far this is so.

The danger dependent on the purity of the carbide arises from the presence in some samples of compounds liable to yield spontaneously inflammable gases. These gases are two in number; (*a*) the compounds known as phosphuretted hydrogen or phosphene, which will ignite on contact with air, and (*b*) siliciuretted hydrogen, which has the same property.

These experiments were specially directed to ascertaining whether these gases do exist in acetylene made from ordinary commercial carbide, and to what extent, and secondly, as to whether the quantity so present is a cause of danger.

Samples of ordinary commercial carbide were purchased from various dealers, and care was taken that the English, American, and Continental carbide should be all represented.

The samples were numbered I. to XII., and an equal weight of each being employed, acetylene was generated from them by the action of water, and the phosphuretted hydrogen in the gas was then determined with the following results:—

| Number of Sample. | Percentage of Phosphuretted Hydrogen. | Number of Sample. | Percentage of Phosphuretted Hydrogen. |
|---|---|---|---|
| I. | 0·43 | VII. | 0·72 |
| II. | 0·91 | VIII. | Trace |
| III. | 2·30 | IX. | 0·51 |
| IV. | 0·58 | X. | 0·02 |
| V. | 0·62 | XI. | 0·77 |
| VI. | 0·32 | XII. | 0·80 |

Average from all samples, 0·65 per cent.

It is seen from this that the phosphuretted hydrogen varied from a trace, too small to estimate, up to 2·30 per cent., the sample yielding this result being of Continental manufacture.

The acetylene was generated from these samples of carbide by allowing water to drip upon pieces the size of hazel nuts, the method of production most likely to give rise to spontaneous ignition had there been any tendency to it, but in no case did it occur.

The samples of carbide varied very much in purity, the lowest yield of acetylene being $2\frac{1}{2}$ c. ft. per lb., whilst the highest closely approached the theoretical quantity, but no relation was found between the quality of the carbide and the percentage of phosphuretted hydrogen in the acetylene, some of the poorest samples of carbide yielding a gas very free from this impurity.

It seemed possible that a concentration of the phosphorus compounds might take place in the outer crust formed around the ingot during manufacture, and experiments were made to see if this were so.

By the courtesy of the Acetylene Illuminating Company a portion of an ingot with its crust intact was obtained, the crystalline material of the ingot containing 96·7 per cent. of true carbide, whilst the crust, although varying very much in composition according to its distance from the ingot, contained an average of 51·9 per cent. of carbide. A second sample of ingot and crust gave even better results, the ingot containing 99 per cent. of carbide and the crust 77 per cent.

Analyses were made of the acetylene from these samples, but the results showed no indication of the poorer material being richer in phosphorus compounds than the ingot itself.

On breaking up some ingots of calcic carbide, metallic nodules manifestly differing from the pure carbide are observed, and are far more abundant in specimens of foreign manufacture than in English-made carbide.

These nodules are generally found in a spherical or oval shape, and have evidently separated from the molten mass on the crystallising of the true carbide.

From this it seemed highly probable that a concentration of the impurities might take place, and that the presence of these nodules might be an active source of danger.

In order to verify this point calcic carbide was obtained in bulk, and as before both English and foreign makes were represented. This carbide was then carefully broken up, and the nodules and anything which appeared to differ from true carbide were collected.

The nodules differed somewhat in appearance, two distinct varieties being clearly noticeable.

A. Grey nodules, not attracted by the magnet, not oxidised when exposed to air or heated as a solid lump in blowpipe flame. Specific gravity 3·5 to 5·8. In a fine powder some had a very faint garlic-like odour, gained 5 per cent. when heated for half an hour, and attacked platinum when heated on it for some time. These nodules gave no gas when acted on by water.

B. Steel-like nodules which were strongly magnetic, and became coated with iron rust when exposed to moisture and air for some time, and which gave no gas when acted on by water. Specific gravity 6·3 to 6·8.

Analysis showed that the foreign matter present in the carbide consisted of:—

Carborundum or carbide of silicon,
Metallic silicides,
Iron, occasionally calcium, and traces of
Phosphorus and sulphur compounds.

One of the nodules on analysis yielded:—

| | Per Cent. |
|---|---|
| Silicon | 30·76 |
| Iron | 58·07 |
| Calcium | 2·65 |
| Aluminium | 3·01 |
| Magnesium | 0·64 |
| Carbon, &c. | 4·89 |
| | 100·00 |

None of these substances gave spontaneously inflammable gases when acted upon by water, but in a few instances a nodule was found which evidently contained magnesium silicide, and which when powdered and acted upon by hydrochloric acid gave a few bubbles of spontaneously inflammable gas.

In some cases we found that the carbon deposited by holding a cold surface in an acetylene flame contained traces of silica, and this seems to support the idea that siliciuretted hydrogen may occur in the acetylene, but if it does, the amount is so small as certainly to give no danger of spontaneous ignition.

A sample of gas which appeared to give a considerable quantity of silica was burnt under such conditions as to allow of the silica being collected, and on estimating the quantity formed, it was found to correspond to 0·01 per cent. of siliciuretted hydrogen in the original gas.

Having satisfied ourselves that the average percentage of phosphuretted hydrogen present in acetylene was below 1 per cent. we next proceed to ascertain what percentage of spontaneously inflammable phosphuretted hydrogen must be present in order to make a gaseous mixture which would ignite on coming in contact with air. It is generally supposed that the phosphuretted hydrogen present in acetylene is derived from phosphides of calcium present in the carbide, and which has been produced by the use of phosphatic lime or from phosphates in the mineral ash of the coke employed, such phosphides being decomposed by water with liberation of phosphuretted hydrogen.

In view of the generally accepted theory of its production, we made phosphuretted hydrogen by acting on calcic phosphide with water and tried to determine what proportion of this gas had to be mixed with acetylene in order to give spontaneous ignition. We found, however, that the mere contact with water necessitated in mixing the gases rendered the resulting mixtures non-spontaneously inflammable even when hot water was used, and that the phosphuretted hydrogen alone failed to ignite after being in contact with water for some time. Phosphuretted hydrogen was then made by acting on phosphorus with boiling sodic hydrate, and it was found that using hot water it required over *80 per cent.* to render the acetylene and phosphuretted hydrogen mixture spontaneously inflammable.

From this series of experiments it was perfectly clear that when once the mixture of gas is made even a high percentage of phosphuretted hydrogen would be perfectly harmless except in so far as it would give rise to deleterious products of combustion and might lower the igniting point of the acetylene, but that no risk of spontaneous ignition exists.

If, however, one considers the methods by which the carbide is brought in contact with water in the various forms of acetylene generators and the very high temperature which may be attained by the action of water upon the carbide, it is evident that there are cases in which water being allowed to drip on the carbide or even to rise slowly around the carbide might give spontaneous inflammability to a mixture containing air, which, when only moderately heated, would be absolutely free from this danger.

A series of experiments was now instituted to see what proportion of calcic phosphide mixed with calcic carbide would cause spontaneous ignition on contact with air. In order to get these experiments strictly comparable it was necessary that the ingredients used in each experiment should be in the same state of division and that a similar amount of water should be in each case added to the mixture.

Pure crystalline carbide and fresh calcic phosphide were reduced to powder and sieved down to uniform size. Mixtures of these two ingredients were then made and put in charges of 5 grms. into small porcelain vessels into each of which 5 c.c. of water was added, and it was found that on testing from 50 per cent. downwards, even when only 1 per cent. of calcic phosphide was present, spontaneous ignition of the evolved gases would in many cases take place, and it was also noted during these experiments that the acetylene came off more readily than the phosphuretted hydrogen, and that the ignition was generally brought about by a flash from a bubble of phosphuretted hydrogen after the first rush of acetylene had passed off. In other words the calcic phosphide was less readily decomposed by water than the calcic carbide, so that would be 1 per cent. of calcic phosphide in the original mixture would mean a far higher percentage by the time the calcic phosphide came to be decomposed, and it was manifest also that the temperature created by the action of a limited quantity of water upon the carbide had a great deal to do with the result.

Other experiments were then made with mixtures of carbide and phosphide by plunging the mixture under the surface of several times its own volume of water, and it was then found that 25 per cent. of phosphide might in some cases be present without leading to spontaneous ignition, this of course being due to the cooling effect of the mass of water present.

These experiments seemed to show that English calcic carbide of ordinary commercial manufacture may be used without any danger for generating acetylene gas, provided always that the generator is of such construction as to prevent overheating.

The next point to be determined was the amount of heat developed during the decomposition of the calcic carbide by water in the generator, and the conditions which tend to increase or lessen the intensity of the action. In these experiments, in order to secure uniformity and to make the results strictly comparable, a considerable quantity of calcic carbide of uniform quality was, by the kindness of the Acetylene Illuminating Co., supplied broken to uniform size.

Samples from various parts of the bulk were taken and analysed, the results obtained varying from a minimum of 89·3 to 92·9 per cent., the mean of a considerable number of analyses being 91·3 per cent. Greater uniformity than this could not be expected in the bulk, as it was found that if a well crystallised piece of carbide were broken up into three pieces, each piece would vary slightly from the others, as shown in the following analyses:—

(*a.*) 92·3 per cent. of pure carbide,
(*b.*) 90·7 " "
(*c.*) 91·9 " "

and with commercial carbide this must be so, as in the electric furnace the mass is never sufficiently liquid to ensure complete mixing.

The first determination attempted was the number of calories liberated per grm. of carbide decomposed.

A rough calorimeter was made by jacketing a beaker 12·5 c.m. in diameter with cotton wool. This arrangement answered perfectly well as it was found that when a litre of water at 45 C. was placed in it the temperature only fell to 41·8 C. after standing 10 minutes in a room at 18·6 C., a loss so small as to have no practical influence on the following determinations:—

I. A single piece of carbide of known weight was dropped into a litre of water at known temperature in the insulated beaker, and the moment gas ceased to come off the increase of temperature in the water was noted.

The results of these experiments were as follows:—

I.

| Grms. of Water taken. | Grms. of Calcic Carbide. | Rise in Temperature C. | Corresponding Calories liberated. | Number of Calories liberated per Grm. of Carbide. | Time of Reaction in Seconds. |
|---|---|---|---|---|---|
| 1,000 | 42·7 | 17·4 | 17,400 | 407 | 62 |
| 1,000 | 28·9 | 11·4 | 11,400 | 394 | 91 |
| 1,000 | 19·7 | 8·2 | 8,200 | 416 | 73 |

or as a mean result 1 grm. of calcic carbide on decomposition liberates 406 calories.

II. Broken-up carbide the pieces of which weighed from 0·8 to 4·8 grms. were weighed into quantities of 30, 40, and 50 grms. respectively, and these were thrown into a litre of water in the beaker with the following results:—

II.

| Grms. of Water taken. | Grms. of Calcic Carbide. | Rise in Temperature C. | Corresponding Calories liberated. | Number of Calories liberated per Grm. of Carbide. | Time of Reaction in Seconds. |
|---|---|---|---|---|---|
| 1,000 | 50 | 17·6 | 17,600 | 352 | 248 |
| 1,000 | 50 | 18·4 | 18,400 | 368 | 86 |
| 1,000 | 50 | 18·3 | 18,300 | 366 | 123 |
| 1,000 | 50 | 18·4 | 18,400 | 368 | 106 |
| 1,000 | 40 | 15·6 | 15,600 | 390 | 109 |
| 1,000 | 40 | 15·8 | 15,800 | 395 | 101 |
| 1,000 | 40 | 15·0 | 15,000 | 375 | 193 |
| 1,000 | 40 | 15·5 | 15,500 | 387 | 110 |
| 1,000 | 30 | 11·7 | 11,700 | 390 | 76 |
| 1,000 | 30 | 10·8 | 10,800 | 360 | 89 |
| 1,000 | 30 | 11·8 | 11,800 | 393 | 114 |
| 1,000 | 30 | 11·4 | 11,400 | 380 | 85 |

or 1 grm. of carbide liberates 377 calories.

III. The last experiment was again repeated but the carbide was a little more finely broken. The mean result being that 1 grm. of carbide liberates 384 calories.

These figures show the somewhat unexpected result that the more finely broken-up pieces developed the least heat, but a moment's consideration shows the reason for this. The weighing out of an exact quantity of the small carbide is a slower operation than taking the weight of one large piece, whilst the larger surface presented by the broken carbide to the action of moisture in the air all tend to cause the decomposition of a larger proportion of the fine than of the large lump carbide, and hence render it of poorer quality, whilst the more rapidly the gas is evolved the less thoroughly will it be cool, and subsequent experiments fully confirmed these as being the causes of the discrepancies.

III.

| Grms. of Water taken. | Grms. of Calcic Carbide. | Rise in Temperature ° C. | Corresponding Calories liberated. | Number of Calories liberated per Grm. of Carbide. | Time of Reaction in Seconds. |
|---|---|---|---|---|---|
| 1,000 | 50 | 19·4 | 19,400 | 388 | 492 |
| 1,000 | 50 | 19·6 | 19,600 | 392 | 142 |
| 1,000 | 50 | 19·8 | 19,800 | 376 | 351 |
| 1,000 | 50 | 19·1 | 19,100 | 382 | 144 |
| 1,000 | 40 | 15·8 | 15,800 | 395 | 78 |
| 1,000 | 40 | 15·7 | 15.700 | 392 | 94 |
| 1 000 | 40 | 15·5 | 15.500 | 387 | 162 |
| 1,000 | 40 | 15·3 | 15,300 | 382 | 72 |
| 1,000 | 30 | 11·2 | 11,200 | 373 | 71 |
| 1.000 | 30 | 11·1 | 11,100 | 370 | 78 |
| 1,000 | 30 | 11·6 | 11,600 | 387 | 69 |
| 1,000 | 30 | 11·5 | 11,500 | 383 | 109 |

It is manifest, therefore, the 406 calories will most nearly represent the heat evolved by the decomposition of 1 grm. of 91·3 per cent. calcic carbide, and this would be equivalent to 444·6 calories for pure carbide.

This portion of the work had already been completed when experiments were published from the Imperial Technical Institute at Charlottenburg to determine the rise of temperature given when Neuhausen carbide is decomposed by immersion in a large excess of water. No analysis of the carbide is given, and the results, which were reprinted in the Journal of Gas Lighting for March 22, page 645, are the best tribute that could be paid to the superiority of the English carbide, as the mean of the Charlottenburg determinations gives 260·5 calories per grm. of carbide, which would mean that the samples used contained only some 58 to 59 per cent. of true carbide.

Taking 406 calories as the heat evolved on the decomposition of a good commercial carbide, it is at once evident that this action evolves about one-twentieth the heat developed by the complete combustion of carbon, but as the intensity of the temperature developed is a function of the time needed to complete the action, and as the decomposition of the carbide in contact with water is extremely rapid, it is manifest that the temperature developed may be so high as to lead to serious disaster.

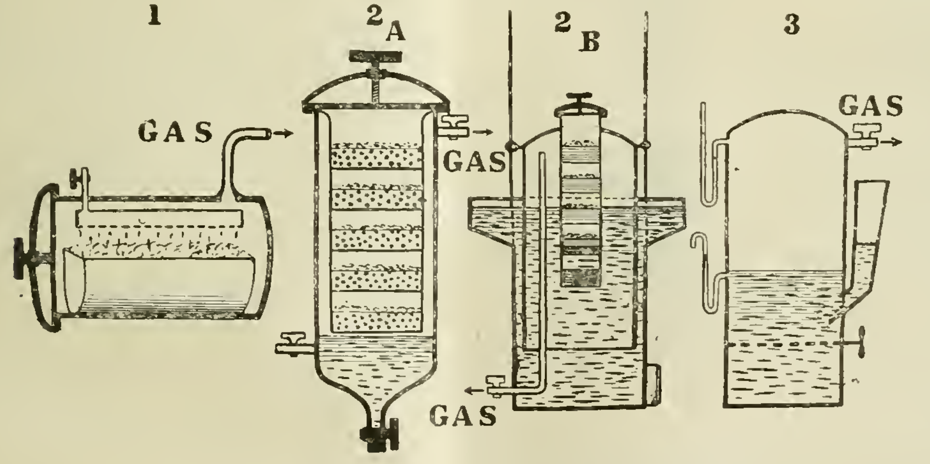
All the generators at present before the public may be conveniently classified under three heads :—

I. Those in which water is by various devices allowed to drip or flow in a thin stream on to a mass of carbide, the evolution of the gas being regulated by the stopping of the water feed.

II. Those in which water in volume is allowed to rise in contact with the carbide, the evolution of the gas being regulated by the water being driven back from the carbide by the increase of pressure in the generating chamber.

III. Those in which the carbide is dropped or plunged into an excess of water ;

and attempts were now made to obtain definite data as to the temperatures developed when the chemical action between the carbide and the water was taking place under these various conditions.



TYPES OF GENERATORS

In all the following experiments the temperatures reached in the mass of decomposing carbide were measured by the Le Chatelier thermo-couple, the general arrangement of which has been so often described that it is unnecessary to again repeat it.

A resistance of 19·15 ohms was introduced into the circuit, and the couple was calibrated with every possible precaution, immersion in a paraffin bath, the temperature of which was checked by a standard thermometer, being used to fix the points up to 300° C., whilst the boiling points of sulphur and tin protochloride were taken to give 448 and 606° C., whilst above this point it is now well known that the line is perfectly straight.

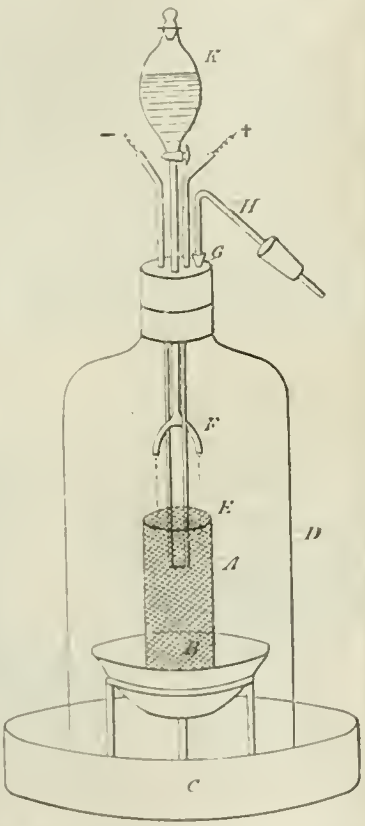
The first set of experiments was made to determine the temperature generated when water drips upon carbide, and considerable difficulty was found in arranging the apparatus in such a way as to make it a fair test of what really takes place in practice. After several failures the form of apparatus which was finally adopted was as follows :—

A cylindrical cage of wire gauze (A) for holding the carbide was stood in a porcelain dish (B) supported by an iron tripod standing in the circular dish (C), which acted as a mercury seal for the bell jar (D). The tubulure of the bell jar was closed by a cork carrying the two glass tubes insulating the thermo-couple, the dripping funnel (K) fitted with a stop cock and a double jet (F) at its lower extremity, and a wide brass tube (G) closed with a cork through which passed the delivery tube (H) (Fig. 2, see next page).

Having about half filled the carbide cage (A) with the 91·3 per cent. carbide it was placed in position, standing on a piece of asbestos card in the porcelain basin and the bell jar placed over it, the bottom being sealed with 2 c.m. of mercury in the bottom dish, the cork was put in position, the thermo-couple being raised or lowered until it was at the required depth in the carbide cage, which was then filled up over the couple by dropping in carbide through the brass tube. When the quantity of carbide to be used had been charged in, the brass tube was closed by a cork bearing the delivery tube, which in turn was connected to a condenser and collecting bottle, and the water was run in in known quantities by the dripping arrangement, readings of temperature being taken every minute.

The charge of carbide employed in every case was half a pound, and a very large number of experiments was made, as it soon became evident that owing partly to the non-conducting properties of the lime formed, which in many cases crumbled off from the surface of the decomposing carbide and coated the thermo-couple, and partly also to the couple only being about 7 mm. in length and so only recording the temperature of a very small portion of the charge of carbide, the temperature readings were often manifestly too low, as actions, such as the formation of tar vapours, could be observed going on in parts of the charge whilst the thermo-couple was registering a temperature 200 or more degrees less than was known to be needed for the action visibly taking place.

Fig. 2.



Moreover, the streams of water impinging upon the carbide must not flow directly on to the thermo-couple or it is manifest that correct readings cannot be obtained.

The first dozen experiments were necessary to show the various factors which had to be taken into account in making the determinations, and when the necessary experimental conditions were realised, then the determinations began to show something like uniformity in result. The data deduced from the experiments are that when water is allowed to drip or flow in a thin stream upon a mass of calcic carbide, the temperature rapidly rises until after about 18 to 25 minutes the maximum is reached, this varying according to circumstances to from 400 to 700 C., and it is probable that in some parts of the mass the higher limit is nearly always attained, traces of tar being generally found in the residual lime, and in some cases in sufficient quantity to make the lime yellow and pasty, whilst vapours of benzene and other polymerisation products freely pass off with the gas.

The maximum temperature rarely lasts a minute, and the fall in temperature proceeds rapidly from 15 to 20 minutes and then more slowly until the action ceases.

The readings were in each case taken every minute for an hour, and it will be sufficient here to give one set as fairly typical of the time and temperature factors.

*Thermo-couple Readings per Minute.*

227 grms. of carbide used and 330 grms. of water dripped on during the first 40 minutes.

| Minute. | C. | Minute. | ° C. | Minute. | ° C. | Minute. | ° C. |
|---|---|---|---|---|---|---|---|
| 1 | .. | 16 | 452 | 31 | 558 | 46 | 417 |
| 2 | 97 | 17 | 616 | 32 | 554 | 47 | 420 |
| 3 | 154 | 18 | 648 | 33 | 553 | 48 | 425 |
| 4 | 168 | 19 | 674* | 34 | 551 | 49 | 423 |
| 5 | 182 | 20 | 644 | 35 | 540 | 50 | 421 |
| 6 | 204 | 21 | 630 | 36 | 508 | 51 | 417 |
| 7 | 209 | 22 | 623 | 37 | 497 | 52 | 414 |
| 8 | 218 | 23 | 625 | 38 | 470 | 53 | 402 |
| 9 | 223 | 24 | 623 | 39 | 430 | 54 | 398 |
| 10 | 244 | 25 | 619 | 40 | 393 | 55 | 390 |
| 11 | 250 | 26 | 616 | 41 | 417 | 56 | 387 |
| 12 | 265 | 27 | 600 | 42 | 418 | 57 | 383 |
| 13 | 284 | 28 | 580 | 43 | 417 | 58 | 375 |
| 14 | 290 | 29 | 580 | 44 | 415 | 59 | 378 |
| 15 | 317 | 30 | 572 | 45 | 418 | 60 | 375 |

* Maximum reading.

Leaving the question of the temperature developed in this class of generator, another important point is the length of time over which generation of gas continues after the addition of water to the carbide has ceased. The general idea which seems to exist amongst makers of automatic apparatus of this type is that all they have to do in order to stop the generation of acetylene is to stop the water supply; this, however, is an utter fallacy, as liberation of gas continues with ever-increasing slowness for sometimes an hour and three quarters after the water supply has ceased, whilst the gas so evolved is very considerable in volume. The length of time over which the generation extends will, of course, depend to a certain extent upon the amount of water added, the percentage of carbide undecomposed, and the temperature at which the mass of carbide happens to be when the water supply ceases, whilst the generation will itself depend upon—

*a.* The dehydration of the calcic hydrate first formed.

*b.* The decomposition of water condensed from the gas present as the temperature of the generator falls.

When water acts upon calcic carbide the first reaction probably is $CaC_2 + H_2O = CaO + C_2H_2$, whilst the lime so formed then takes up another molecule of water forming calcic hydrate $CaO + H_2O = Ca(HO)_2$. At temperatures of from 420—430 C., however, calcic hydrate begins to part with its water of hydration, and the affinity for water exerted by any undecomposed carbide still present leads to the reaction—$CaC_2 + Ca(HO)_2 = 2CaO + C_2H_2$.

A large number of experiments were made by placing a known weight of carbide in a generating cylinder, running in known volume and weight of water in a given time, and carefully measuring the volume of gas for the first 10 minutes and again when the action had practically ceased, and not more than 1 c.c. of gas was evolved in 10 minutes.

Tabulating the results obtained in the experiments we obtain the following:—

| Weight of Carbide in Grms. | Weight of Water in Grms. | Time taken to flow in in Minutes. | Gas c.c. in 10 Minutes. | Time to complete evolution. | Volume of Gas at end of evolution. | Percentage of Carbide not decomposed. |
|---|---|---|---|---|---|---|
| 18 | 9·0 | 4 | 2,100 | 80 | 2,900 | 50·0 |
| 18 | 9·0 | 6 | 2,300 | 90 | 2,800 | 51·5 |
| 18 | 9·0 | 8 | 2,400 | 70 | 2,700 | 43·4 |
| 18 | 13·5 | 4 | 2,900 | 60 | 3,300 | 43·0 |
| 18 | 13·5 | 6 | 3,000 | 55 | 3,500 | 39·7 |
| 18 | 13·5 | 8 | 3,300 | 65 | 3,600 | 37·9 |
| 18 | 18·0 | 4 | 3,700 | 60 | 4,100 | 29·3 |
| 18 | 18·0 | 6 | 3,600 | 62 | 4,200 | 27·6 |
| 18 | 18·0 | 8 | 3,700 | 65 | 4,200 | 27·8 |
| 36 | 18·0 | 4 | 4,600 | 88 | 4,300 | 54·3 |
| 36 | 18·0 | 6 | 4,650 | 81 | 4,350 | 53·2 |
| 36 | 18·0 | 8 | 4,400 | 57 | 5,560 | 52·0 |
| 36 | 27·0 | 4 | 5,600 | 130 | 6,600 | 43·1 |
| 36 | 27·0 | 6 | 6,000 | 72 | 6,650 | 42·7 |
| 36 | 27·0 | 8 | 5,850 | 64 | 6,850 | 41·0 |
| 36 | 36·0 | 4 | 7,000 | 69 | 7,850 | 32·2 |
| 36 | 36·0 | 6 | 7,100 | 54 | 8,400 | 27·6 |
| 36 | 36·0 | 8 | 7,400 | 49 | 8,500 | 26·7 |
| 54 | 27·0 | 4 | 5,900 | 105 | 6,600 | 62·1 |
| 54 | 27·0 | 6 | 5,800 | 78 | 6,300 | 63·3 |
| 54 | 27·0 | 8 | 5,800 | 85 | 6,400 | 62·6 |
| 54 | 36·0 | 4 | 6,000 | 102 | 6,950 | 60·0 |
| 54 | 36·0 | 6 | 6,300 | 79 | 7,500 | 56·9 |
| 54 | 36·0 | 8 | 7,000 | 109 | 8,900 | 48·8 |
| 54 | 54·0 | 4 | 6,600 | 90 | 8,800 | 49·4 |
| 54 | 54·0 | 6 | 7,700 | 103 | 8,900 | 48·8 |
| 54 | 54·0 | 8 | 7,600 | 105 | 10,400 | 40·0 |

These results are of very great interest, as they not only show clearly the facts already pointed out, but indicate that in any automatic apparatus on this principle the cut-off should be so arranged that at least one-fourth of the total holder capacity is still available to store the slowly generated gas.

Another very important deduction to be arrived at from the figures is the large excess of water over and above the theoretical quantity required to ensure complete decomposition of the carbide by this process, this being to a certain extent dependent upon the form of the generator.

According to theory 64 parts by weight of carbide require only 36 parts by weight of water to completely decompose them and convert the lime into calcic hydrate. This would mean that each pound of calcic carbide needs a little under half a pint of water to complete the action, whilst a glance at the table shows that owing to the evaporation due to the heat of the action, half the added water is driven off as steam with the acetylene or left mechanically adhering to the lime, and the smallest quantity likely to complete the action would be a pint to a pound of carbide, whilst in reality the only safe way is to add sufficient water to drown the residue.

If this is not done the lime forms so protective a coating to the carbide that small quantities often remain undecomposed, and if the residues are thrown into a drain or cesspool, the evolution of acetylene would give an explosive mixture, which on account of its low point of ignition would be a serious danger.

Points of considerable interest to the generator maker are the space occupied by a given weight of carbide, the volume of the lime formed from it on decomposition, and the volume of gas that can be evolved from a given space filled with carbide.

The density of calcic carbide is 2·2, and therefore, a cubic foot of solid carbide would weigh 137 pounds. In practice, however, the weight of carbide which can be got into a cubic foot space depends upon the size to which it is broken, and with the size commercially sent out a fair average would be 80 pounds per cubic foot of carbide space, and this weight of carbide at 5 cb. ft. per lb. would yield 400 cb. ft. of acetylene.

One pound of pure calcic carbide yields 1·15 lbs. of slaked lime (one kilo. of carbide yields 1,156 grams of slaked lime), and the volume this will occupy depends entirely upon the way in which the water is brought in contact with it.

In an automatic generator of the first class where water drips slowly upon the carbide in sufficient quantity to decompose it but not to flood it, the lime swells up and occupies 2 to 2·5 times the bulk of the original carbide, when, however, the water flows in more rapidly, the impact of the water beats down the lime and the space occupied is not so large.

In generators of the second type in which water rises from below, the weight of the undecomposed carbide above it presses down the lime below and keeps it in a compact mass occupying about one half more space than the carbide from which it was formed.

With the third type of generator it really becomes a question of the rate at which the excess of undissolved calcic hydrate settles, and this will be discussed later on.

The large proportion of water vapourised during the evolution of the acetylene at once draws attention to the necessity of arranging all the generator connections in such a way that condensation shall not lead to stoppage of the delivery pipes.

Where a holder capable of taking the full make of gas from the largest charge of carbide the generator can contain is employed, a drip pipe or catch box between the generator and the holder is all that is practically necessary, but where a generator is supposed to be sufficiently automatic not to need a holder, every precaution must be taken to ensure complete condensation before distribution.

It must also be clearly borne in mind that the liquid products condensible from the gas are of the most corrosive character both to paint and metal, and one of the problems the generator maker will find himself confronted with in the near future is the rapidity with which the paint in the interior of the holder and tank gets attacked.

In order to determine the composition of the liquid products, the gas on leaving the apparatus was made to pass through a Liebig's condenser, but it at once became manifest that with the apparatus I have described, by far the largest proportion of the condensation went on in the bell jar, the walls running down with liquids and even tar.

Another form of apparatus was therefore adopted consisting of a distillation flask with side tubulure, and the bottom of the flask having been covered with sand, the carbide was placed in it, and the tubulure connected with the condenser, water being run in upon the carbide from a dripping funnel as before.

The heat generated caused frequent breakage of the flask, but a considerable quantity of a strongly alkaline yellowish liquid, containing ammonium sulphide, ammonium hyposulphite, and phosphorus compounds was condensed.

There is no doubt that acetylene under certain conditions will attack such metals as copper and silver with formation of explosive acetylides, and this action is accelerated by the presence of ammonia and moisture, but the liquid condensing from the acetylene would probably to a certain extent prevent this by coating any copper or copper alloys exposed to it with a dense crust of sulphide.

It is manifest, however, that no copper or brass should be used in generator fittings, partly because of the action of the acetylene itself but still more because of the rapid action the ammonium sulphide would have upon them, and which would quickly cause the "setting" hard of taps or valves made of copper alloys.

If the generator and holder be painted inside with an ordinary paint the alkalinity of this liquid would cause rapid stripping, partly due to saponification of the vehicle and partly to disintegration caused by conversion of the lead in the priming, boiled oil, and if white lead is used, in the paint itself into sulphide.

It will probably be found expedient for these reasons to use enamelled-lined tubes in the generator, and to coat all parts of the apparatus liable to come in contact with the crude gas with a specially prepared paint capable of withstanding alkalis and containing no lead.

In the second class of generators in which contact between the carbide and water is brought about either by water rising from below to a mass of carbide suspended in some form of cage, or by a cage of carbide suspended in a moveable bell which as it falls dips the carbide into water and again withdraws it as excessive generation of gas causes the bell to rise, it was to be expected that many of the drawbacks of the previous class were to be found, and experiment soon showed that under certain conditions these generators were far worse than the former.

I have several times come across phenomena in the practical working of generators of this class which could only be accounted for on the supposition that the decomposing point of the acetylene had been reached, and the following experimental results fully demonstrate not only that this is so, but the complications which arise from it.

The same bell jar was used as in the former set of experiments, the arrangement of cork, tube for charging, thermo-couple, and delivery tube being also the same, but the cage carrying the carbide was suspended to the cork, whilst a collar fitted around the neck of the bell jar admitted of its being made into the bell of a gas holder by suspending it in a glass tank of water, lines attached to the collar being passed over pulleys in a frame above and so arranged as to allow the bell to be counterpoised.

The couple was so arranged as to have a central position in relation to the walls of the holder, and to be about three-quarters of an inch (17 mm.) below the surface of the carbide, the twist of platinum and platinum-rhodium being as before about 7 mm. in length.

The charge of half a pound (226·8 grms.) of carbide having been introduced into the cage with the thermo-couple in position, the counterpoise weights were removed, and the cock on the delivery tube being opened, the bell sank in the water until the carbide cage reached the surface, when the sudden evolution of gas caused the bell to again rise until the rate of delivery exceeded the rate of generation, when it again sank and immersed the carbide, this action continuing until the carbide was all decomposed.

The thermo-couple being in the upper part of the charge, the rise of temperature is not at first shown, but as the action approaches the neighbourhood of the couple, it registers a rapid increase of temperature, and the action going on is generally rendered invisible by the steam, white vapours and even brown tar fumes coming off from the lower portion of the charge.

In from 12 to 18 minutes from the start, the maximum temperature is reached at the spot affecting the couple, and soon after the temperature falls as rapidly as it rose.

The following table gives the temperature readings in four experiments:—

*Thermo-couple Readings per Minute.*

| Minutes. | Centigrade. I. | II. | III. | IV. |
|---|---|---|---|---|
| 1 | .. | .. | .. | .. |
| 2 | .. | .. | .. | .. |
| 3 | .. | .. | 80 | .. |
| 4 | .. | .. | 122 | .. |
| 5 | 102 | 137 | 170 | .. |
| 6 | 119 | 178 | 218 | .. |
| 7 | 180 | 200 | 251 | 150 |
| 8 | 204 | 246 | 287 | 178 |
| 9 | 227 | 288 | 326 | 233 |
| 10 | 249 | 338 | 413 | 245 |
| 11 | 270 | 413 | 727 | 342 |
| 12 | 298 | 620 | 754 | 393 |
| 13 | 336 | 734 | 568 | 764 |
| 14 | 4 8 | 727 | 466 | 793 |
| 15 | 461 | 706 | 428 | 807 |
| 16 | 594 | 701 | 385 | 793 |
| 17 | 703 | 636 | 347 | 761 |
| 18 | 654 | 597 | 168 | 725 |
| 19 | 570 | 478 | 131 | 623 |
| 20 | 514 | 381 | .. | 577 |
| 21 | 443 | 254 | .. | 594 |
| 22 | 190 | .. | .. | 252 |
| 23 | 123 | .. | .. | 124 |
| 25 | 98 | .. | .. | .. |

From this it is seen that excessive heating took place in every case, whilst in one it was well above the decomposing point of acetylene, a thin cloud of black smoke being formed immediately around the carbide, whilst tar vapour poured off from it, and on removing the residue after the experiment it was found to be coated with soot and loaded with tar.

On several occasions the whole of the charge on removal from the generator, when the maximum temperature was reached, was found to be at a glowing red heat, and I was struck by the fact that the smell of acetylene could hardly be distinguished in the strong tarry odour that was evolved.

The moment that acetylene is subjected to the action of high temperatures changes of great complexity at once commence. These at first are purely synthetical; at temperatures which are low as compared to those which are described above the acetylene begins to condense to benzene; as the temperature rises the condensation of four molecules of acetylene yield styrolene; a further increase in the temperature may cause the styrolene and benzene to interact, yielding anthracene and hydrogen, and it is probably at this point that the brown tar vapours appear while naphthalene makes its appearance. These changes, however, still have to be accurately studied. At this temperature, moreover, a fresh set of interactions start, the nascent hydrogen combines with acetylene to form ethylene, and this body under the action of heat breaks down to methane and acetylene once more.

The earlier actions of necessity lead to a great loss in the volume of the acetylene. Dr. Haber found that 15 litres of acetylene when heated for a considerable period to 638 C. left only 10 litres of gas.

Probably no such condensation as this takes place in an acetylene generator. When the outer layer of carbide decomposes, the gas is evolved so rapidly that there is no time for the heat to act upon it, but as the decomposition spreads into the centre of the mass, the acetylene generated has to pass through the external layers, which, as shown, may be at a temperature above the point of its decomposition, and it is under these conditions that a considerable volume of gas is lost, and the tar, often found in the residue, or distilled out into generator and tubes, is formed.

In generators in which excessive heating takes place, this tar is likely to cause considerable trouble as it is of a very viscous character, and if it condenses in the delivery tubes causes the lime-dust and carbon particles to collect and bring about stoppage.

A still more important evil, however, is to be found in the alteration which takes place in the composition of the gas, and which reduces the illuminating value of the gas to a serious extent.

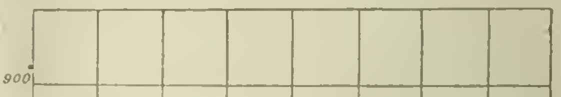
During one of the experiments samples of the gas were taken as the maximum temperature was approached, and were analysed with the following results:—

| — | I. | II. |
|---|---|---|
| Acetylene | 70·0 | 69·7 |
| Saturated hydrocarbons | 11·3 | 11·4 |
| Hydrogen | 18·7 | 18·9 |
| | 100·0 | 100·0 |

and this alteration in composition reduces the illuminating value of the gas from 240 candles to about 126.

If the temperatures determined in these experiments be arranged as curves in which time and temperature are taken as factors, it is at once apparent that a very considerable proportion of the generation takes place at a temperature above 600 C., about which point polymerisation commences. As benzene forms a large proportion of it, it is carried forward as vapours and remains suspended even in its passage through the gas holder and delivery pipes. Benzene requires three times the volume of air for combustion that acetylene does, and the result is that the most perfect acetylene burner shows a tendency to smoke directly any quantity of benzene is formed.

Fig. 3.



Still more serious, however, is the action of the burner tip. One of the greatest troubles in the utilisation of pure acetylene is the question of finding a burner in which to consume it, and it was soon realised that the best of the burners first introduced could only be used for a few hundred hours before a growth of carbon appeared on the nipple, distorted the flame and brought about smoking of the most pronounced character. A year ago it was thought that this trouble had been overcome by the introduction of the Naphey burner and the various imitations of it that were at once put upon the market, but extended experience shows that even these burners are not infallible, and many who have watched the continuous use of acetylene, especially in those parts of the Continent where the gas has been adopted in its pure state as a town supply, declare that the burner question is as far off solution as ever. If a burner which has started smoking be examined, an arborescent growth of filiform carbon is noticed at the aperture or slit, and the general idea is that this has been formed by the overheating of the acetylene by the nipple causing its decomposition with formation of the carbon deposit. On breaking the steatite top of the burner, however, it is found that the material is carbonised for a considerable distance into the body of the steatite, and it is manifest that this has been caused by the deposition of a liquid hydrocarbon, which has soaked into the material and been carbonised there.

If smoke or tar vapour be examined under a high microscopic power it is seen that they consist of minute vesicles or bubbles in a most marvellously active condition of movement, and fulfilling in a most perfect manner the conception one forms of molecular motion. Ever bombarding each other but never colliding, these small vesicles filled with gaseous matter continue their career until some mechanical action bursts them and deposits the minute trace of liquid which formed the skin of the microscopic balloon.

It is for this reason that the most successful forms of washer for extracting tar during the manufacture of gas consist of fine jets or orifices through which the gas passes at considerable velocity and comes in contact with a baffle which breaks up the vesicles; and anyone with experience in water-gas making knows the trouble that arises from filiform growths of carbon on the burner when, owing to an insufficient temperature in the cracking and superheating chambers, the carburetted gas contains vapours instead of permanent gaseous products.

When acetylene has been made in a generator at an undue temperature, it carries with it benzene vapour, which, as it commences to condense, assumes this vesicular form, and on coming to the extremely minute holes which form the apertures of the burner, the mechanical scrubbing which it encounters causes the breaking up of the vesicles and the deposition of the benzene and other hydrocarbons held in suspension by benzene, which soak into the steatite and carbonise. The presence of finely-divided carbon has a great effect in determining the decomposition of acetylene itself, so that a rapid growth of carbon takes place at the burner, and no ordinary cleaning of the deposited carbon from the exterior will ever make the nipple fit for constant use again.

It will be found with experience that the prevention of smoking in a burner will be overcome quite as much by attention to the temperature in the generator as to the burner itself, and where a generator is in use which gives overheating, a well-arranged scrubbing apparatus that would get rid of the benzene from the gas would be found a distinct advantage in stopping burner troubles.

At first sight these results seem an absolute condemnation of the second class of apparatus, but the fact remains that they constitute a very large percentage of the generators on the market, and that the best of these show no signs of the great overheating made manifest by these experiments.

The reason of this apparent anomaly is that under certain conditions, which can be clearly defined, excessive heating is avoided.

The rising bell which draws a mass of wet carbide above the surface of the water should at once be discarded, it is bad from every point of view. But generators in which water rises from below and so attacks the carbide can be made safe if the arrangements are such that the water is never driven back from the carbide and the bulk of carbide is sufficiently subdivided.

Under these conditions the slowly rising water is always in excess at the point where it decomposes the carbide, so that the evaporation by rendering heat latent keeps down the temperature, and although the steam so formed partly decomposes the carbide in the upper portion of the charge, the action is never sufficiently rapid to give anything approaching a red heat. In order to fulfil these conditions it is necessary that there should be a holder of considerable capacity and that the leading tube conducting the gas from the generator to the holder should be of sufficient diameter to freely conduct away the gas, the water at the same time being allowed to rise in the generator so slowly as to do away with any risk of over-generation.

In the best generators of this class these conditions are more or less approached, and it is unusual to find that the melting point of tin, 228° C., has been reached in the charge of carbide during decomposition.

Where apparatus of this class are automatic and have no rising holder to take the gas, it is found that they work satisfactorily when supplying the number of lights for which they were designed, but if they are over-driven and the action becomes too violent, excessive heating takes place, whilst the turning off of the gas and consequent driving back of the water from the carbide also has a tendency to cause it. If, however, the water has risen sufficiently slowly, the carbide below the surface has been practically all decomposed, so that the heating only takes place over a limited zone.

The maker of apparatus that are liable to give rise to excessive heating invariably deny the possibility of such an action taking place with their generators, and when it is proved to them that it is so, fall back upon the defence that even if the mass does become red hot there is no particular danger.

In such generators the active danger of explosion only exists whilst any air is left mixed with the acetylene, and in the form of apparatus which have holders to take the gas as it is formed, the air remaining in the generator is swept rapidly over into the holder and out of the range of the source of heat, but with automatic generators this is not always the case, and the air space in the generators should always be made as small as possible, and some arrangement should be adopted if possible by which the air in the generator could be rinsed out by a little of the previously produced acetylene, before decomposition of the carbide by water commences.

Under these circumstances danger from explosion during generation would disappear, but the drawbacks of smoky flames, reduced illuminating power, and choking tubes would still remain.

Undoubtedly the best forms of generators, and the only ones which from a scientific point of view should be employed, are those of the third class, in which carbide is allowed to fall into an excess of water, but unfortunately there are but few examples of this class at present made.

In such generators, as long as there is water present, it is impossible to get above a temperature of 100° C., whilst with a properly arranged tank the temperature never exceeds the air temperature by more than a few degrees. Under these conditions the absence of polymerisation and the washing of the nascent and finely-divided bubbles of gas by the lime water in the generator yields acetylene of a degree of purity unapproached by any other form of apparatus.

The one thing that has militated against it is that it is not easy to design such a generator which shall be automatic, and as this seems to be the craze from which the generator makers are at present suffering, its advantages have been apparently overlooked by them, although fully recognised by all scientific men.

In designing such a generator, the tank containing the water into which the carbide falls should be provided with a false bottom so as to leave at least 8 ins. to 1 ft. of water below the point at which the carbide is decomposed for the lime sludge to settle in, and experiments were made to see the rate at which the settling of the excess of lime took place.

In these experiments known weights of calcic carbide were dropped into a beaker containing a litre of water, and the results obtained were as follows:—

I.

| Water taken. | Calcic Carbide. | Volume of Lime paste deposited during 30 mins. | Volume of Lime paste deposited during 90 mins. | Volume of Lime paste deposited during 30 mins. Calc. for 1 grm. $CaC_2$. | Volume of Lime paste deposited during 90 mins. Calc. for 1 grm. $CaC_2$. |
|---|---|---|---|---|---|
| Grms. | Grms. | c.c. | c.c. | c.c. | c.c. |
| 1,000 | 50 | 480 | 360 | 9·6 | 7·2 |
| 1,000 | 50 | 470 | 340 | 9·4 | 6·8 |
| 1,000 | 50 | 440 | 350 | 8·8 | 7·0 |
| 1,000 | 50 | 470 | 370 | 9·4 | 7·4 |
| 1,000 | 40 | 430 | 330 | 10·7 | 8·0 |
| 1,000 | 40 | 420 | 320 | 10·5 | 8·0 |
| 1,000 | 40 | 400 | 310 | 10·2 | 7·7 |
| 1,000 | 40 | 410 | 320 | 10·5 | 8·0 |
| 1,000 | 30 | 340 | 240 | 11·0 | 8·0 |
| 1,000 | 30 | 320 | 220 | 10·3 | 7·3 |
| 1,000 | 30 | 330 | 240 | 11·0 | 8·0 |
| 1,000 | 30 | 330 | 240 | 11·0 | 8·0 |
| | | | Mean | 10·2 | 7·6 |

II.

| Water taken. | Calcic Carbide. | 30 mins. | 90 mins. | 30 mins. Calc. for 1 grm. $CaC_2$. | 90 mins. Calc. for 1 grm. $CaC_2$. |
|---|---|---|---|---|---|
| 1,000 | 50 | 450 | 340 | 9·0 | 6·8 |
| 1,000 | 50 | 440 | 330 | 8·8 | 6·6 |
| 1,000 | 50 | 450 | 320 | 9·0 | 6·6 |
| 1,000 | 50 | 440 | 320 | 8·8 | 6·2 |
| 1,000 | 40 | 390 | 310 | 9·7 | 7·7 |
| 1,000 | 40 | 410 | 310 | 10·0 | 7·7 |
| 1,000 | 40 | 390 | 300 | 9·7 | 7·5 |
| 1,000 | 40 | 390 | 300 | 9·7 | 7·5 |
| 1,000 | 30 | 310 | 210 | 10·3 | 7·0 |
| 1,000 | 30 | 300 | 240 | 10·0 | 8·0 |
| 1,000 | 30 | 290 | 230 | 9·6 | 7·6 |
| 1,000 | 30 | 300 | 220 | 10·0 | 7·3 |
| | | | Mean | 9·8 | 8·6 |

After 30 minutes the volume of lime was 10 c.c. per grm. of carbide.

After 90 minutes the volume of lime was 8·1 c.c. per grm. of carbide.

So that approximately, after an hour's standing, each kilo. of calcic carbide will give 10 litres of lime sludge, or 1 lb. of calcic carbide will yield 8 pints, which can be got rid of by a sludge cock at the bottom of the apparatus. The rapidity with which settling takes place is of course slightly affected by the form of the apparatus.

There are many other points of interest connected with the various forms of generators and their practical working, but time forbids discussion of these on the present occasion, and I can only hope that the facts brought before you may be found of utility by the workers who are endeavouring to facilitate the use of this beautiful illuminant.

In conclusion, I desire to express my warm appreciation of the help given me by Dr. A. Polis in the experimental work detailed in this paper.

### Discussion.

Mr. Boverton Redwood considered that the Society was greatly indebted to Prof. Lewes for the manner in which he had put the subject before them, for he had not only given them information of much interest to chemists, but, with his characteristic grasp of the practical side of the question, had introduced deductions which could not fail to be of great practical value, not merely to those who had occasion to use acetylene as a source of light, but also to those who were devising appliances for the production of it from calcium carbide. Possibly the paper might lead to a not inconsiderable massacre of the innocents in the shape of the offspring of the fertile brains of inventors, but, on the other hand, inventors would have reason to thank Prof. Lewes for the suggestive remarks which he had made. It was evident that, apart from the danger which might arise from the creation of undesirably high temperatures in the conversion of calcium carbide and water into acetylene, the process could not be carried out economically and efficiently if such an amount of heat as had been referred to were produced in the generator. The lecturer had made it clear that in some forms of apparatus there was considerable loss of acetylene, due to the high temperature evolved, and that the action which he had described might lead to the formation of a smoky flame. Some members present were old enough to remember the camphene lamp, in which rectified oil of turpentine was burned with a wick. A beautiful flame, in some respects resembling that of acetylene, though of lower illuminating power, was thus obtained, but those who had had experience of that lamp would readily call to mind the nuisance created when smoking commenced. If, then, by the moderation of the temperature at which the acetylene was produced the tendency to smoking of the flame could be prevented, Prof. Lewes would have conferred a boon on those engaged in the problem of constructing suitable appliances for the generation of the gas, as well as on those who employed it as an illuminant. It was quite clear that a great future lay before acetylene, but probably there was still room for improvement in the form of the burner employed. Prof. Lewes had alluded to an approaching exhibition of acetylene generating apparatus. He (the speaker) thought it was high time that some action was taken to indicate to the public which types of apparatus were best adapted for use in the generation of the gas, from the point of view both of safety and of economy. It would not, in his opinion, have been out of place if the local authorities, or even the Government, had taken some action in this matter, or if the insurance companies had carried out some experiments with the object of showing the public which appliances might be safely employed; but as none of these bodies had thought fit to do so, it was a matter for congratulation that the Imperial Institute, in conjunction with the Society of Arts, should have made arrangements for holding this exhibition, and he thought that both manufacturers and users of the apparatus were under obligations to those institutions for the course they had adopted.

Mr. M. F. G. Worth, *à propos* of the author's reference to Foyers, said that they had done their best there to make carbide since 1894. It had sometimes been objected that their carbide was of low quality, but he had always held that that was not the fault of the carbide, but of the apparatus used; and after listening to Prof. Lewes's paper he could most cordially support the commendations of the last speaker, for the author had clearly proved that in the vast majority of cases failure in the use of acetylene gas was due to faults in the generator. Their experiments at Foyers had been in progress since 1895, and carried them over all the ground traversed by the author that night, and had involved the trial of several hundreds of burners, each costing about 20., and nearly all of them bad. The difficulties to be overcome were so great that they had eventually decided to drop the manufacture of generators, and confine themselves to the carbide. If makers would but avoid claiming that their machines would give 60 lights when they would really only provide 10, many of the disadvantages the author had referred to would be avoided. Referring to the three classes of apparatus described by Prof. Lewes, he had for some time been met with the objection that the third class would never give off 5 cb. ft. of gas. He would like to ask Prof. Lewes whether a mass of carbide dropping into water and coming into contact with oxygen and hydrogen in immense quantities could possibly produce a much greater local heat than if the water rose gradually to the carbide. If it were possible, that would not account for the fact that with the class of generator in which the carbide was dropped into the water poorer results were obtained than in the other class.

Mr. W. J. Atkinson Butterfield considered the paper the best *résumé* on the subject that had appeared. Noticing that the author had stated that there were two methods of bringing about the reaction, he had expected that only two classes of generators would be referred to—one in which the reaction was brought about by bringing carbide into a large excess of water, and the other in which a small quantity of water was brought in contact with an excess of carbide. It seemed to him that there was a vast distinction between these two classes of apparatus. The division into three classes which Prof. Lewes had given had the weight of authority, for it was to be adopted in the coming exhibition in London. Dr. Caro had, however, recently given a rather different classification. He also adopted three divisions but combined Prof. Lewes's Nos. 1 and 2*b* in one class and made another class comprising 2*a* and a type not represented that evening, in which water flowed over vertical partitions in a horizontal tray, and so flooded portions of the carbide in turn. His third class was the same as Prof. Lewes's No. 3. He had studied over 200 specifications of generators, and thought they could be logically divided into only two classes, *viz.*, (1) those having an excess of water, and (2) those having an excess of carbide at the *locale* of the reaction; and these two distinct methods of bringing about the reaction governed the temperatures which prevailed in the generators. The possible temperature had been stated by Gerdes to be about 1,200° C. He had calculated that 1,030° C. would represent the theoretically possible rise of temperature, and Prof. Lewes had that night given figures which showed the actual rise [in which, of course, conduction had been taken into account] to be 700° C. or thereabouts. These figures differed from those of Dr. Caro, who stated that in the dripping apparatus he found a rise of only about 200° C., and in the contact apparatus, and the one in which the water flowed horizontally, the rise was something under 300° C. Probably Prof. Lewes's means of determination were better than those of Dr. Caro, who had used alloys and a maximum-recording thermometer. He was much interested in the figures given by Prof. Lewes for the quantity of phosphoretted hydrogen in the gas, ranging from 2·3 in the worst to 0·5 in the English. Dr. Caro, in a similar examination, had found a maximum with the dip apparatus of about 0·5 per cent., and his average was only 0·038 per cent. It appeared, therefore, that continental carbide in general was not so impure as had been represented. He was also interested to learn that calcium phosphide was less easily decomposed by water than calcium carbide. He believed that it was a curious fact that the first quantities of gas generated from carbide contained a higher percentage of phosphoretted hydrogen than those generated later. Prof. Lewes had said that in the dripping apparatus he had found the "after-development" amount to a quarter of the whole yield, whereas he believed other observers had found 30 per cent., and with the dip class of apparatus as much as 40 per cent. He supposed that was explained by the fact that more aqueous vapour passed over the layers of carbide in the dip apparatus, owing to the higher temperature, and had a more complete action on the carbide. In the experiments in which Prof. Lewes showed the effect of the presence of benzene on the acetylene flame he noticed that he carefully regulated the supply of pure acetylene, so that the flame with it was smokeless, but he had not regulated the supply of acetylene saturated with benzene. Perhaps he could say what would happen if he had so regulated the supply of benzenised acetylene as to get the least smoky flame with it.

Prof. F. **Clowes** heartily supported Mr. Redwood's eulogy. He felt that he owed a personal debt of gratitude to Prof. Lewes; for, some years ago, when he first brought acetylene to the public notice at Nottingham, Prof. Lewes had kindly placed at his disposal all the information on the subject which was then available. He had since then learned that M. Pictet had brought out an admirable series of researches, under the title "L'Acetylène: son passé, son présent, son avenir," which, until recently, was the latest information on the subject of the preparation and use of acetylene. M. Pictet maintained that the form of generator in which the carbide was allowed to drop into a large volume of constantly cooled water was the only one which was absolutely safe. He stated that in the case of a generator in which the water was allowed to drip upon the carbide, or in which the water was drained off from the carbide after it had wetted the carbide, there was not only decomposition of the acetylene, but also danger of explosion from the action of the intensely heated lime upon the acetylene. Prof. Lewes had much diminished their apprehension of danger by stating that such an explosion could only occur when the pressure within the generator reached at least two atmospheres; and in the apparatus referred to by Prof. Lewes such a pressure could not be attained under any ordinary conditions. He would like to know whether Prof. Lewes had experienced in his experiments any such evidence of danger as M. Pictet had referred to.

Mr. R. J. **Friswell** confessed that in listening to the paper he had experienced some delight in finding that the inorganic chemist had to struggle with some of the difficulties that perpetually beset the organic chemist—in which reactions were greatly modified according to the masses of the bodies and the order in which they were brought together. *Primâ facie*, it appeared impossible that an excess of water could do any harm, and therefore it seemed natural to introduce the carbide into the presence of a large quantity of water. It was interesting to find that when the opposite course was followed, using generators of type No. 1, and introducing a comparatively small quantity of water into a large excess of carbide, one got, not acetylene, but a number of bodies produced by polymerisation. That was the ordinary experience of the organic chemist. Again, it was extremely interesting to learn that benzene was produced at so low a temperature as 700° C. He regarded that substance as the father of the substances produced by the distillation of coal. The great difficulty in working with carbide appeared to be that one of the products of reaction was insoluble. That was always a terrible difficulty to grapple with. The accumulation of a solid body such as lime or hydrate of lime around the remaining core of carbide led to the production of this smoke nuisance, and to the generation of benzene and other bodies, and even to such complications as tar. If he might make a suggestion to those dealing with these problems, it would be to introduce agitation. If the general conditions admitted of that, it might possibly lead to the obtainment of a purer product and a larger yield.

Mr. Otto **Hehner** questioned the conclusiveness of the benzene experiment shown by Prof. Lewes. No doubt benzene did cause the acetylene to smoke; but had the lecturer tried to remove benzene, if present, from improperly prepared acetylene, by, for instance, passing it through fluid paraffin, which would almost certainly dissolve the benzene, without interfering with the acetylene? If one considered that naphthalene could be added to ordinary coal-gas without producing a smoky flame, one should hardly expect that the addition of benzene would necessarily produce smoky flames. Prof. Lewes's explanation that a steatite burner, when once infected with benzene, was incurable, also appeared to him (Mr. Hehner) open to doubt. The burner, on getting hot, undoubtedly did become black throughout its mass, but, if the gas-holes remained unobstructed, there seemed to be no valid reason why the gas should continue to smoke. Probably Prof. Lewes had information on the subject which would explain the matter.

Mr. Eugene **Ackermann** pointed out that one of the chief desiderata in the successful use of acetylene was the purification of the gas evolved, and that could hardly be realised in the cheaper forms of apparatus now in the market. It would be far more economical, and lead to better results, and would avoid much of the danger attached to the use of acetylene, if people purchased well-made apparatus and paid more attention to keeping them clean and in good working order.

Mr. O. **Guttmann** mentioned that the Prussian Railways had now adopted a system invented by Pintsch, whereby acetylene-gas was mixed with oil-gas and yielded a perfectly safe illuminant according to the official tests made by the Prussian Government. It was probable that that mixture contained a quantity of benzene, and it would be interesting to know whether it had the effect of producing a smoky flame, and, if so, how such effect was avoided.

Mr. W. A. **Bone** desired to have some further information with respect to the decomposition of acetylene by heat. The author had stated that at a temperature of 800° to 1,000° acetylene was decomposed into its elements, and that the gas analysed by him contained: acetylene, 70; methane, 11; hydrogen, 19. He understood Prof. Lewes to say that the acetylene yielded first ethylene, and that this body, by splitting off carbon, yielded methane. He would like to know whether Prof. Lewes had actually found ethylene present in the gases produced by passing acetylene over a surface heated to 800° or 1,000°, and, if not, on what grounds he made the statement that methane resulted from acetylene through ethylene; and, secondly, whether he had proved by isolating it that the body thus found actually was methane.

Prof. Lewes, in reply, said that Mr. Redwood's kindly remarks left him practically nothing to answer, and therefore he would pass on at once to the remarks made by Mr. Worth, who pointed out that he had had long experience of generators—an experience naturally brought about by his connection with the manufacture of carbide; and he brought to light one or two very interesting points, and that which had interested him most was, perhaps, the statement that in the form of generator (class 3) in which the carbide was in excess, the yield from acetylene was not as good as in the other forms. There was no doubt that in certain generators there was overheating. At first sight one would say that it was practically impossible in a generator with excess of water to get a temperature higher than that of boiling water. In badly made generators, however, in which the carbide rested on the floor of the generator, the lime settling upon and around the decomposing carbide formed so good a non-conductor that he had seen tin melted in generators of this character. In that class of generator it was found important to keep the water constantly entering and leaving the apparatus. Where a false bottom was employed, and a bed of water was provided below and above, the amount of lime sludge settlement was equal to about 8 lb. per 1 lb. of carbide in the hour; but by regulating the flow of water through the generator, and dropping fresh carbide from time to time on to the false bottom, no overheating would result, and the quantity of acetylene obtained would come up nearly to the full yield, but it must not be forgotten that a small quantity would disappear with the change of water. A properly made generator of that class would, in his opinion, be the best obtainable. Mr. Butterfield made some admirable remarks, and gave some valuable data from Dr. Caro's paper on the subject. It was perfectly true that Prof. Caro found that in the dripping apparatus he got a temperature not exceeding 200° C., but he thought that was rather erroneous. Prof. Caro was such a good worker that one did not like to speak doubtingly of his results; but he used sticks of alloy, and when one had a mass of decomposing carbide the heat would be of a local character, and it was possible that the stick, which was a good conductor, might not be melted in such a way as to correctly register the temperature. There must be considerable space between the sticks, and the waves of heat might miss the sticks of alloys. There was a difficulty in registering the temperature even with the thermo-couple, because if the couple happened to be between two large pieces of carbide, the flaking of lime from their surfaces surrounded the thermo couple with a non-conducting material which prevented proper registration of the temperature, and it was only after a large number of experiments that one could get true results. Mr. Butterfield also referred to the calcium phosphide in the experiment not giving off its phosphoretted hydrogen until after most of the carbide had decomposed, whereas it was sometimes found that the largest quantity was given off in the first rush of gas. That was a point which had caused him to speak guardedly in the paper, as he was not clear in his own mind that the phosphoretted hydrogen came from calcium phosphide. It would require much research before coming to a conclusion as to its source, or even that of some of the other impurities.

Prof. Clowes had asked about Prof. Pictet's researches on acetylene. Prof. Pictet, as the members were aware, made his well-known researches with regard to liquefying acetylene, and where he spoke of the explosion point being reached he was working from experiments on acetylene produced under pressure during its generation. Under those conditions a temperature of 800° C. might be attained, and the decomposition, instead of being local, would spread throughout the whole mass, and thus a bad explosion would be caused. At ordinary pressures the only condition in which an explosion could take place was when the temperature rose with great rapidity and air was present. The maximum temperature was not reached for at least eight minutes after the water was brought into contact with the carbide, and the rapidity with which the gas came off at first was so great that it would rinse the air out of the apparatus before an explosion could take place. The quantity of air needed to bring about an explosion was small, and so manufacturers had to make their air spaces very small. Mr. Friswell made some interesting remarks with regard to agitation. He thought they were agreed, however, that in generating acetylene gas the great trouble was that generation was too rapid, and agitation might rather increase that trouble, although it would be valuable if at stated intervals, when the lime had become too thick, a rocking tray could be introduced to clear the carbide. With regard to the formation of benzene, he imagined that the temperature at which benzene began to show itself was between 600° and 700° C. Coming to the question of the smoky burner, the point which was raised was that having a burner burning acetylene with a smokeless flame, was there any reason why, when benzene became mixed with the acetylene, that should not burn smokelessly also? It was merely a question of adapting the burner to the material to be burnt. A burner suitable for burning pure acetylene was unsuitable for use when benzene was present in the acetylene. On burning acetylene with benzene in it a smoky flame resulted; as soon as the benzene had gone off the smoke ceased and a pure acetylene flame was obtained. Mr. Ackermann spoke with regard to purification, which was an important feature. A certain amount of purification was obtained in the third class of generator, in which the lime water present washed the escaping gas, and most of the impurities remained behind. Although the phosphoretted hydrogen was not entirely removed it was reduced by the washing it received, and the more acetylene was purified the better it would be for ordinary combustion.

In regard to the use on the German State Railways of acetylene mixed with oil-gas, it was found that when 20 per cent. of acetylene was mixed with oil-gas and compressed to about 10 atmospheres, the illuminating value of the oil-gas was practically doubled, and that would open a considerable field for acetylene in this country. It would be found that nearly all the troubles connected with acetylene disappeared when the gas was burned in a diluted form. Oil-gas could be consumed from a burner with far larger apertures than pure acetylene, and it was the smallness of the apertures which provided the means of scrubbing out benzene. The last speaker was disturbed by the appearance of methane in the analysis. The work of Berthelot showed that at certain temperatures acetylene and nascent hydrogen combined to form ethylene, and if that were heated it would break down into methane and acetylene once more. The analysis given was made under ordinary conditions, so that it would be more correct to have spoken of "saturated hydrocarbons" rather than of methane, as ethane might have been present.

*Meeting held on Monday, June 20th*, 1898.

DR. R. MESSEL IN THE CHAIR.

## A NEW SYNTHESIS OF GUAIACOL.

BY WATSON SMITH.

(*This Journal*, 1898, 314—315.)

The Chairman read a letter, dated May 26th, from the Chemische Fabrik von Heyden of Radebeul, Saxony, calling attention to a statement made in discussion on the above paper, that the first specimen of synthetic guaiacol in crystalline form, shown at Chicago in 1893, was of French manufacture; whereas the specimen in question emanated from their works near Dresden, and they were the first to produce it.

## ALUMINIUM AS A HEATING AND REDUCING AGENT.

BY DR. HANS GOLDSCHMIDT AND CLAUDE VAUTIN.

(*See also* pp. 584 and 612.)

Although it is well known that the oxide of aluminium ($Al_2O_3$) is a remarkably stable body, no experimenters have hitherto taken advantage in a practical way of the enormous affinity exercised for oxygen by aluminium to effect reductions of other oxidised bodies. A few experiments on a laboratory scale, chiefly on the reduction of metallic chlorides, and fluorides, have been recorded, amongst which may be mentioned those of Wöhler, Tissier, and Deville. Wöhler attempted to produce the metals chromium and manganese by heating their chlorides with aluminium, but only partial success was obtained, as it was found impossible to obtain the metals free from aluminium. He also produced an alloy of titanium and aluminium by heating together titanic acid, cryolite, and metallic aluminium. He states that the use of cryolite is to cause the formation of fluoride of titanium, which shows that his object was to bring into action a fluoride of the metal and not to effect direct reduction of the titanic acid. The brothers Tissier heated together aluminium with oxide of manganese, but they state that no reaction takes place. Deville produced an alloy of aluminium and silicon by heating together the metal and silica, and Beketoff produced a substance containing 33 per cent. of barium by heating baryta with aluminium and barium chloride. Lastly, Greene and Wahl separated manganese from its oxide by direct reduction.

Although these results are not very encouraging it is a remarkable fact that the value of aluminium as a deoxidiser has been so long overlooked. It is probable that the comparatively high cost of the metal and the explosive nature of the mixtures deterred some from attempting to use it on a large scale.

The fact that metallic aluminium is a powerful reducing agent may be inferred from the figures representing the heats of combination of aluminium with oxygen, sulphur, &c. For example, the heat or formation of [$Al^2O^3$] is about 360,000 calories, while that of [$K^2O$] is 97,000. Again the heat of formation of [$Al^2Cl^6$] = 321,000 against [$Fe^2Cl^6$] = 192,000 and [Na Cl] = 97,300, but judging by results, even these figures failed to promote further research.

In the latter part of 1892 and early in 1893, one of the authors was engaged in investigating certain processes for the production of aluminium, and for special reasons a quantity of pure aluminium sulphide was required. Attempts were made to produce it by the usual text book methods, such as heating alumina in the vapour of carbon bisulphide, or heating together alumina, carbon, and sulphur, but these failed to produce the sulphide in sufficient quantity and of the required purity. It then occurred to the author to reduce sulphide of lead by means of metallic aluminium in imitation of the well-known reaction between galena and metallic iron. To carry out the experiment, about 5 lb. of practically pure galena were crushed and mixed with the theoretical quantity of aluminium in coarse lumps to produce $Al_2S_3$ and metallic lead in accordance with the equation—

$$3\,PbS + Al_2 = Al_2S_3 + 3Pb$$

The mixture was charged into a crucible, and placed in a furnace. As soon as the contents of the crucible had attained to a full red heat, reaction took place, with evolution of enormous heat. The contents of the crucible were cast into a mould, and it was found that the reaction as expressed above had taken place, yielding practically theoretical amounts of the products. The aluminium sulphide produced is of a greenish colour; it decomposes water readily with the production of sulphuretted hydrogen, though it may be kept in air-tight tins or bottles for an indefinite time. It fuses at a clear red heat, and the metal can easily be obtained from the fused mass by electrolysis. The presence of sulphide of lead as an impurity is guarded against by keeping a slight excess of aluminium in the charge, which excess of metal will be found after casting the molten product as a hard, brittle, and easily detachable button on the surface of the lead. Aluminium will not alloy with lead, but it will form alloys with iron, silicon, copper, silver, gold, &c. The excess of metallic aluminium therefore performs the double function of keeping the sulphide of aluminium pure, and of removing all iron, copper, &c., from the lead, giving to the latter remarkable purity and softness. Further, by judiciously mixing the aluminium and the galena, this excess button removes any precious metals which may be contained in the sulphide of lead, much in the same manner as zinc desilverizes lead in the Parkes process.* The concentration of the precious metals in the excess button presents only a theoretical interest, as the present price of aluminium entirely excludes its use for that purpose. This first successful experiment led to further attempts at reducing the sulphides of other metals in spite of the frequently repeated statement that aluminium sulphide could be reduced by metallic iron, zinc, copper, &c. The supposed decomposition of aluminium sulphide by metallic iron has been the basis of numerous patents for the production of aluminium. The patentees describe at length a process for producing the sulphide, after which the specification draws to a conclusion with the statement that the sulphide may be decomposed by iron, copper, or even by lead. As a matter of fact, it is the reverse action which takes place. Nearly all metallic sulphides, except those of the sodium and barium groups and magnesium, are readily decomposed by metallic aluminium with the characteristic feature of the evolution of great heat. So much so that on reducing common sulphide of iron in a small crucible, the molten iron, free from carbon, melts into one mass which can be cast into a mould, while the temperature of the furnace, in which the reaction took place, is some hundreds of degrees below the melting point of the metal. It is interesting to note that iron produced in this manner, using a magnesia-lined clay crucible, is obtained absolutely free from carbon, the only impurities being traces of aluminium and sulphur, and by using a small excess of the sulphide the aluminium may be eliminated. Among the sulphides that are decomposed by aluminium may be mentioned those of cobalt, nickel, molybdenum, and zinc.

Our next experiments were carried out with metallic oxides and aluminium; here, the range of possible reductions extends to the oxides of the alkalis and the alkaline earth metals, all of which yield up their oxygen, though with difficulty, to aluminium. Practically all the metallic oxides are reducible by this method, the chief exception being the oxide of magnesium. Trials have been made with the following oxides of metals, all of which are reduced by aluminium, either pure or in the form of alloys:—Chromium, manganese, iron, copper, titanium, boron, tungsten, molybdenum, nickel, cobalt, zirconium, vanadium, niobium, tantalum, cerium, thorium, barium, calcium, sodium, potassium, lead, and tin; however, the experiments with some of these metals must not be considered to be concluded.

The failure of the brothers Tissier to effect reduction of the oxide of manganese is probably due to one of two causes; either they failed to heat the charge sufficiently to promote reaction, which is improbable, as the temperature required is not high, or else they had not the aluminium in a form best suited for the purpose. We find that in nearly every case the aluminium must be in a powdered or granulated condition, and intimately mixed with the finely crushed oxide or sulphide to be reduced. There are, however, a few exceptions to this rule, for example, to effect the reduction of lead salts, it is not advisable to have the aluminium in a very fine state of division, because owing to the enormous heat produced a simultaneous reaction throughout the whole charge would cause sudden vaporisation of the lead, with the result that the whole charge would be blown out of the crucible with explosive violence.

We have found that aluminium, like lead and other metals, can be easily granulated by agitating the molten metal in a suitable vessel at the moment when on cooling it reaches a certain critical temperature just between the solid and liquid state.

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* The addition of a small percentage of aluminium to the zinc used in the Parkes process permits of a higher temperature being used; it prevents the formation of oxide of zinc and effects better separation of the precious metals.

As before mentioned, the most striking feature of all these re-actions is the evolution of enormous heat. So great is the temperature produced that the most infusible metals are melted, while the slag, consisting of nothing but alumina, is sufficiently molten to allow all the reduced metal to sink through it and to collect into one fused mass. It is estimated that the temperature produced is at least 3,000° C, and by means of this heat, work can be done which hitherto required the electrical furnace for its performance.

The authors have made practical use of the reducing power of aluminium in the production of chromium from its oxide on the large scale, but they soon recognised the fact that to make this process commercially successful, it would be necessary to do away with external heating of the charge, both on account of high fuel costs and on account of crucible wear.

It has been proved by experiment that it is unnecessary to heat the whole of the mixture from which the metal is to be obtained to the temperature at which reaction takes place; that is to say, it is only necessary to start the reaction at one point of a given charge as the local heat produced is sufficient to cause the whole charge to re-act, the heat passing gradually through the entire mass. In the reduction of chromic oxide by aluminium the temperature to which it is necessary to heat one point of the charge before the reaction takes place is about 1,050° C, or a bright red heat.

At first, however, it was found very difficult to cause the mixture to ignite at one point without external heating, but this trouble was overcome by using a "fuse" of a highly inflammable nature which was capable of setting up an initial impulse which subsequently caused the combustion of the whole charge. The action of the "fuse" or "cartridge" is also based on the heat produced by oxidising aluminium. By supplying the metal with a compound rich in oxygen a highly inflammable mixture is obtained, which at the same time produces intense heat. Of the various compounds tried, peroxide of barium was found to give the best results though various other per-salts may be used. A suitable mixture of the metal with the peroxide is made up into a paste with a cementing material, which on hardening forms a cartridge, capable on ignition, of imparting the requisite initial temperature to the charge. The cartridge is supplied with a piece of magnesium ribbon whereby its ignition is readily accomplished. By this process a considerable amount of chromium has already been produced by the authors.

A few experiments illustrating the heating effects of this process will be shown, and it is probable that, using cheap impure aluminium and a cheap source of oxygen such as oxide of iron, that the process as a heat producer possesses considerable commercial value as it can be supplied in cases where it would not be convenient to use other sources of heat. For heating purposes alone, it would be necessary to add some material such as magnesia or lime to the mixture of metal and oxide so as to prevent the running together of the metal and slag produced; thus leaving, after firing, a brittle cindery slag which can easily be removed by tapping with a hammer.

For heating purposes, as in the production of metals, the cartridge would have to be used to start the reaction in the mixture which is to produce the heat.

As we have already pointed out, a very important use to which the process may be put is for the production of pure carbon-free metals, though future work will probably lead to other important applications; nor must it be forgotten that by this method not only pure metals but also alloys can be obtained, many of which it is exceedingly difficult to produce by any other process. For example, the authors have produced ferro-boron containing 20 to 25 per cent. boron; ferro-titanium (20 per cent. and 40 per cent. of titanium, the latter being non-magnetic); ferro-chrome; chrome-manganese, &c. But we hope to show by means of experiments that the adaptation of this method to heating purposes possesses very wide application, and it is possible that in point of utility it will rival the reduction process.

It may be mentioned that the alumina slag produced possesses properties superior to ordinary rock corundum both as to hardness and texture, owing probably to the total absence of water in its composition. The slag also possesses other interesting characteristics. Thus for example, the slag formed during the production of chromium will be found to be interspersed with red crystals which must be taken to be rubies, chromium being the colouring matter of the natural ruby. They are, however, of no value on account of their small size.

Finally, phosphates, sulphates and nitrates are also reduced by aluminium, though the latter act feebly in comparison with the sulphates.

The authors wish to acknowledge the useful work done by Mr. Hugh K. Picard, who, as assistant to one of them, successfully carried out most of the earlier experiments.

## APPENDIX.

The following experiments will serve to illustrate the paper:—

1. Burning of the cartridge, the so called "inflammable-cherry."

2. Heating of an iron rivet of ½ lb. weight by means of a cemented heating mixture, which surrounds the rivet so that the gradual passing of the heat is visible.

3. White heating of a rivet of about 7 lb. weight, the heating mixture being surrounded with sand. As the heat is kept from dissipating by the sand, the whole mass can be put, as shown, into a wooden tub.

4. Application of the method for hard-soldering a flange on to a tube of 1 in. diameter; instead of putting the flange (prepared with hard solder and borax as the coppersmiths do it) into a charcoal fire, it is placed in the heating mixture. For this purpose only about 3½ oz. of aluminium are required. If impure or raw aluminium be used at, say, 6*d.* per pound, the total expense will be 1½*d.* for the heating mixture.

5. Production of wrought iron from oxide of iron and aluminium. The reaction in this case is brought about without the aid of a cartridge, but by means of a mixture of peroxide of sodium ($NaO_2$) and carbide of calcium ($CaC_2$), ignited by a drop of water. The same effect is produced by a mixture of peroxide of sodium with aluminium, magnesium, zinc, or sulphide of antimony.

6. Production of a regulus of metallic chromium of about 10—12 lb. weight in a magnesia-lined crucible. In this crucible a mixture of $Cr_2O_3$ and $Al_2$ is put, and ignited; then a further quantity of this mixture is added till the crucible is filled up. The reaction lasts only a few minutes.

7. Production of sodium (Na) from caustic soda (NaOH) and aluminium in a paper tube. The sodium distils off with a bright flame. The initial temperature of the reaction is very low; this experiment is therefore interesting, as aluminium, as is well known, has been made with sodium from chloride of aluminium ($Al_2Cl_6$).

Beketoff found in 1888 that aluminium decomposes the hydroxides of the alkalis, and he distilled potassium and rubidium in an iron tube. Deville, however, expressly mentioned that he did not succeed in decomposing the molten alkalis with aluminium.

8. Reaction between a mixture of gypsum ($CaSO_4$) and aluminium in a paper tube; the temperature of reaction is here very high. First the paper tube is set on fire by a match, but the temperature so produced not being high enough to start the reaction, a cartridge is applied, and then the mixture goes off like a rocket.

The following metals, alloys, &c. were exhibited:—

### *Metals.*

1. Chromium, containing 95—97 per cent. Cr and 3—4 per cent. Fe.
2. Chromium, 99·5—99·8 per cent. Cr, containing also traces of silicon and iron.
3. Manganese, 98—99 per cent. Mn.
4. Iron, hammered, equal to best pure steel.
5. Vanadium, chemically pure.
6. Niobium, chemically pure.

*Alloys.*

7. Iron, containing 40 per cent. of titanium (not magnetic).

8. Iron, containing 20 per cent. of titanium (magnetic).

9. Iron, containing 25 per cent. of boron.

10. Manganese, containing 30 per cent. of chromium for making alloys with copper, manganese, and chromium (so-called chrom-manganin).

11. Copper, containing 10 per cent. of chromium (has nearly the colour of copper).

12. Alloys of lead and barium.

13. A plate of iron of about half an inch in thickness in which a hole has been melted by the heating mixture.

14. Different pieces of corundum (slag, fused $Al_2O_3$); some of these pieces containing crystals of rubies; also corundum crushed.

15. A piece of bronze.

Breaking strain in lb. per sq. in. = 27·874 tons, or 62,440 lb.

Elongation = 34 per cent.

# Manchester Section.

*Meeting held on Friday, April 1st, 1898.*

MR. GEORGE E. DAVIS IN THE CHAIR.

## ON THE COMPARATIVE EFFECT OF LIME AND OTHER CHEMICALS UPON SEWAGE.

BY J. CARTER BELL, A.R.S.M.

The following are the results of many months' experiments, carried out under similar conditions as to quantities, time, and temperature. In the first instance experiments were carried out in the laboratory upon 700 c.c. of settled sewage, this quantity being convenient as it represented 10 gallons. From the following table it will be seen that other chemicals were tried in comparison with lime, in order to find out the best precipitant, and one which gave the least amount of sludge. Antinonnin, mercuric chloride, and acetate of lead were used, merely from scientific curiosity, and these expensive chemicals did not produce such good results as the cheaper lime.

TABLE 1.

*The Comparative Action of Various Chemicals upon the same Sample of Clear or Settled Sewage.*

| —— | Free Ammonia. | Albuminoid Ammonia. | Appearance. | Per Cent. of Purification. | Oxygen required for 4 Hours. | Per Cent. of Purification. |
|---|---|---|---|---|---|---|
| | Grs. per Gall. | Grs. per Gall. | | | Grs. per Gall. | |
| Settled sewage | 2·2 | 0·76 | Black | .. | 5·6 | .. |
| „ filtered through paper | 2·2 | 0·48 | Opalescent | 37 | 4·2 | 25 |
| Chloride of iron filtered through paper | 2·0 | 0·16 | Clear | 79 | 2·4 | 57 |
| Antinonnin | 2·0 | 0·16 | „ | 79 | 3·2 | 43 |
| Hydrogen peroxide | 2·0 | 0·44 | Opalescent | 42 | 1·5 | 73 |
| Mercuric chloride | 2·0 | 0·76 | Very cloudy | Nil | 5·6 | Nil |
| Chlorine | 1·6 | 0·32 | Cloudy | 57 | 3·1 | 44 |
| Sodium peroxide | 2·0 | 0·68 | „ | 10 | 5·0 | 10 |
| HCl and $MnO_2$ | 0·12 | 0·08 | Clear | 89 | 2·1 | 62 |
| $FeSO_4$ and lime | 2·3 | 0·18 | „ | 76 | 1·8 | 67 |
| HgCl and lime | 1·2 | 0·04 | „ | 94 | 0·7 | 87 |
| $FeSO_4$ | 2·0 | 0·76 | Cloudy | Nil | 5·3 | 5 |
| Perchloride of iron and lime | 2·0 | 0·24 | Clear | 68 | 2·0 | 64 |
| Carbolic acid | 2·2 | 0·76 | Black | Nil | 20·5 | Nil |

In the cases of powerful antiseptics, such as mercuric chloride, hydrogen peroxide, &c., no physical change was apparent: the sewage was sterile, but very unsightly. This perhaps would not matter, if all works in the watershed produced sterile effluents; but it is undoubtedly pleasant to look upon a bright and sparkling effluent.

Turning to the cheaper chemicals in this table, it will be observed that there is very little difference in the percentages of purification.

Lime has a bad name, but that arises from not knowing how to use it. People think that if an effluent shows an alkalinity of one or two grains to the gallon, enough lime has been used, which is a mistake. It is natural to think that when you have excess of lime present, all that can be precipitated by lime is thrown down. The lime does not follow the usual rule. In the following table this is plainly seen.

The following five experiments were made upon the same sample of settled sewage, using lime as the precipitant.

The lime was used in the proportion of 10, 15, 20, 25, and 30 cwt. to the million gallons of sewage.

With 10 cwt. per million gallons, giving 3 grains of lime in excess, very little purification took place, the effluent looking very much like the original sewage; and even 15 cwt. did not give a brilliant effluent. It was not until 20 cwt. was reached that any satisfactory purification took place, and in this case an increased quantity of lime failed to effect any further improvement in albuminoid

TABLE 2.

[All results are in grains per gallon.]

| —— | Sewage. | 10 Cwt. | 15 Cwt. | 20 Cwt. | 25 Cwt. | 30 Cwt. |
|---|---|---|---|---|---|---|
| Appearance | Black | Very like sewage. | Cloudy | Clear | Clear | Clear |
| Free ammonia | 1·6 | 1·2 | 1·0 | 1·0 | 1·0 | 1·0 |
| Albuminoid ammonia | 0·64 | 0·56 | 0·36 | 0·28 | 0·28 | 0·22 |
| Per cent. of purification | | 12·5 | 43·7 | 56·2 | 56·2 | 56·2 |
| Oxygen for 4 hours | 5·1 | 4·2 | 3·4 | 2·4 | 2·3 | 2·3 |
| Free lime in effluent | .. | 3·0 | 4·5 | 5·5 | 7·0 | 8·0 |
| Total dry sludge | .. | 21·7 | 30·5 | 39·0 | 41·0 | 41·0 |
| Tons to million gallons, containing 10 per cent. of total solids | .. | 13 tons 16 cwt. | 19 tons 9 cwt. | 24 tons 17 cwt. | 26 tons 2 cwt. | 26 tons 2 cwt. |
| Mineral matter | .. | 18·7 | 24·0 | 30·2 | 31·2 | 32·0 |
| Organic matter | .. | 3·0 | 6·5 | 8·8 | 10·1 | 9·0 |
| Lime in sludge | .. | 11·2 | 15·0 | 18·0 | 18·0 | 19·0 |

ammonia, and absorbed oxygen. A small quantity of lime added combines with the dissolved carbonic acid, and is thus lost, and can have no effect upon the dissolved organic matter.

The lime may vary in quality, as the following sample will show:—

| — | No. 1. | No. 2. | No. 3. | No. 4. |
|---|---|---|---|---|
| Lime | 92·55 | 98·14 | 85·21 | 88·64 |
| Carbonate of Lime | 7·12 | 1·21 | 12 62 | 10·22 |
| Silica, Alumina, and Iron | 0·24 | 0·32 | 0·18 | 0·23 |
| Water | 0·01 | 0·33 | 1·99 | 0·91 |

No. 3 sample, badly slaked, would give very poor results. According to some authorities, the strength of the solution varies according as more or less water has been used to slake the lime. I do not agree altogether with the above, but the amount of water used for slaking has a considerable influence upon the resulting milk of lime. It was the custom formerly, at Salford, to dry-slake the lime, and then to sift it and mix with water; a dangerous method for the workmen, who had to work with cloths tied over their mouths in an atmosphere of lime dust. I advised the Committee to adopt wet slaking of the lime, with the result of producing a much better milk of lime. I have found that 1 part of lime to 4 parts of water disintegrates the lime into an impalpable powder. Four experiments were performed with 1 part of lime to 1, 2, 3, and 4 parts of water. The first was in the granular condition; second partially so; and three and four were in an impalpable powder. The four were made up to the same bulk with water allowed to settle for 15 minutes and 12 hours.

| — | 15 minutes. | 12 hours. |
|---|---|---|
| | Grains per Gallon. | Grains per Gallon. |
| No. 1 contained | 106·4 | 93 |
| " 2 " | 105·0 | 93 |
| " 3 " | 103·6 | 93 |
| " 4 " | 102·2 | 93 |

Given a slight excess of lime, 1 c.c. of the lime water will contain from ·00125 to ·0013 grammes of lime, or about 90 grains to the gallon. But it does not follow if one weighs out the quantity of lime which is equivalent to the 90 grains that one will have a water equal to that strength.

The objectors to lime as a sewage purifier say that it dissolves organic matter, and that the resulting effluent contains more organic matter than the original sewage. Now, if you compare the analyses of the effluents which I have placed before you—the 20 cwt., 25 cwt., and 30 cwt. of lime to the million gallons—the latter ought to contain considerably more organic matter than the former; but they are all much the same in regard to albuminoid ammonia and the oxygen test, though the lime rises from 5 to 7 and 8 grains to the gallon. The amount of sludge precipitated also rises from about 14 tons to 26 tons to the million gallons, the difference being between 10 cwt. of lime and 30 cwt. In each case 150 or more million gallons were used. In the first experiment 20 cwt. of lime were used, and over 4,000 tons of sludge were produced; in the second experiment, where no lime was used, only about 1,000 tons of sludge were precipitated in the tank. The difference of 3,000 tons must be due to the purifying action of the lime, therefore it seems absurd to say that lime has no purifying action.

Laboratory experiments showed that sulphate of alumina or persulphate of iron were the best to use as regards the amount of sludge formed, for in these cases only a little over 4 tons were produced; whereas in the cases of lime and baryta the quantities were 25 and 30 tons respectively. This means a cost for removal of sludge of say, 1s. a ton, though it does not cost quite so much. The alumina treatment, according to these experiments, would cost about 30s. the million gallons, whereas the lime would be over 40s. As a result of these laboratory experiments I advised the Committee to use sulphate of alumina.

When this experiment was carried out with 167 million gallons, which were flowing for 14 days, and to each million gallons were added 18 cwt. of sulphate of alumina, the quantity of sludge formed was 4,923 tons. Samples of sewage and effluent were taken every hour for the 14 days, and at the end of each day the samples were mixed so as to obtain the average for the day. The daily sample was analysed, and at the end of the 14 days the average was taken of the albuminoid ammonia and the oxygen required for 4 hours.

Table III.

*Experiments on Settled Sewage to Determine the best Chemical Sewage Precipitant and that giving the least Sludge.*

[All results are in grains per gallon.]

| Quantity for One Million Gallons. | Free $NH_3$. | Albuminoid Ammonia. | Percentage of Purification. | Oxygen, 4 Hours. | Lime. | Precipitated Sludge. | Mineral. | Organic. | Tons of Sludge to Million Gallons containing 90 per Cent. of Water. |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Tons. cwt. |
| 10 cwt. lime, 15 cwt. copperas. | 1·2 | 0·14 | 50 | 0·95 | Nil | 28·3 | 23 1 | 5·2 | 17 12 |
| 25 cwt. lime | 1·2 | 0·11 | 60 | 0·81 | 3 | 41·1 | 34 7 | 6·4 | 25 12 |
| 15 cwt. alumina | 1·5 | 0·11 | 60 | 0·89 | Nil | 7·5 | 3·4 | 4·1 | 4 12 |
| 5 cwt. lime, 15 cwt. alumina | 1·5 | 0·11 | 60 | 0·98 | Nil | 7·3 | 4·1 | 3·2 | 4 10 |
| 25 cwt. Wardle persulphate of iron. | 1 5 | 0·11 | 60 | 0·70 | Nil | 8·1 | 4·0 | 4·1 | 5 0 |
| 25 cwt. barium | 1·5 | 0·11 | 60 | 0·95 | BaO 6 | 47·3 | 42·8 | 4 5 | 30 0 |
| 25 cwt. lead A | 1·6 | 0·11 | 60 | 1·0 | Nil | 26·4 | 20·0 | 6·4 | 16 10 |
| 25 cwt. mercuric chloride | 1·6 | 0·28 | Nil | 2·5 | Nil | Nil | Nil | Nil | Nil |
| 25 cwt. chloride of lime | 1·7 | 0·29 | Nil | 2·6 | Nil | Nil | Nil | Nil | Nil |
| 25 cwt. carbolic acid | 2·0 | 0·31 | Nil | 2·8 | Nil | Nil | Nil | Nil | Nil |
| 25 cwt. sulphurous acid | 1·6 | 0·28 | Nil | 6·5 | Nil | Nil | Nil | Nil | Nil |

| | Grains per gallon. |
|---|---|
| Average albuminoid ammonia in sewage | 0·33 |
| " " " effluent | 0·22 |

Percentage of purification, 33.

| | |
|---|---|
| Average oxygen required for four hours' sewage | 3·1 |
| " " " effluent | 2·1 |

Percentage of purification, 32.

| — | Grains per Gallon. | |
|---|---|---|
| | Sewage. | Effluent. |
| The highest amount of albuminoid ammonia in the sewage and the effluent during the 14 days was. | 0·50 | 0 28 |
| The lowest was | 0·22 | 0·17 |
| Highest oxygen required for four hours. | 4·1 | 3·0 |
| Lowest oxygen required for four hours. | 1·7 | 1 2 |

At the commencement of this experiment six clean tanks were prepared, holding about two and a half million gallons. These tanks were nearly 700 ft. long, about 8 ft. wide, and varied in depth from 5 to 8 ft. The treated sewage ran into No. 1, then into No. 2, and so on to No. 6, whence it flowed into the Ship Canal.

It will be interesting to note the quantity of sludge in each tank at the end of the 14 days, and the amount of solid matter in the sludge, mineral and organic.

| Tank. | Tons of Sludge. | Total Solid Matter at 212° F. | Mineral. | Tons of Dry Solid Matter. |
|---|---|---|---|---|
| No. 1 | 827 | 8·5 | 4·1 | 70·3 |
| „ 2 | 1,506 | 4·3 | 1·8 | 64·7 |
| „ 3 | 1,496 | 3·4 | 1·3 | 50·8 |
| „ 4 | 816 | 3·1 | 1·2 | 25·3 |
| „ 5 | 178 | 4·5 | 1·2 | 8·0 |
| „ 6 | 100 | 3·2 | 1·2 | 3·2 |

The average amount of total solids in this sludge was 4·5 per cent. This alumina sludge was allowed to stand at rest for 12 days, so that some of the water might separate.

| | | | |
|---|---|---|---|
| 1st day.... | 8 volumes of water. | 7th day.. | 20 volumes of water. |
| 2nd „ .... | 12 „ „ | 8th „ .. | 21 „ „ |
| 3rd „ .... | 14 „ „ | 9th „ .. | 22 „ „ |
| 4th „ .... | 16 „ „ | 10th „ .. | 23 „ „ |
| 5th „ .... | 18 „ „ | 11th „ .. | 23 „ „ |
| 6th „ .... | 19 „ „ | 12th „ .. | 23 „ „ |

This keeping of the sludge would raise the total solids to a little over 5·5 per cent.

Next 179 million gallons of sewage were treated with sulphate of alumina and lime in the proportions of 9 cwt. 22 lb. alumina sulphate and 8 cwt. of lime to the million gallons.

| | Grains per Gallon. |
|---|---|
| Average albuminoid ammonia in sewage ............ | 0·326 |
| „ „ „ effluent............ | 0·245 |
| Percentage of purification, 25. | |
| Average oxygen required for four hours' sewage ..... | 2·9 |
| „ „ „ effluent .... | 1·9 |
| Percentage of purification, 34. | |

*Sludge formed in the Six Tanks.*

| Tank. | Tons of Sludge. | Total Solid Matters at 212° F. | Mineral Matter. | Organic. | Tons of Dry Sludge. |
|---|---|---|---|---|---|
| No. 1 | 1,254 | 18·1 | 10·2 | 7·9 | 227 |
| „ 2 | 1,534 | 14·5 | 7·4 | 7·1 | 222·5 |
| „ 3 | 1,449 | 8·7 | 4·1 | 3·6 | 126 |
| „ 4 | 116 | 8·5 | 4·6 | 3·9 | 9·85 |
| „ 5 | 67 | 10·7 | 4·3 | 6·4 | 7·17 |
| „ 6 | 34 | 21·0 | 11·4 | 9·6 | 7·14 |

The sludge here was much higher in total solids than in any other experiment, due to the heavy rainfall (2·02 in.) during the 14 days.

As this experiment was not wholly satisfactory, owing to the large quantity of solid matter washed into the tanks, it was decided to try another 14 days' test, with sulphate of alumina and lime in about the same proportions:—

| | Grains per Gallon. |
|---|---|
| Average albuminoid ammonia in sewage ............ | 0·357 |
| „ „ „ effluent............ | 0·245 |
| Percentage of purification, 31. | |
| Average oxygen required for four hours' sewage ..... | 2·6 |
| „ „ „ „ effluent .... | 1·7 |
| Percentage of purification, 34. | |

These two experiments are remarkably close as regards the purity of the effluents. Taking the oxygen percentage purification the numbers are identical, and the albuminoid ammonia the same in both experiments.

But the amount of sludge in the tanks was widely different. Instead of 4,454 tons there was now 3,028 tons. The rain for this fortnight was only 0·506 in., against 2·019 in.

*Quantity of Sludge in Tanks from Alumina and Lime Effluent.*

| Tank. | Tons of Sludge. | Total Solids at 212° F. | Mineral. |
|---|---|---|---|
| 1 | 806 | 11·3 | 6·4 |
| 2 | 1,229 | 9·1 | 5·4 |
| 3 | 504 | 8·5 | 4·5 |
| 4 | 197 | 7·0 | 3·6 |
| 5 | 128 | 8·0 | 4·3 |
| 6 | 164 | 8·0 | 3·9 |

The quantity of sewage purified during the 14 days was 168 million gallons, which deposited 3,028 tons, or 18 tons to the million gallons.

A 14 days' experiment was tried with sulphate of iron and lime. The quantity of sewage treated was 166 million gallons. For each million gallons was used 14 cwt. of sulphate of iron and 7 cwt. 1 qr. of lime.

The quantity of sludge formed in the tanks was 4,927 tons

| Tank. | Tons of Sludge. | Total Solid Matter at 212° F. | Mineral. |
|---|---|---|---|
| 1 | 1,095 | 9·5 | 4·7 |
| 2 | 1,538 | 4·4 | 2·4 |
| 3 | 636 | 4·2 | 2·0 |
| 4 | 824 | 4·2 | 2·1 |
| 5 | 518 | 6·1 | 2·8 |
| 6 | 316 | 6·7 | 3·0 |

After 16 days' settling, the average sludge gave 41 volumes of water. The analysis of this water was:—

| | Grains per Gallon. |
|---|---|
| Free ammonia ........................ | 6·7 |
| Albuminoid ammonia .................. | 4·7 |
| Oxygen required for four hours........ | 10·2 |

The highest albuminoid ammonia in grains to gallons of the sewage was 0·39, and the lowest was 0·25. The average of the 14 days was 0·30.

Highest albuminoid ammonia in effluent was 0·30. The lowest was 0·22. The average of the 14 days was 0·27.

This is rather a poor result, as the purification is only a little over 9 per cent.

The oxygen purification gives a better result.

The average oxygen number in grains per gallon for the settled sewage was 3·4, and in the effluent 2·3, or a percentage purification of 32.

A 14 days' experiment was tried with chloride of lime or bleaching powder, using 1 cwt. per million gallons. This quantity was too small to precipitate any of the organic matter in solution, and the sludge which was formed in the tanks was simply due to the suspended matter. The quantity of sewage treated was 150 million gallons during the fortnight, and 1,120 tons of sludge were deposited in the tanks.

| Tanks. | Total Solids at 212° F. | Mineral Matter. | Organic Matter. |
|---|---|---|---|
| 1 | 13·4 | 6·8 | 6·6 |
| 2 | 13·7 | 7·4 | 6·3 |
| 3 | 12·3 | 6·6 | 5·7 |
| 4 | 12·8 | 7·4 | 5·4 |
| 5 | 11·8 | 6·6 | 5·2 |
| 6 | 11·1 | 6·2 | 3·9 |

This sludge on standing gave 26 vols. of water after seven days:—

| | |
|---|---|
| First day........................... | 13 volumes. |
| Second day.......................... | 18 „ |
| Third day .......................... | 22 „ |
| Fourth day.......................... | 23 „ |
| Fifth day........................... | 24 „ |
| Sixth day........................... | 25 „ |
| Seventh day......................... | 26 „ |

on the eighth day it decomposed and mixed with the supernatant liquid.

The above experiment was interesting, as it proved conclusively that lime has the power of precipitating organic matter from solution, for in no fortnightly experiment with lime was so small a quantity of sludge formed as the above.

Thus—

| | | | | | | |
|---|---|---|---|---|---|---|
| 168 | million gallons of sewage | with | 144 | tons of lime, gave | 4,742 | tons of sludge. |
| 162 | „ | „ | 150 | „ | 4,211 | „ |
| 150 | „ | „ | 145 | „ | 4,100 | „ |
| 160 | „ | „ | 153 | „ | 4,421 | „ |
| 150 | „ | „ | 154 | „ | 4,846 | „ |
| 179 | „ | „ | 121 | „ | 3,882 | „ |
| 173 | „ | „ | 109 | „ | 3,404 | „ |
| 145 | „ | „ | 135 | „ | 3,684 | „ |

In all my small and large experiments I have found that lime is able to precipitate 30 or 40 per cent. of the soluble organic impurity, which is as much as the other precipitants were able to do.

To produce a good clear effluent, there should be at least from 8 to 10 grains of lime in the gallon of effluent in excess. A small quantity of lime effects very little purification, as the following experiments will show:—

Two million gallons of sewage were treated daily for some days with from 30 cwt. to 40 cwt. of lime to the million gallons, and eight millions were treated with 10 cwt. of lime to the million gallons. The two effluents were allowed to flow away side by side down an incline plane, and the contrast was most remarkable: on one side there was a dark turbid stream, while on the other there was a bright clear effluent.

The following analyses show the effect of a free use of lime:—

| Lime, 10 cwt. to Million Gallons. | | Lime, 30 cwt. to Million Gallons. | |
|---|---|---|---|
| Albuminoid Ammonia. | Oxygen required for 4 Hours. | Albuminoid Ammonia. | Oxygen required for 4 Hours. |
| Grains to gallons. | Grains to gallon. | Grains to gallon. | Grains to gallon. |
| 0·22 | 3·2 | 0·11 | 1·4 |
| 0·25 | 3·4 | 0·14 | 1·5 |
| 0·28 | 3·8 | 0·12 | 1·3 |
| 0·21 | 2·8 | 0·09 | 1·2 |
| 0·23 | 3·0 | 0·11 | 1·4 |
| 0·29 | 3·6 | 0·12 | 1·4 |

The lime varied from 1 to 2 grains to the gallon in the 10 cwt. experiments, and from 8 to 12 grains in the 30 cwt. experiment.

Two million gallons of sewage were treated with about 30 cwt. of lime to the million gallons, and the effluent was allowed to flow into six tanks. It was allowed to rest there for four hours, at the end of that time sewage and effluents were analysed, with the following results:—

| — | Free Ammonia. | Albuminoid Ammonia. | Per Cent. of Purification. | Oxygen for 4 Hours. | Per Cent. of Purification. | Lime. |
|---|---|---|---|---|---|---|
| | Grains per Gallon. | | | | | |
| Sewage .... ... | 1·7 | 0·56 | .. | 3·9 | .. | Nil. |
| Effluent, No. 1 tank. | 1·1 | 0·25 | 55 | 1·8 | 54 | 12·3 |
| Effluent, No. 2 tank. | 1·2 | 0·25 | 55 | 1·8 | 53 | 10·3 |
| Effluent, No. 3 tank | 1·2 | 0·22 | 60 | 1·9 | 51 | 10·0 |
| Effluent, No. 4 tank. | 1·2 | 0·28 | 50 | 1·9 | 51 | 10·0 |
| Effluent, No. 5 tank. | 1·4 | 0·25 | 55 | 1·8 | 53 | 10·0 |
| Effluent, No. 6 tank. | 1·4 | 1·7 | 69 | 1·8 | 53 | 12·0 |

The following 12 samples of effluents were taken from 12 separate tanks after standing four hours. The sewage had been treated with about 25 cwt. of lime, and after the mixture had travelled along a channel of 700 ft. in length before running into the tanks. It seems that to obtain the best results a certain quantity of lime is necessary; all above that is so much waste. Thus, it will be seen from the following table, that the effluent which contains the most lime is not better than the one which contains half the quantity. The quantity of sewage treated in this experiment was five million gallons, and one would have expected that the quantity of lime would have been the same in each tank. The cause of this difference is the bad mixing of the lime with the sewage; this is one of the causes of the poor results sometimes obtained from lime precipitation:—

| — | Free Ammonia. | Albuminoid Ammonia. | Oxygen for 4 Hours. | Lime. | Appearance of Effluent. |
|---|---|---|---|---|---|
| | Grains to Gallon. | Grains to Gallon. | Grains in Gallon. | Grains in Gallon. | |
| Sewage ........ | 1·9 | 0·45 | 3·9 | .. | .. .. |
| Effluent No. 1 tank. | 1·8 | 0·30 | 2·2 | 4·6 | Clear; slight sediment. |
| No. 2 north tank | 1·5 | 0·39 | 2·5 | 4·9 | „ „ |
| „ 3 „ „ | 1·7 | 0·36 | 2·4 | 7·8 | „ „ |
| „ 4 „ „ | 1·5 | 0·33 | 2·2 | 7·8 | Clear; no sediment. |
| „ 5 „ „ | 1·5 | 0·33 | 2·1 | 9·8 | „ „ |
| „ 6 „ „ | 1·7 | 0·30 | 2·2 | 11·7 | Very clear. |
| No. 1 south tank | 1·7 | 0·25 | 1·9 | 5·8 | Clear. |
| „ 2 „ „ | 1·8 | 0·19 | 1·6 | 6·3 | „ |
| „ 3 „ „ | 1·8 | 0·30 | 2·0 | 6·3 | Clear; slight sediment. |
| „ 4 „ „ | 2·2 | 0·30 | 2·1 | 10·2 | Clear. |
| „ 5 „ „ | 2·2 | 0·33 | 2·5 | 4·9 | Rather cloudy. |
| „ 6 „ „ | 2·1 | 0·39 | 2·8 | 1·4 | Very cloudy, with sediment. |

Many managers think if the milk of lime is run into the inlet channel with the sewage that is quite sufficient to make a homogeneous mixture. This is not the case, for the lime will often be carried along in a certain current, and in all parts of the channel the lime will be in variable proportion. That this is so is shown by the following three experiments. Samples of the effluent were taken at six equidistant places along the channel, and the lime estimated in grains per gallon:—

| No. 1. Not Mixed. | | | No. 2. Well Mixed. | | | No. 3. Not Mixed. | | |
|---|---|---|---|---|---|---|---|---|
| 17·0 | 6·5 | 14·5 | 8·2 | 8·0 | 8·0 | 10·5 | 14·0 | 9·5 |
| 19·0 | 6·0 | 15·5 | 8·0 | 8·2 | 8·0 | 10·0 | 12·0 | 9·0 |
| 21·0 | 6·0 | 16·5 | 8·0 | 8·0 | 8·0 | 10·0 | 7·5 | 10·0 |
| 23·0 | 6·0 | 14·0 | 8·0 | 8·5 | 8·0 | 11·0 | 7·5 | 11·0 |
| 10·0 | 6·5 | 14·5 | 7·5 | 8·0 | 8·0 | 11·0 | 8·0 | 11·0 |
| 10·5 | 10·0 | 15·0 | 7·0 | 7·5 | 8·5 | 8·0 | 7·5 | 10·5 |

The three sets of figures in each experiment represent samples taken from the centre of the channel and the two sides. It is most important that the chemicals and sewage be thoroughly mixed before dilution, for if the latter takes place the lime fails to precipitate organic matter. The lime falling to the bottom of the channel is soon converted into carbonate of lime, and is thus lost. In the channel already spoken of, the precipitated lime was estimated in the sludge formed at six different places. No. 1 was point of inlet, and No. 6 was the point of outlet, the other numbers being equidistant between.

| | | | | | | |
|---|---|---|---|---|---|---|
| No. | 1 | dry sludge | contained | 12·6 | per cent. | of lime. |
| „ | 2 | „ | „ | 19·6 | „ | „ |
| „ | 3 | „ | „ | 16·8 | „ | „ |
| „ | 4 | „ | „ | 24·7 | „ | „ |
| „ | 5 | „ | „ | 24·7 | „ | „ |
| „ | 6 | „ | „ | 32·0 | „ | „ |

I have always advocated the use of lime water to prevent this great waste of lime; much better results can be obtained then by the use of solid lime.

The following experiments have been made on different days with lime and lime water, using the same sewage in both cases, and the same quantity of lime. The quantity of lime used has been 25 cwt. to the million gallons.

| Lime Water. | | | Solid Lime. | | |
|---|---|---|---|---|---|
| Albuminoid Ammonia. | Oxygen for 4 hours. | Lime. | Albuminoid Ammonia. | Oxygen for 4 hours. | Lime. |
| Grains to gallon. | Grains. | Grains. | Grains to gallon. | Grains. | Grains to gallon. |
| 0·17 | 1·5 | 3·5 | 0·25 | 2·6 | 4·0 |
| 0·14 | 1·4 | 4·4 | 0·30 | 2·3 | 6·2 |
| 0·14 | 1·6 | 3·9 | 0·28 | 2·4 | 3·2 |
| 0·22 | 1·9 | 3·0 | 0·28 | 2·8 | 4·2 |
| 0·22 | 1·4 | 8·8 | 0·25 | 2·6 | 9·2 |
| 0·14 | 1·2 | 9·3 | 0·19 | 2·3 | 7·8 |
| 0·14 | 1·5 | 6·8 | 0·21 | 2·4 | 5·5 |
| 0·20 | 1·8 | 4·0 | 0·25 | 2·6 | 3·5 |

In every experiment the lime water has given the best results, amounting sometimes to as much as 50 per cent. improvement. In all these precipitation processes there seems to be a number which the precipitant cannot attack. The albuminoid ammonia falls to about 0·14 in the gallon and there remains, and no addition of ordinary cheap chemical seems to disturb that number; as fortnightly experiments had been tried with sulphate of alumina, iron, &c., it became necessary for comparison sake to try similar experiments with varying quantities of lime; it will be sufficient if the minimum and maximum lime experiments are given in detail.

The number of gallons of sewage treated in this No. 1 lime experiment was 173 millions. Rainfall for the 14 days was 1·601 inches. Quantity of lime used to one million gallons was 12 cwt. 2 qrs.

The maximum albuminoid ammonia in the sewage in grains to gallon was 0·44.

The minimum albuminoid ammonia in the sewage in grains to gallon was 0·22.

The average for the fortnight was, for sewage, 0·34, and for the effluent 0·31.

This, practically speaking, gave no purification, only about 8 per cent.

The average oxygen required for four hours was, sewage 2·6 grains to gallon, and effluent 2·4 grains to gallon—only 7 per cent. of purification. The quantity of sludge formed in the tanks was 3,404 tons.

| —— | Tons. | Total Solids. | Mineral. |
|---|---|---|---|
| No. 1 tank contained.. | 786 | 11·6 | 7·2 |
| „ 2 „ „ .. | 1,204 | 9·1 | 6·0 |
| „ 3 „ „ .. | 373 | 13·6 | 8·6 |
| „ 4 „ „ .. | 412 | 11·6 | 7·3 |
| „ 5 „ „ .. | 330 | 13·6 | 6·7 |
| „ 6 „ „ .. | 299 | 12·8 | 6·1 |

The second lime experiment was about 20 cwt. to the million gallons; sewage treated, 162 million gallons; rainfall for the 14 days, 0·740 inch. Average albuminoid ammonia in settled sewage, 0·402 grains per gallon; effluent, 0·278; which gives a purification of 30 per cent.

The average oxygen required for four hours was, sewage 3·2 grains to gallons, and effluent 2·1 grains to gallon, or a purification of 34 per cent. The quantity of sludge formed in the tanks was 4,211 tons.

| —— | Tons. | Total Solids. | Mineral. |
|---|---|---|---|
| No. 1 tank contained.. | 518 | 10·8 | 7·2 |
| „ 2 „ „ .. | 1,523 | 9·3 | 5·9 |
| „ 3 „ „ .. | 1,103 | 7·2 | 3·9 |
| „ 4 „ „ .. | 671 | 5·9 | 3·8 |
| „ 5 „ „ .. | 224 | 8·2 | 3·9 |
| „ 6 „ „ .. | 172 | 8·9 | 5·3 |

The average sludge gave, after standing for one week, 23 volumes of water.

On comparing the lime effluents and percentages of purification with the effluents from the sulphate of alumina and sulphates of iron, very little difference is found when a sufficient quantity of lime is used. It is remarkable that the first five tests should work out so closely in regard to the oxygen purification test, for, practically speaking, it is nearly the same in each, 34 per cent.

TABLE 4.

| Sewage, in Millions of Gallons. | Rainfall. | Chemicals to One Million Gallons. | Total Quantity of Sludge in Tanks. | Total Quantity calculated to contain 10 per Cent. of Total Solids. | Quantity of Sludge to One Million Gallons, 10 per Cent. of Total Solids. | | Average Total Solids of Sludge in Tanks. | Average Albuminoid Ammonia in Effluent, in Grains to Gallon. | Percentage of Purification. | Average Oxygen required for Four Hours for Effluent, in Grains to Gallons. | Percentage of Purification. | Cost of Chemicals, including Removal of Sludge, per Million Gallons. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Tons. | Tons. | Tons. | Cwt. | Percent. | | | | | £ s. d. |
| (1) 179 (lime and alumina). | 2·019 | Lime, 8 cwt.; alumina sulphate, 9 cwt. 22 lb. | 4,454 | 5,996 | 33 | 10 | 14·5 | 0·245 | 25 | 1·9 | 34 | 2 13 6 |
| (2) 169 (lime and alumina). | 0·566 | Lime, 7 cwt. 3 qrs.; alumina sulphate, 10 cwt. 2 qrs. | 3,028 | 2,830 | 16 | 14 | 9·3 | 0·245 | 31 | 1·7 | 34 | 1 18 8 |
| (3) 167 (alumina alone). | 0·278 | Sulphate of alumina, 18 cwt. 1 qr. | 4,923 | 2,223 | 13 | 6 | 4·5 | 0·220 | 33 | 2·1 | 32 | 2 11 0 |
| (4) 166 (lime and iron sulphate). | 0·793 | Lime, 7 cwt. 1 qr.; iron sulphate, 14 cwt. | 4,927 | 2,857 | 17 | 3 | 5·8 | 0·286 | 9 | 2·3 | 32 | 2 0 6 |
| (5) 186 (iron and lime). | .. | Perchloride of iron, 1 ton; lime. 6 cwt. | 3,472 | 3,298 | 18 | 13 | 9·5 | 0·250 | 26 | 2·2 | 24 | 3 2 6 |
| (6) 7½ (electrical) | .. | Nil—electrical .. | 120 | 71 | 9 | 15 | 5·9 | 0·168 | 39 | 2·1 | 33 | 3 0 0 |
| (7) 6 (iron persulphate). | .. | Persulphate of iron, 1 ton. | 106 | 51 | 17 | 13 | 4·8 | 0·160 | 48 | 1·6 | 44 | 3 7 0 |
| (8) 150 (chloride of lime). | 1·197 | Chloride of lime, 1 cwt. | 1,120 | 1,478 | 9 | 17 | 13·2 | 0·422 | Nil | 3·6 | Nil | 0 18 0 |
| (9) 162 (lime) ... | 0·740 | Lime, 20 cwt. .... | 4,211 | 3,537 | 21 | 15 | 8·4 | 0·278 | 30 | 2·1 | 34 | 1 15 0 |
| (10) 168 (lime)... | 1·606 | Lime, 17 cwt. .... | 4,742 | 3,983 | 23 | 11 | 8·4 | 0·266 | 22 | 2·9 | 23 | 1 14 10 |
| (11) 160 (lime)... | 0·918 | Lime, 15 cwt. 2 qrs. | 3,965 | 3,885 | 24 | 9 | 9·8 | 0·325 | 7 | 1·8 | 22 | 1 14 9 |
| (12) 179 (lime)... | 0·722 | Lime, 13 cwt. 2 qrs. | 3,882 | 3,960 | 22 | 3 | 10·2 | 0·303 | 5 | 2·9 | 3 | 1 11 0 |
| (13) 173 (lime)... | 1·601 | Lime, 12 cwt. 2 qrs. | 3,404 | 3,812 | 22 | 2 | 11·2 | 0·315 | 8 | 2·4 | 7 | 1 10 4 |
| (14) 150 (lime)... | 0·517 | Lime, 18 cwt. .... | 4,080 | 3,182 | .. | | 7·8 | 0·248 | 25 | 2·2 | 26 | 1 1 0 |

For comparison sake I have included in the following table the electrical process, though the whole of the sewage was not purified by this process. This experiment was carried on for 72 days, and during this time 7¼ million gallons were treated. The weight of sludge was in round numbers 120 tons, or 16½ tons to the million gallons. This quantity includes the solid matter which was in the sewage in suspension, and which amounted to many tons. This being deducted leaves so many tons precipitated from the iron plates which were used as electrodes. This quantity was easily calculated. The number of plates was 2,176, each plate measured 2 ft. by 1 ft. 6 ins. by ½ in. The weight of these plates before the commencement of the experiment was 54 tons; at the close of the experiment they were taken out, dried, and weighed. The weight was 53 tons 7 cwt. 0·14 lbs. The plates were covered with hydrated oxide of iron; this was all cleaned off, and, after this, the plates weighed 52 tons 4 cwt. 2 qrs. 14 lbs., showing a loss of 1 ton 15 cwt. 1 qr. 24 lbs.; this gives a little over three grains to the gallon of sewage. The average albuminoid ammonia from 52 samples was 0·168 grain to gallon, and the percentage of purification has been 39. This is higher than any in the list, but on the other hand one must notice the cost, which is 3*l.* the million gallons, this being nearly the highest in price. The extra 5 per cent. of purification would hardly warrant the extra expenditure. This is a very neat and elegant process, and when joined to filtration the effluents are all that one would desire.

In one respect the sludge is not utterly useless, for consisting so largely as it does of oxide of iron, it has been made into paint by calcination. This was tested by a manufacturer of paints, who reported that it made a good oxide of iron paint, but not equal to the best brands upon the market.

The composition of the calcined sludge is: Silica, 30·95; ferric oxide, 47·80; alumina, 8·52; carbonate of lime, 9·12; phosphoric acid, magnesia, alkalies, 3·61.

Experiments were made upon sewage to which 10 cwt. of lime were added to the million gallons; it was then treated by the electric current; these experiments were not successful, the percentage of purification being only 27.

While on the subject of sludge, I think I cannot do better than introduce the analyses of the various sludges produced in the several experiments.

It will be noticed that the amount of total solid matter in the alumina and iron sludges is very low compared with the lime sludge, and one advantage with the lime sludge is that the water separates more readily than it does from an alumina sludge. This is very important when the sludge is sent to sea, as the object is to send as much total solids as possible, and not water. Sludge should be kept for a certain number of days, so that the excess water may separate. In the following table will be seen a list of sludges, giving the total solids, and the amount of water which separates after standing 24 hours.

TABLE 4A.

SLUDGE AS BEING PUMPED AT SEWAGE WORKS.

| Total Solids. | Total Moisture. | Per Cent. of Water Separating out after 24 Hours. |
|---|---|---|
| Per Cent. | Per Cent. | |
| 6·3 | 93·7 | 35·0 |
| 11·25 | 88·75 | 11·5 |
| 11·0 | 89·0 | 15·0 |
| 3·4 | 96·6 | 66·0 |
| 8·0 | 92·0 | 23·0 |
| 3·6 | 96·4 | 60·0 |
| 5·0 | 95·0 | 45·0 |
| 7·8 | 92·2 | 28·0 |
| 7·8 | 92·2 | 27·5 |
| 7·0 | 93·0 | 27·0 |
| 9·78 | 90·22 | 13·0 |
| 7·51 | 92·49 | 22·0 |
| 10·5 | 89·50 | 8·0 |
| 10·5 | 89·50 | 15·0 |
| 7·61 | 92·39 | 31·0 |
| 9·43 | 90·57 | 12·0 |
| 11·13 | 88·87 | 14·0 |
| 9·0 | 91·00 | 13·0 |
| 11·7 | 88·30 | 13·0 |

*Sludge as being Pumped at Sewage Works*—cont.

| Total Solids. | Total Moisture. | Per Cent. of Water Separating out after 24 Hours. |
|---|---|---|
| Per Cent. | Per Cent. | |
| 13·6 | 86·40 | 2·0 |
| 15·7 | 84·30 | Nil |
| 12·8 | 87·20 | 2·0 |
| 8·8 | 91·20 | 12·0 |
| 7·4 | 92·60 | 7·0 |
| 7·96 | 92·04 | 7·5 |
| 11·86 | 88·14 | 7·0 |

The average total solids of the sludge sent to sea for 400 journeys is 12·8 per cent.

*Analyses of Sludge as taken from Tanks.*

| — | Alumina. | Ferrous Sulphate. | Lime. |
|---|---|---|---|
| Total solids.......... | 4·5 | 5·8 | 11·2 |
| Water ................ | 95·5 | 94·2 | 88·8 |

| — | Bleaching Powder. | Electrical. | Ferric Chloride. |
|---|---|---|---|
| Total solids .......... | 12·5 | 5·9 | 9·5 |
| Water ................ | 87·5 | 94·1 | 90·5 |

*Analyses of Sludge dried at 212° F.*

| — | Alumina. | Ferrous Sulphate. | Lime. |
|---|---|---|---|
| Silica ................. | 20·93 | 17·91 | 13·37 |
| Oxide of iron.......... | 1·83 | 6·90 | 1·88 |
| Alumina .............. | 9·72 | 8·79 | 6·93 |
| Copper ............... | 0·05 | .. | .. |
| Lime ... ............. | 14·10 | 11·18 | 25·3 |
| Magnesia ............. | 0·20 | 0·86 | 1·74 |
| Phosphoric acid....... | 0·50 | 0·92 | 0·26 |
| Nitrogen.............. | 0·94 | 1·35 | 1·37 |

| — | Bleaching Powder. | Electrical. | Ferric Chloride. |
|---|---|---|---|
| Silica ................. | 23·3 | 30·95 | 15·85 |
| Oxide of iron.......... | 6·42 | 47·80 | 5·84 |
| Alumina.............. | 2·50 | .. | 7·12 |
| Copper ............... | .. | .. | .. |
| Lime ................. | 10·39 | .. | 15·61 |
| Magnesia ............. | 0·20 | .. | 1·56 |
| Phosphoric acid ...... | 1·75 | .. | 0·74 |
| Nitrogen ............. | 1·10 | .. | 1·25 |

The wet alumina sludge will contain about 0·5 per cent. of alumina, yet a patent has been taken out to treat this sludge with sulphuric acid to form sulphate of alumina.

The drawback to all precipitation processes is the uncertainty attending them. For some days you may have a good effluent, and then other days bad. The above table clearly proves that to obtain good results from lime not less than from 20 to 25 cwt. should be used, and that in the state of lime water.

Much better results could have been obtained than those shown in the above table if larger quantities of chemicals had been employed; this is well shown by the following series of experiments.

| Sulphate Alumina to Million Gallons. | Albuminoid Ammonia. | Percentage Purification. | Oxygen for 4 hours. | Percentage Purification. |
|---|---|---|---|---|
| | Grs. per gall. | | Grs. per gall. | |
| 5 cwt. ......... | 0·399 | 30·3 | 4·6 | 2·4 |
| 10 „ ......... | 0·260 | 40·2 | 3·8 | 7·3 |
| 15 „ ......... | 0·250 | 43·8 | 3·6 | 12·1 |
| 20 „ ......... | 0·238 | 46·5 | 2·8 | 31·7 |
| 30 „ ......... | 0·105 | 76·4 | 1·0 | 75·6 |
| 40 „ ......... | 0·100 | 77·5 | 0·94 | 77·0 |

Sulphate of aluminæ was added in the proportion of 5 up to 40 cwt. per million gallons of sewage, and it will be noticed how sensibly the percentage of purification rises.

The following experiments were performed to prove that lime was as good a purifier of sewage as sulphate of iron and lime.

The sulphate of iron effluent was made at the sewage works by using millions of gallons of sewage and adding iron sulphate, 15 cwt.; lime, 5 cwt., to million gallons. The lime effluent was made in the laboratory by adding lime water to the same sewage at the rate of one ton of lime to the million gallons.

| Albumenoid Ammonia in Grains per gallon. | | Percentage Purification. | |
|---|---|---|---|
| Sulphate of Iron Effluent. | Lime Effluent. | Sulphate of Iron Effluent. | Lime Effluent. |
| 0·40 | 0·24 | 16 | 50 |
| 0·40 | 0·24 | 37 | 62 |
| 0·40 | 0·38 | 16 | 20 |
| 0·36 | 0·24 | 42 | 62 |
| 0·40 | 0·20 | 35 | 67 |
| 0·44 | 0·36 | 31 | 43 |
| 0·36 | 0·28 | 23 | 41 |
| 0·52 | 0·32 | 7 | 43 |
| 0·36 | 0·20 | 35 | 64 |
| 0·40 | 0·20 | 28 | 64 |
| 0·64 | 0·32 | 20 | 60 |
| 0·40 | 0·24 | 16 | 50 |

This gives an average albuminoid ammonia of 0·42 for iron, and 0·26 for lime, the average percentage purification being for iron 25, and for lime 52. The amount of precipitate caused by lime in the settled sewage was as follows:—

*Precipitate produced by adding Lime to various samples of Settled Sewage collected on different days. Weight of Precipitate dried at 212° F., in Grains per Gallon.*

| Total Solid Matter. | Mineral. | Organic. | Per Cent. Mineral. | Grains of Alkalinity calculated as Lime in Sludge. |
|---|---|---|---|---|
| 46 | 32 | 14 | 69·5 | 18·5 |
| 68 | 40 | 22 | 67·6 | 18·1 |
| 60 | 39 | 21 | 65·0 | 17·5 |
| 45 | 21 | 24 | 46·6 | 12·8 |
| 78 | 51 | 27 | 65·3 | 18·3 |
| 63 | 45 | 18 | 71·4 | 19·1 |
| 73 | 46 | 27 | 63·0 | 18·5 |
| 64 | 40 | 24 | 62·5 | 17·8 |
| 60 | 34 | 26 | 56·6 | 15·2 |
| 48 | 33 | 15 | 68·7 | 19·1 |
| 67 | 45 | 22 | 67·1 | 17·8 |
| 61 | 40 | 21 | 65·5 | 18·0 |

There is a fair uniformity in the above figures, and taking the average of the 12 experiments there are 21 grains of organic matter precipitated from solution. The alkalinity of the ignited or calcined precipitate is remarkably uniform. This alkalinity is not solely due to lime, as there were only 16 grains of lime added to the gallon, which is about equal to a ton to the million, and as the effluent always contained from 3 to 4 grains of free lime, which, together with the above, makes considerably over 20 grains calculated as lime.

The following five experiments will show the amount of lime added to one gallon of the same settled sewage, grains of lime in effluent, and grains of alkalinity calculated as lime in precipitate.

All results in grains to gallon.

| Grains of Lime to One Gallon of Sewage. | Lime in Effluent. | Precipitate dried at 212° F. | Mineral Matter. | Organic Matter. | Per Cent. | | Grains of Alkali calculated as Lime in Precipitate. |
|---|---|---|---|---|---|---|---|
| | | | | | Mineral. | Organic. | |
| 8 | 3·0 | 21·7 | 18·7 | 3·0 | 86·1 | 13·9 | 11·2 |
| 12 | 4·5 | 30·5 | 24·0 | 6·5 | 78·6 | 21·4 | 14·8 |
| 16 | 5·5 | 39·0 | 30·2 | 8·8 | 77·4 | 22·6 | 18·0 |
| 20 | 7·0 | 41·0 | 31·2 | 9·0 | 76·0 | 24·0 | 18·0 |
| 24 | 8·0 | 41·3 | 32·0 | 10·0 | 77·4 | 22·6 | 18·7 |

This plainly shows that the lime precipitates organic matter, and that 16 grains will do nearly double the work of eight, but that a further addition in this instance was of very little use. The percentage of organic matter increased from 13·9 to 22·6.

In the experiments which I have given comparing lime effluents with effluents produced from sulphate of iron, some may object and say that I am comparing effluents produced at the works by the million gallons, with effluents produced in the laboratory. Of course the latter will be far better mixed with the chemicals than they can be at the works, though I do not see why better mixture should not take place at the sewage works. To overcome the objection of wrong comparisons, I have made special experiments with about three million gallons of sewage treated with lime, and three million gallons treated with sulphate of iron and lime.

For the iron effluent were used 15 cwt. of sulphate of iron and 8 cwt. of lime; for the lime effluent were used 30 cwt. of lime.

The effluents were run into six clean tanks, and allowed to rest for about four hours.

Practically speaking these two sets of effluents are the same, the average albuminoid ammonia and oxygen of the iron being 0·30 and 1·9, and the lime 0·28 and 1·7.

Similar experiments were performed with sulphate of alumina and lime, and sulphate of alumina alone, using about three million gallons in each case, and about 10 cwt. of sulphate of alumina with 8 cwt. of lime, and in the second case 18 cwt. of sulphate of alumina alone to the million gallons.

All results in grains per gallon.

| — | No. 1.—Sulphate of Iron Effluent. | | |
|---|---|---|---|
| | Albuminoid Ammonia. | Oxygen for Four Hours. | Lime. |
| 1. Tank... | 0·22 | 1·9 | 1·0 |
| 2. „ ... | 0·25 | 1·8 | 1·0 |
| 3. „ ... | 0·29 | 1·9 | 0·75 |
| 4. „ ... | 0·33 | 1·8 | 1·0 |
| 5. „ ... | 0·36 | 1·9 | 0·85 |
| 6. „ ... | 0·36 | 2·1 | 1·0 |

| — | No. 2.—Lime Effluent. | | |
|---|---|---|---|
| | Albuminoid Ammonia. | Oxygen for Four Hours. | Free Lime. |
| 1. Tank... | 0·39 | 1·7 | 6·0 |
| 2. „ ... | 0·28 | 1·8 | 5·5 |
| 3. „ ... | 0·25 | 1·8 | 10·5 |
| 4. „ ... | 0·24 | 1·8 | 14·5 |
| 5. „ ... | 0·28 | 1·7 | 13·0 |
| 6. „ ... | 0·26 | 1·5 | 14·0 |

All results in grains per gallon.

| — | No. 3.—Sulphate of Alumina and Lime. | | No. 4.—Sulphate of Alumina alone. | |
|---|---|---|---|---|
| | Albuminoid Ammonia. | Oxygen for Four Hours. | Albuminoid Ammonia. | Oxygen for Four Hours. |
| 1. Tank. | 0·20 | 2·1 | 0·21 | 2·2 |
| 2. " . | 0·20 | 2·3 | 0·28 | 2·9 |
| 3. " . | 0·25 | 2·6 | 0·28 | 3·0 |
| 4. " . | 0·34 | 2·7 | 0·25 | 2·2 |
| 5. " . | 0·28 | 3·7 | 0·22 | 2·1 |
| 6. " . | 0·28 | 3·0 | 0·22 | 2·2 |

Here there is an average of 0·26 and 2·7 for No. 3, and 0·24 and 2·4 for No. 4 average :—

| — | Albuminoid Ammonia. | Oxygen in Four Hours. |
|---|---|---|
| No. 1. Sulphate of iron and lime. | 0·30 | 1·9 |
| No. 2. Lime only ........... | 0·28 | 1·7 |
| No. 3. Sulphate of alumina and lime. | 0·26 | 2·7 |
| No. 4. Sulphate of alumina only. | 0·24 | 2·4 |

The following table gives the analyses of the effluents prepared by lime water in the laboratory day after day for a fortnight, and, for comparison, the effluents produced at the sewage works on the same day, using the same quantity of lime in each case, about 25 cwt. to the million gallons.

All results in grains per gallon.

| Sewage Works Effluent. | | | Laboratory Effluent. | | |
|---|---|---|---|---|---|
| Albuminoid Ammonia. | Oxygen for Four Hours. | Free Lime. | Albuminoid Ammonia. | Oxygen for Four Hours. | Free Lime. |
| 0·42 | 2·8 | 3·0 | 0·19 | 1·2 | 8 |
| 0·34 | 2·5 | 4·5 | 0·21 | 2·4 | 5 |
| 0·30 | 2·1 | 3·0 | 0·19 | 1·2 | 6 |
| 0·34 | 1·7 | 4·0 | 0·21 | 1·6 | 7 |
| 0·28 | 2·5 | 2·0 | 0·19 | 0·8 | 6 |
| 0·28 | 2·6 | 2·0 | 0·20 | 1·6 | 5 |
| 0·38 | 2·8 | 6·0 | 0·21 | 2·1 | 2 |
| 0·34 | 3·5 | 6·0 | 0·20 | 2·2 | 8 |
| 0·28 | 2·5 | 5·0 | 0·24 | 2·3 | 5 |
| 0·21 | 2·2 | 3·0 | 0·28 | 2·3 | 5 |
| 0·17 | 1·6 | 3·0 | 0·20 | 2·2 | 7 |
| 0·31 | 2·6 | 2·0 | 0·21 | 1·0 | 7 |
| 0·28 | 3·9 | 1·0 | 0·16 | 1·0 | 6 |
| 0·31 | 3·0 | 1·0 | 0·28 | 1·5 | 7 |

In both of the above cases the same settled sewage was used; samples were taken every hour for 24 hours, so that a good average might be obtained.

The average albuminoid ammonia of the works effluent for the 14 days is 0·30, which gives a percentage purification of 37. The average of the laboratory effluent is 0·21, which gave a percentage purification of 56.

The four hours oxygen test average was for works effluent 2·6, or 35 per cent. of purification, whereas the average of laboratory effluent was only 1·6, which gave 60 per cent. of purification.

It is so often said that the sludge in the tanks injures the effluent considerably, particularly in the case of lime. I have now made a table of the analyses of the first two days of the fortnight and the last two days of these special experiments, and it will be seen that there is very little difference between the beginning and the end. I have given the rainfall, so that it may account for some differences.

Table giving the analyses of effluents on first two and last two days of each fortnight's experiment to show that the sludge in the tanks has not had a very injurious effect upon the effluents.

| Date. | — | Effluent. | | |
|---|---|---|---|---|
| | | Albuminoid Ammonia in 100,000. | Rain. | Oxygen, Four Hours, Grains in Gallon. |
| 1896.<br>Oct. 10 | Sulphate of Alumina and Lime | 0·28 | 0·751 | 1·2 |
| " 11 | " " " | 0·24 | 0·236 | 1·1 |
| " 24 | " " " | 0·44 | 0·321 | 1·7 |
| " 25 | " " " | 0·32 | 0·074 | 1·0 |
| Oct. 26 | Sulphate of Alumina and Lime | 0·36 | 0·100 | 1·0 |
| " 27 | " " " | 0·36 | 0·161 | 2·3 |
| Nov. 5 | " " " | 0·36 | Nil | 2·5 |
| " 6 | " " " | 0·36 | Nil | 2·2 |
| Nov. 9 | Lime: 18 cwt. per million... | 0·32 | Nil | 1·1 |
| " 10 | " " ... | 0·40 | Nil | 1·9 |
| " 19 | " " ... | 0·36 | 0·015 | 1·5 |
| " 20 | " " ... | 0·48 | 0·015 | 1·9 |
| Nov. 23 | Lime: 18 cwt. per million... | 0·48 | Nil | 4·1 |
| " 24 | " " ... | 0·40 | Nil | 3·6 |
| Dec. 6 | " " ... | 0·20 | 0·414 | 2·0 |
| " 7 | " " ... | 0·32 | 0·120 | 1·8 |
| Dec. 8 | Lime: 18 cwt. per million... | 0·32 | 0·005 | 2·6 |
| " 9 | " " ... | 0·34 | 0·03 | 2·9 |
| " 22 | " " ... | 0·44 | .. | 3·2 |
| " 23 | " " ... | 0·44 | .. | 2·9 |
| 1897.<br>Jan. 12 | Sulphate of Alumina only... | 0·32 | Nil | 1·2 |
| " 13 | " " ... | 0·32 | Nil | 2·2 |
| " 24 | " " ... | 0·36 | 0·065 | 1·6 |
| " 25 | " " ... | 0·40 | 0·146 | 1·6 |
| Jan. 29 | Lime: 12 cwt. per million... | 0·56 | 0·080 | 4·3 |
| " 30 | " " ... | 0·52 | 0·110 | 3·0 |
| Feb. 7 | " " ... | 0·40 | Nil | 1·8 |
| " 8 | " " ... | 0·60 | Nil | 2·1 |
| Feb. 9 | Lime: 13 cwt. per million... | 0·40 | 0·02 | 2·1 |
| " 10 | " " ... | 0·40 | 0·003 | 1·7 |
| " 21 | " " ... | 0·36 | 0·163 | 2·9 |
| " 22 | " " ... | 0·28 | 0·112 | 2·0 |
| Feb. 23 | Lime: 18 cwt. per million... | 0·40 | Nil | 1·4 |
| " 24 | " " ... | 0·44 | 0·006 | 2·7 |
| Mar. 8 | " " ... | 0·38 | Nil | 2·8 |
| " 9 | " " ... | 0·44 | 0·088 | 2·1 |
| Mar. 10 | Lime: 18 cwt. per million... | 0·32 | 0·013 | 2·1 |
| " 11 | " " ... | 0·40 | Nil | 2·6 |
| " 24 | " " ... | 0·40 | 0·101 | 2·7 |
| " 25 | " " ... | 0·40 | Nil | 1·6 |
| Mar. 26 | Lime: 18 cwt. per million... | 0·46 | 0·167 | 3·0 |
| " 27 | " " ... | 0·40 | 0·177 | 2·3 |
| Apr. 13 | " " ... | 0·40 | 0·073 | 2·5 |
| " 14 | " " ... | 0·40 | 0·200 | 2·6 |
| Apr. 27 | Sulphate of iron and lime ... | 0·36 | Nil | 1·6 |
| " 28 | " " ... | 0·44 | 0·006 | 2·4 |
| May 10 | " " ... | 0·40 | Nil | 2·5 |
| " 11 | " " ... | 0·56 | 0·171 | 2·3 |

In comparing lime with other chemicals, there now only remain two other precipitants for me to compare on the large scale with lime, and they are perchloride of iron, and basic persulphate of iron. With regard to the perchloride of iron that was not so successful as the lime precipitant. The analysis of the solution used was as follows :—

| | |
|---|---|
| Specific gravity...................... | 1·323 |
| Protochloride of iron ............... | 1·120 |
| Perchloride of iron .................. | 22·214 |
| Chloride of aluminium.. ........... | 1·421 |
| Sulphate of alumina ................. | 2·120 |

There were also small quantities of lime, magnesia, alkalies, which were not estimated. These I look upon as accidental impurities. Broadly speaking, the solution consisted chiefly of perchloride of iron in rather a basic condition.

Some 40 years since a preparation was brought out under the name of "Dale's Muriate of Iron," and Drs. Hofmann and Frankland made a report to the Metropolitan Board of Works. In referring to various processes for the treatment of sewage, they say, "In fact, of all the processes, old and new, referred to us, there is only one which appears to satisfy in the necessary degree the conditions involved in the circumstances of the case (to combine efficiency and economy). The agent to which we allude is that described as Dale's Muriate of Iron, essentially a solution of perchloride of iron.

Their trials led them to the conclusion that the perchloride of iron was vastly superior to either lime or chloride of lime. The quantity of sewage used in these experiments was 7,500 gallons of London sewage, and they gave, as the cost of purifying, one million gallons of sewage with—

| | £ | *s.* | *d.* |
|---|---|---|---|
| Ferric chloride................ | 1 | 13 | 3 |
| Chloride of lime .............. | 2 | 2 | 10½ |
| Lime ........................ | 3 | 6 | 6 |

They report, "That after two days the sewage disinfected by lime became slightly tainted, while the chloride of lime and perchloride of iron remained perfectly odourless. At the end of three days the limed sewage had become decidedly offensive."

This does not accord with my experience, working for some years upon the Salford sewage, and sewage from the surrounding districts. But it does seem strange to think that any sewage, which was treated with 5 tons of lime to the million gallons, could be offensive in three days. It is true that when the lime is added to the sewage there is a peculiar odour evolved, which I attribute to trimethylamine, but in all my experience I have never found a sewage treated with half the above quantity of lime become offensive in three days.

An effluent with that quantity of lime in solution would be sterile, for from experiments which have been carried out, it has been found that water containing 0·0074 per cent. of lime is sufficient to kill the typhus bacillus, while a strength of 0·0246 destroys the cholera bacillus within a few hours. If the above prices were reversed, it would be more in accordance with to-day's market, for to prepare an effluent with perchloride of iron which shall not decompose, could not be done at the above price.

These experimental trials are not always comparable with those working upon hundreds of millions of gallons.

The report says, "Another important element, in estimating the comparative fitness of an agent for the treatment of sewage, is, the time required for clarification after the addition of the disinfectant. In this respect, also, the results of the preceding experiments lead us to give a decided preference to perchloride of iron." This again does not agree with my experience on the large scale, for I have found that lime used in sufficient quantity is the best and quickest of all clarifiers. In regard to perchloride of iron, an experiment was performed which lasted 16 days, on a continuous flow of sewage through the six large tanks spoken about before, using millions of gallons on each day; and to save a great array of figures, I give a whole number instead of decimals, as a difference of a few hundred gallons will not sensibly alter the results.

Those having to do with alumina and iron precipitates know that these two, being of a flocculent nature, are easily disturbed by a flowing liquid, and thus the suspended matter must be greater than in the case of lime, which settles down to a denser sediment, and is not so easily disturbed.

186 million gallons were treated with about 1 ton of perchloride of iron and about 6 cwt. of lime to the million gallons. The resulting effluent was not any better than lime. The quantity of sludge formed in the tanks was 3.472 tons, or about 18 tons 13 cwt. to the million gallons. This is rather less than lime, but if the quantity of suspended matter, which went away in the effluent, be added, it will make the iron sludge equal the lime, if not more.

The following table gives the number of grains of suspended matter in each gallon of effluent which flows away, and, for comparison, a similar experiment was performed with a lime effluent.

| Perchloride of Iron. | | Lime. | |
|---|---|---|---|
| Gallons of Sewage. | Suspended Matter in Effluent. | Gallons of Sewage. | Suspended Matter in Effluent. |
| Millions. | Grains to Gall. | Millions. | Grains to Gall. |
| 11 | 10·5 | 8 | 2·5 |
| 8 | 10·5 | 10 | 1·5 |
| 5 | 3·0 | 9 | 1·5 |
| 8 | 12·0 | 10 | 3·0 |
| 2 | 12·0 | 11 | 3·0 |
| 9 | 9·5 | 9 | 2·0 |
| 11 | 10·0 | 8 | 1·0 |
| 11 | 8·0 | 10 | 2·0 |
| 8 | 9·5 | 8 | 1·5 |
| 5 | 10·0 | 11 | 3·5 |
| 12 | 5·5 | 10 | 2·5 |
| 10 | 8·5 | 8 | 2·0 |
| 11 | 4·0 | 11 | 3·0 |
| 4 | 7·5 | 7 | 1·0 |
| 10 | 11·5 | 9 | 2·5 |
| 10 | 6·5 | 10 | 2·0 |

The best iron precipitant for sewage when filtration is not practicable, is the basic persulphate of iron. This salt is so sensitive that immediately it is brought into contact with organic matter, which is in solution in the sewage, a great percentage of this organic matter is precipitated as a flocculent mass. About 6 million gallons of sewage were treated with about 1 ton of the iron salt to the million gallons. Taking the average of the effluents they were exceedingly good. The average albuminoid ammonia from 58 samples was 0·16 grain to a gallon, and the average amount of purification was 48 per cent. This is considerably higher than any in the list, but the price is also very much higher. The average of the four hours oxygen test was only 1·6, giving a percentage purification of 44, and the total quantity of sludge formed was 106 tons. The cost of purifying a million gallons, including removal of sludge, would be 3*l.* 7*s.*; this is higher than any in the list.

The great drawback to using solutions of chemicals at sewage works is the uncertainty of their composition. When the perchloride of iron experiment was tried, the different samples from the 200 tons varied very much, but the solutions sent in by Sir Thomas Wardle were always uniform. The following are a few of the samples delivered at the works:—

| —— | 1. | 2. | 3. | 4. | 5. | 6. |
|---|---|---|---|---|---|---|
| Specific gravity ..... | 1·51 | 1·51 | 1·50 | 1·51 | 1·52 | 1·50 |
| Ferric oxide......... | 19·21 | 19·42 | 18·61 | 19·3 | 19·21 | 18·91 |
| Ferrons oxide....... | 0·34 | 0·40 | 0·52 | 0·47 | 0·23 | 0·42 |
| Sulphuric acid ...... | 21·42 | 22·56 | 20·5 | 22·1 | 21·22 | 21·2 |

These were very basic; there are several basic sulphates varying more or less, according to the amounts of sulphuric acid and iron which they contain. The number of basic sulphates which are said to exist is 15, and if the water is left out of the calculation, the percentage of ferric oxide contained in these bodies varies between 22 and 93 per cent.

If the above are calculated out it will be found that they are very basic, and therefore very sensitive to change; even dilution causes the precipitation of oxide of iron, and this nascent precipitate is enabled to attach to itself organic matter, exactly in the same manner as in the case of dyeing.

To sum up all the results given, it plainly shows that a pure precipitation process will only remove at the most about 50 per cent. of the impurity, and that to obtain greater purification recourse must be had to filtration.

During the last eight years I have experimented with about 25 different processes which have been tried at the

Salford sewage works, and it was only the combination of a precipitating process with that of filtration which produced satisfactory effluents. Even these filtration processes varied in percentage of purification from 50 to 90 per cent. calculated upon the settled sewage. I wish it to be clearly understood that in all my sewage experiments, my purification numbers are upon the settled sewage, except otherwise stated. In my opinion this is the only true method of judging the merits of any process, and it is most fallacious to quote from the shaken sewage. For instance, a shaken sewage might require 6·0 grains of oxygen to the gallon, and the effluent after some patent treatment would require 2·0 grains. It would then be said that there was 66 per cent. of purification, when in reality there had been none, for when the clear sewage was examined it would be found that it only required 2·0 grains of oxygen to the gallon, the same as the effluent, and the albuminoid ammonia numbers would be found to correspond.

As an illustration of this method, I had to report to the corporation that a patent process for the purification of sewage was worthless, the purification being under 20 per cent. But the chemist to the company reported a purification of 75 per cent., principally derived from what had settled when the sewage passed through the tanks.

The mechanical sand filters, such as the Riddell and Thwaites, produced most excellent effluents as regards brightness and clearness, but judged by the albuminoid ammonia and oxygen tests the effluents were not equal to those produced by other filters made up of patent materials.

Not any of the patent filters filtered the whole of the effluent, which amounted to something like 10 to 12 million gallons a day, with the exception of the Riddell, which filtered nearly 1 million gallons daily, the other experimenters only filtered about 100,000 gallons during the 24 hours, the rate of filtration to the square yard being about 500 gallons. The filters were made up of various materials. Thus one filter was made up as follows: on the top of the bed of patent material was a layer of clean sand from 6 to 9 ins. in thickness; below this was a layer of the patent material, mixed with sand, about 12 ins. thick. Below this stratum was a layer of 3 ins. of sand; then an equal layer of coarse gravel; then a layer of pebbles of the same thickness; then next some large stones about 2 ins. in diameter, in which were embedded land drain pipes, so that the effluent is evenly collected from the whole of the filter bed.

Another filter was made up of some carbonaceous material and clay, which mixture had been heated in closed cylinders for some hours, the iron oxide in the material being reduced to metallic iron. This is similar to the process of making malleable iron from hematite, by embedding the mineral in charcoal and heating to a low temperature for some hours. The whole of the oxide will be reduced to malleable iron. A third filter was made up of top sand, 12 ins.; patent material, 8 ins.; sand, 6 ins.; small gravel, 4 ins.; larger gravel, 6 ins. A fourth was made of 8 ins. of top sand; then 12 ins. of patent material and sand in equal proportions; then 4 ins. of fine sand, 4 ins. of sea gravel, and 4 ins. of broken material, such as bricks, &c. A fifth was made up of sand alone; while the sixth was made from a waste material produced during the manufacture of aniline. This last was totally unfit for a filter, as it was to a certain extent soluble in water. This last was tried in comparison with a sand filter, the same effluent being run on to each. After about a week's trial it was found that the effluent was fast dissolving the filter. The following gives total solids in grains to gallon :—

| Sand Filter. | Patent Filter. | Sand Filter. | Patent Filter. |
|---|---|---|---|
| 90 | 441 | 78 | 244 |
| 84 | 282 | 128 | 267 |
| 82 | 268 | 92 | 201 |
| 77 | 274 | 100 | 232 |

At the rate the filter was being dissolved it would require to be entirely renewed in less than a month.

The first four filters named gave first-class effluents, and price would be the only thing to guide one in the matter of choice. These four filters were worked for days and weeks, and the albuminoid ammonia varied from 0·1 to 0·13 grain to gallon, and the four-hours oxygen test varied from 0·3 to 0·6 of a grain to gallon. The rate of filtration was about 500 galls. to the square yard in the 24 hours; the filters rested for some hours out of the 24. The effluents which were run on to these filters had all been treated with sulphate of alumina, sulphate of iron, or some other chemical, but no lime was used. This treatment produced a good but expensive effluent, ranging from 50s. to 60s. the million gallons. All the experimenters were afraid of using lime for their effluents, as it would block the filters and cement them into a solid mass.

I have experimented with various filters from 1 ft. in thickness to 8 ft. In nearly all cases the effluents have been lime-treated, and in no case have the filters become choked. Filters were tried with two and three ventilating floors, but this so-called improvement is a needless expense, because the filters which had the ventilating layers, though giving first-class effluents, were not so good as the effluents from the filters which had no ventilators.

It would be most tedious to recite all the analyses from the various filters, but I may sum up results by saying that the 5-ft. filters gave as good an effluent as the 8-ft.

The two 8-ft. filters have been working for some years side by side, one with two ventilating floors and the other with none. Practically speaking, these filters never rest, but are worked night and day; the effluent is sprayed on to the bed by means of special jets. The surfaces of the filters are never allowed to be covered with water, the object being for the effluent to drag down the air, thus aerating the filth if it requires it.

It will be quite sufficient in speaking about the analyses of the effluents to simply give the oxygen required for four hours, and percentage of purification.

Of course the purity of the effluent must be effected by the speed with which it passes through the filter. Thus, one of the filters (5-ft.) was worked day after day with increasing quantities of effluent from 500 to 1,000 galls. to the square yard.

| Gallons to Square Yard. | Oxygen required for Four Hours. | Gallons to Square Yard. | Oxygen required for Four Hours. |
|---|---|---|---|
| | Grs. per Gall. | | Grs. per Gall. |
| 500 | 0·45 | 750 | 0·64 |
| 500 | 0·40 | 750 | 0·72 |
| 500 | 0·40 | 800 | 0·57 |
| 550 | 0·35 | 800 | 0·64 |
| 550 | 0·40 | 800 | 0·60 |
| 550 | 0·48 | 850 | 0·58 |
| 600 | 0·50 | 850 | 0·74 |
| 600 | 0·40 | 850 | 0·80 |
| 600 | 0·40 | 900 | 1·20 |
| 650 | 0·56 | 900 | 0·84 |
| 650 | 0·48 | 900 | 1·10 |
| 650 | 0·44 | 950 | 0·96 |
| 700 | 0·62 | 950 | 0·84 |
| 700 | 0·68 | 950 | 1·20 |
| 700 | 0·60 | 1000 | 1·10 |
| 750 | 0·56 | | |

The percentage of purification varied from 60 to 95 per cent. I need not say which were the high percentages, because as the quantity of effluent increased so did the purification decrease, and it has been found that the best results have been produced at 500 galls. to square yard.

I must not forget to mention that all these effluents had been passed through roughing filters before going on to the fine filters. The roughing filters may be made of coke, cinders, and sand, and may pass from three to four thousand gallons to the square yard, the filters acting more as a strainer than a filter.

The following analyses will show that very little purification is done by the roughing filter. It was thought that if the effluent was brought into a very fine spray, and then delivered on to the filter, it would rapidly oxidise the putrescent matter. Practically speaking, very little oxidation took place.

To prove what the roughing filters could do, also the spraying of the effluent, combined with the ventilating and non-ventilating filters, the following samples were taken.

Analyses were made of the sewage and effluents, but here it will be sufficient to give the oxygen absorbed for four hours:—

No. 1. Sewage.
„ 2. Lime effluent.
„ 3. Effluent after roughing filter.
„ 4. Effluent caught from spray before going on to fine filter.
„ 5. Effluent from ventilated filter.
„ 6. Effluent from non-ventilated filter.

*Grains of Oxygen Absorbed, by Four-Hours Test, in One Gallon.*

| 1. | 2. | 3. | 4. | 5. | 6. |
|---|---|---|---|---|---|
| 4·4 | 3·8 | 3·0 | 2·9 | 0·87 | 0·52 |
| 4·0 | 2·8 | 2·3 | 2·3 | 0·64 | 0·60 |
| 3·8 | 2·9 | 2·2 | 2·2 | 0·70 | 0·60 |
| 2·6 | 2·1 | 2·0 | 1·9 | 0·30 | 0·30 |
| 3·4 | 2·2 | 2·0 | 2·0 | 0·45 | 0·35 |
| 4·6 | 2·8 | 2·6 | 2·5 | 0·75 | 0·65 |
| 4·2 | 2·4 | 2·2 | 2·0 | 0·82 | 0·70 |
| 3·8 | 2·0 | 2·0 | 2·0 | 0·64 | 0·60 |
| 4·9 | 3·1 | 3·0 | 2·9 | 0·78 | 0·65 |
| 2·9 | 2·0 | 1·8 | 1·8 | 0·44 | 0·42 |

These samples were taken on different days. The above shows very plainly that there is no advantage in having ventilated partitions in the filters.

I do not depend upon these ten sets of experiments alone, for the statement which I have made has been confirmed by hundreds of analyses, as samples have been taken every day for months, and even running into years.

All the filters I have spoken about are merely experimental filters, filtering about 100,000 gallons a day, and I am not prepared to say that the filters would last so well when they were called upon to filter millions of gallons in the 24 hours. I do not see any reason why the large filters should fail, when these small ones have been successfully worked for three or four years.

After nearly ten years of experimenting I cannot see much difficulty in treating sewage, if only a proper amount of labour and expense is brought to bear upon it. And in this paper I think I have proved that lime is as good a precipitant as more expensive chemicals.

## New York Section.

*Meeting held at the College of Pharmacy, on Friday, May 20th, 1898.*

PROF. CHAS. F. CHANDLER IN THE CHAIR.

### THE DETERIORATION OF RAW CANE SUGAR IN TRANSIT OR STORAGE.

BY EDMUND C. SHOREY, D.SC.

Raw cane sugar is generally sold at a price based on its polariscope test, or, as it is more generally stated, on its polarisation. The polarisation represents, approximately, the amount of sucrose, or pure cane sugar in the raw sugar.

These raw sugars frequently in the course of transit, or when stored for some time, give a lower polariscope test than when they were made, which of course means that there has been a loss of sucrose. This deterioration of raw cane sugar is known wherever such sugar is made, and often results in large loss to the planter who sells, as well as the refiner who buys.

In the case of Hawaiian sugars the loss by deterioration has been brought more prominently to the notice of the planters by the shipment for the past two years of a portion of this sugar to New York *viâ* the Horn, which is a three months' voyage, instead of to San Francisco, which is a voyage of about three weeks. Some lots of sugar shipped from Honolulu to New York are stated to have lost as much as 8 per cent., and to have been in such a condition when taken from the ship's hold, that they "fell all to pieces" unless worked up immediately, entailing considerable loss to the refiners. Quite naturally the reason for this deterioration has been the cause of much thought and speculation on the part of those interested.

It has been stated, and has been generally accepted, that the primary cause is some form of fermentation, by which the sugar is inverted or destroyed. The reasons put forward why this fermentation should take place in some sugars and not in others, have been numerous; and have embraced the whole matter of cane-growing and sugar-making from the condition of the soil to the drying of the sugar in centrifugals.

The reason most prominently put forward has been that sugars which have deteriorated have done so either because they were not well dried, or not sufficiently alkaline, and coupled with this the statement that the deterioration has been brought about by bacteria; prominent among which are those producing lactic and butyric acid. The inference of course is that these bacteria would not produce fermentation in an alkaline medium, or in one with little moisture.

In discussing this matter in the local sugar journal (Planters Monthly) I have pointed out that the bacteria of the lactic and butyric ferments are found in all raw sugars, and that if the deterioration is caused by these bacteria, it may be accepted as an axiom that if one sugar deteriorates and another does not, it is because one presents the proper conditions for growth of these bacteria, and the other does not. I have also pointed out that if the butyric ferment is active in this deterioration the fact that the common butyric ferment is anaerobic must be borne in mind in attempting to solve this problem.

Within the past year I have had the opportunity of studying some lots of sugar which had deteriorated in storage, and as the result of this study I have solved the problem so far as the particular sugars are concerned. How far the explanation will apply to all raw cane sugars is a question which the future only can answer.

The history of the sugars in question—three lots—is briefly as follows.

*Lot No.* 1.

Hawaiian sugar made July 6th, 1897, polarisation 98·1°, contained CaO 0·032 per cent., reducing sugar 0·21 per cent. alkalinity equivalent to 0·022 per cent. CaO.

This sugar was shipped to San Francisco July 12th, and arrived there August 11th, 1897, polarising 97·7°. A portion of this lot—five tons—was not shipped, but kept in the mill where it was made, stored in bags of 125 lbs. each. A sample was taken from this sugar in storage September 4th, which polarised 96·5°, a decline of 1·6°, and a sample taken December 28th polarised 94·0°, a decline of 4·1°.

This sugar at this time was still alkaline, possessing an alkalinity equivalent to 0·0168 per cent. CaO, and contained 4·1 per cent. reducing sugar.

At this time, this sugar, especially in the portion next the bag, was quite moist, but the gain in weight from moisture was not enough to show on the ordinary sugar house scales; less than half a pound per bag. This sugar was originally much drier than the average raw sugar, being dried twice the usual time in centrifugals running 1,500 revolutions per minute, and contained only 0·30 per cent. moisture.

*Lot No.* 2.

Hawaiian sugar made in July, 1897, polarised 98·5°, made from neutral juice, sugar neutral. This sugar was shipped to San Francisco soon after it was made, and polarised there 98·5°. A portion was stored in the mill, and a sample taken January 26th, 1898, was slightly acid, and polarised 96·5°, a decline of 2·0°.

*Lot No.* 3.

Washed sugar, made from No. 1 Hawaiian polarising 98·8° by washing in the centrifugals with water, and drying in the sun, made for local consumption. This sugar was made in July, 1897, polarised 99·5°, and was slighly alkaline. A portion of this lot was stored in the mill in kegs holding 100 lbs. each. In January, 1898, one keg was opened, the portion next the keg, especially at the bottom, was found to be quite damp, and a sample from this portion was acid in reaction, polarised 85·5°, and contained 7·3 per cent. reducing sugar. The sugar in the centre of the keg was apparently unchanged, polarising 99·5°, and containing 0·14 per cent. moisture.

In each of these cases the sugars were stored in rooms which were neither very dry nor very damp. There was in no case any damage from accidental access of water; the floors were quite dry and the moisture taken up by the sugar must have been obtained from the moisture in the air.

It will be noted that the history of lot No. 1 is fuller than that of the others, and it is with this sugar that I wish especially to deal in this paper.

It will be noticed in the first place that the loss was due almost wholly to inversion: the sugar after having lost 4·0° was still alkaline, and the acid produced was only sufficient to reduce the alkalinity from 0·022 per cent. to 0·0168 per cent. In correlation to this stands the fact, that the refiners in San Francisco and New York, when complaining of deterioration, have always spoken of inversion. It is plain, then, that, whatever the cause of deterioration, and whatever the final products, inversion of the cane sugar is the first step.

The alkalinity of the sugar when made was quite high, as was also that of the juice from which it was made; in fact, much higher than is usual in Hawaiian sugar houses. I obtained for comparison two samples of No. 1 sugar from Ewa Plantation, which sugar has the reputation of being less apt to deteriorate than any Hawaiian sugar. These two samples showed an alkalinity respectively of 0·0136 per cent. and 0·014 per cent. CaO, much lower than that of sugar lot No. 1, in fact, lower than this sugar was when it had lost 1·0°.

In view of the well-known fact that the lactic and butyric ferments require slight alkalinity for vigorous growth, it seems rather astonishing that anyone should put forward the theory, that lack of alkalinity, or lack of lime, is the cause of deterioration; coupled with the statement that the lactic and butyric ferments produce deterioration.

Inasmuch as highly alkaline cane juice gives a sugar objectionably high in ash, and also results in difficult working of low grades, and an increased loss in manufacture, it is easily seen that this is an important question. If making an alkaline sugar does not insure against deterioration, the practice is not to be recommended.

Many of the mistakes which have been made in treating this subject have arisen from taking the results of experiments with ferments in sugar solutions, and applying them to sugar in the dry state. Keeping this well in mind, I made a preliminary examination of sugar lot No. 1 in the following manner:—

A solution was made containing 10 grams of the fermented sugar in 100 c.c. solution. Portions of 100 c.c. were treated as follows: the series being duplicated, and the flasks in every case protected from access of dust from the air—

(1) 100 c.c. solution at ordinary temperature 75° F. for 24 hrs. had become neutral, and contained invert sugar 5·2 per cent., a gain of 1·1 per cent.

(2) 100 c.c. boiled one minute, plugged with cotton wool, at end of 24 hours was still alkaline, and contained invert sugar 4 per cent.

(3) 100 c.c. boiled five minutes as above with same result.

(4) 100 c.c. kept for 24 hours at 103° F. was then acid, and contained invert sugar 7·1 per cent., a gain of 3·0 per cent. The acidity was such that 100 c.c. required 2 c.c. tenth normal alkali to bring about neutrality.

(5) 100 c.c. solution, to which had been added 0·5 grm. CaO, kept at the temperature of laboratory 24 hours contained; then invert sugar 4·5 per cent., a gain of 0·5 per cent.

(6) 100 c.c. solution, to which had been added 0·1 grm. CaO, kept at 75° F. for 24 hours contained; then invert sugar 4·7 per cent., and became acid in four days.

From this series it may be inferred, I think, that there was present in the sugar in question some ferment, which in solution at the ordinary temperature produced some acid and inverted some of the sugar; that a temperature of 103° F. this action was more rapid; that boiling for one minute rendered this ferment inactive, and that in quite strongly alkaline solution, the invertive action still went on.

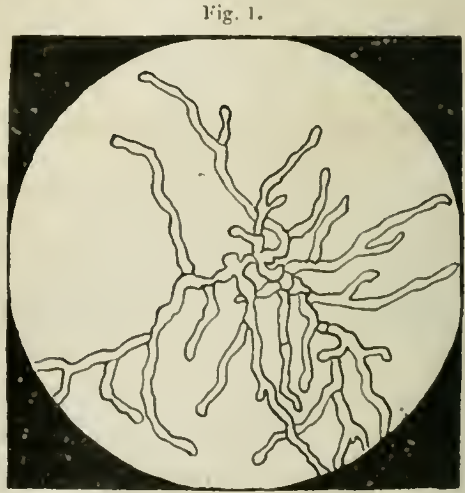
It is generally accepted that the butyric ferments are without the power of inverting cane sugar, while the lactic ferments sometimes seem to have this power. It seemed to me, however, very unlikely that the inversion could be brought about by bacteria. The sugar was quite dry, the crystal separate and distinct, and in appearance was like so much air-dried sand. Even in December, when the deterioration had reached 4·0°, although the sugar was moist, each crystal was still separate and distinct, being simply coated with a thin syrupy film. Bacteria can only reach development in a liquid or semi-liquid continuous medium, and sugar, so long as it continued so dry that the crystals remained distinct and separated by air spaces, would necessarily prove a medium ill-adapted for the growth of such bacteria as produce lactic or butyric acid. Moreover, all the sugars examined showed as the result of deterioration a very small amount of acid, and a comparatively large amount of invert sugar. Now, if lactic acid ferments were the invertive agents, the processes of inversion and production of acid would, undoubtedly, go on together, or so closely that they would seem simultaneous. I was led to conclude, then, that the inversion was not produced by lactic or butyric ferments, but by some other agent.

The same of lot No. 1, taken in September, polarising 96·5°, was placed in a tightly stoppered bottle, and when the final examination of the sugar was made in December, was opened and found to be unchanged.

A 10 per cent. solution of this sample was made, and examined under the microscope, when a pronounced development of a fungus growth was at once discovered, each growth presenting an appearance of which Fig. 1. is a sample. As much of the solution as could be placed under a cover glass 18 mm. diam. contained three or four such growths. Several of these were separated and transferred to sterilised bread, and after two or three days the growth was recognised as the common mould *penicillium glaucum*.

Fig. 1. is shown the mycelium, being formed by the growth from the germs (conidia) which form the several centres of growth. An examination of a bag of this sugar, which up to this time had not been opened, showed that the growth of mould was all through the sugar; that near the bag where the sugar had taken up most moisture from the air, the growth was in an advanced stage, conidiphores

Fig. 1.



being formed, and where the sugar was moist enough to form a semi-liquid mass, the mycelium was beginning to assume irregularly swelled shapes as in Fig. II., or to break up as in *a*, Fig. III. As the centre of the bag was approached, the development became less and less advanced, but the centres of development were just as numerous. As these centres of growth, in addition to being numerous, were separate and distinct, it was proved that the growth of mould throughout the bag proceeded from germs which had been introduced into and disseminated through the sugar before it was placed in bags.

Fig. II.

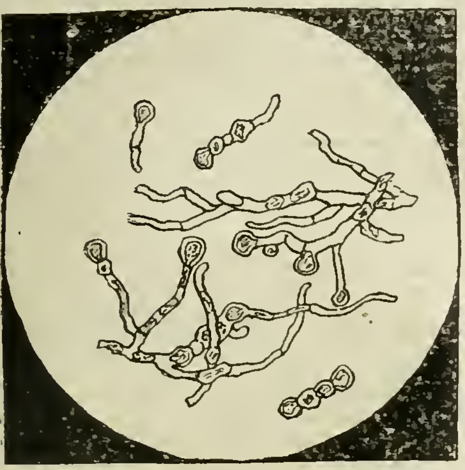
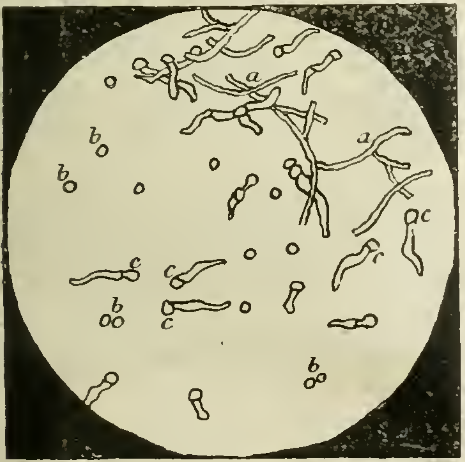


Fig. III.



A portion of the semi-liquid portion taken from next the bag was placed under a bell jar, when in a day or so the mould developed vigorously on the surface, and a portion examined under the microscope presented the appearance shown in Fig. III. *a* is the old mycelium breaking up owing to submission in the semi-liquid mass; *b* are conidia, and *c* are conidia beginning a new growth on the surface of the medium. This portion soon became quite liquid and strongly acid.

It is well known that *penicillium glaucum* has the power of inverting cane sugar, and the rationale of deterioration of raw cane sugar from the growth of this mould seems to be: first, the growth of the mould on the crystals of comparatively dry sugar, such growth being in proportion to the access of moist air; second, this growth of mould inverts some cane sugar, and as inversion proceeds moisture is taken up from the air; and when the moisture taken up is enough to produce a mass like massecuite, the mycelium begins to break up and dissolve, growth taking place only on the surface of the mass; third, at this stage the sugar is in the proper condition, both chemically and mechanically, for the growth of acid-producing bacteria, and if the other conditions of growth are right, the subsequent breaking up of the sugar will be very rapid.

In the case of all samples separate crystals were examined by spreading on a cover glass, and inverting over a hollow slide; when under the microscope, growths similar to that shown in Fig. I. were found growing *on* the crystals, the branches of the mycelium enveloping them much as an octopus might embrace its prey.

A microscopic examination of sugar lots 2 and 3 showed similar growths of the same mould, and they were found even in the dry portion of the washed sugar polarising 99·5°, not in a vigorous condition it is true, but sufficiently to be identified.

The case in hand, then, was as follows. A lot of dry raw sugar was found to be undergoing inversion, due no doubt to the growth of the mould *penicillium glaucum*, which was found growing in such a way throughout the bags that the germs must have become mixed with the sugar before it was placed in the bags. The important questions to be answered were: How did the germs get in the sugar? How can their presence be prevented?

It is well known that the germs (conidia) of *penicillium glaucum* are universally present in the air. I have proved this to be the case here by experiments, the details, of which I will not go into now.

Taking for granted that the germs of this mould are destroyed at the temperature of boiling water, there is only one place in the process of sugar-making as carried on here, where these germs could gain access to the sugar so as to be mixed through it, and that is in *the centrifugals*. It is the practice here to dry the massecuite hot as soon as it is dropped from the vacuum pan, and the sugar is dropped from the centrifugals directly into the bags. In the process of drying sugar in centrifugals, there is produced by the rapid rotation of the machine a downward current of air along the central spindle, into the basket, and outward to or through the sugar. This can be proved by arranging a beam of light, which will show the dust particles in the air in such a way as to shine on the spindle of a running centrifugal, when the rapid current of dust particles downward and outward can easily be seen. The sugar, then, in the rapidly running centrifugal really serves as a strainer or filter for a considerable portion of air, and any germs carried in the air are of course deposited on the inner surface of the dried sugar, and when this is dug out they become mixed through the whole mass. A repeated examination of sugar, dried in a dusty atmosphere, has confirmed this.

It has already been stated that the germs of *penicillium glaucum* are probably always present in the air, and there is no doubt that in windy and dusty weather they are present in very great abundance. It is worthy of note in this connection that, as far as my knowledge goes, all sugars which have deteriorated have been made when there was a large amount of dust in the air, that is in dry windy weather; and it is also to be noted that the plantations which have become noted for producing sugar, which deteriorates in transit or storage, are situated in districts subject to dry weather and high winds. In districts where there is little dust or little wind, so far as I know, the sugar made does not deteriorate.

Since the germs of the invertive mould gain access to the sugar in the centrifugals, the prevention is a comparatively easy matter. Either closed centrifugals must be used; or something must be done to sterilise the sugar before it is dropped into the bags. Steam affords the readiest means of bringing about the latter; and at present I am using it in the following manner. Beside each centrifugal there is placed a 1-inch steam pipe supplying steam at 80 lbs. pressure; this pipe is fitted with a valve and a short piece of hose. After the brake has been put on the centrifugal, and just as it begins to run slowly, steam is turned on, and the end of the hose directed by hand against the surface of the dried sugar for about ten seconds. This use of steam dissolves very little sugar, aids in the drying, and prevents, I am sure, any great number of mould germs going uninjured into the sugar bags.

I have isolated the invertive ferment present in *penicillium glaucum* in an impure form in the following manner, operating both on the fermented sugar and on the mould itself grown on the surface of molasses. The sugar or mould was macerated with about four times its weight of glycerine, filtered, and the filtrate mixed with four times its volume of 95 per cent. alcohol. The dirty white precipitate thrown down was removed by filtration, well washed with alcohol, and dissolved in water. This water solution inverted cane sugar at the ordinary temperature, but the action was much more rapid at 100° to 103° F.

*Penicillium glaucum* thrives best at a temperature of about 68° F., and some of the phenomena, connected with the fermentation and subsequent rapid breaking up of raw sugar, may have been due to the rapid development of this mould at the low temperature of a ship's hold, and subsequent rapid inversion by the invertive ferment secreted by the mould, when the sugar was transferred to the higher temperatures of the refinery or store room.

That alkalinity of sugar is no preventive of deterioration from the growth of *penicillium glaucum* has been shown from the history of the sugars already given, and is still more conclusively proved by the following experiment. A 10 per cent. solution of raw sugar was sterilised by boiling for several hours; after cooling, caustic lime was added to different portions in the following proportion :—

(1) 0·1 grm. CaO per 10 grms. of sugar.
(2) 0·05 „ „ „
(3) 0·01 „ „ „
(4) No CaO

To each of these was then added a few crystals of the fermented sugar, precautions being taken in adding the sugar as well as the lime to prevent contamination from the air. In each of these solutions a growth of *penicillium glaucum* soon appeared, and the growth in No. 1 containing lime to the extent of 1 per cent. of the sugar was quite as vigorous as in the others.

In presenting this explanation of the depreciation of these lots of raw sugar, I do not say that it applies to all sugars which have deteriorated in transit or storage, but it certainly is one that could be applied to all such sugars without doing violence to any known facts or accepted theories; and it proves that the assertion that all sugars which deteriorate do so because too moist or not alkaline enough is unwarranted.

## THE TANNINS FROM SOME INDIA CONIFER BARKS.

BY HENRY TRIMBLE.

The samples of bark considered in this paper were received from Mr. A. E. Wild, Conservator of Forests at Dehra Dún, India; and since they originated on British soil and concern British industry, it is fitting that the results derived from the investigation of them should first be presented to this Society.

Judging from the date of their shipment, these samples were probably collected in February. The following is a brief account of the sources from which the barks were derived, for which I am chiefly indebted to a private communication from Mr. Wild, and to the *Dictionary of Economic Products of India*, by Watt.

*Abies dumosa*, London (in Genera Plantarum referred to *Tsuga*). Indian Hemlock Spruce. This is a large tree met with in North-east Kumaon, Nepal, and Sikkim. The wood is used for shingles and the bark for roofing.

*Abies Webbiana*, Loudon (in Genera Plantarum referred to *Picea Webbiana*, Loudon). The Himalayan Silver Fir. A lofty evergreen, found in the Himalayas from the Indus to Bhután. The wood yields a white resin, and a beautiful violet dye is extracted from the cones. The wood and bark are used respectively for shingles and roofing.

*Cryptomeria Japonica*, Don. Japan Cedar. A medium-sized, handsome tree, native of China and Japan, but cultivated throughout the districts of Darjeeling and Senila. It has a white, soft, uniform wood, much used in Japan.

*Juniperus recurva*, Ham. The Weeping Blue Juniper. A very variable tree, attaining a height of some 30 feet, and found in the temperate and Alpine Himalayas. The wood contains a large amount of resin, and is used, as are the leaves and twigs, for incense. The tree is cultivated for ornament in Europe and America.

*Pinus longifolia*, Roxb. Emodi Pine. Long-leaved Pine, Cheer Pine. A large tree attaining a maximum height of 100 feet, and found on the outer and drier Himalayas from the Indus to Bhután. The total area occupied by it in North-West India is 2,000 to 4,000 square miles. It is one of the most important evergreens to the natives because of its resin, which yields an excellent oil of turpentine. The bark is used for tanning and contains a yellowish dye. The seeds are eaten when food is scarce, and the wood is used for fuel.

*Taxus baccata*, Linn. Yew. An evergreen tree widely distributed in the Eastern Hemisphere, and especially in the temperate Himalayas, where it sometimes attains a height of 100 feet. The tree yields a gummy exudation, which forms a portion of the incense used in Thibet. According to Madden the people of Ladak import yew bark from Kashmir and Kanawar on account of the inner part, which they employ as a red dye. The wood is used for baskets, furniture, and clogs, and is highly prized for bows. The bark is said to be employed as a substitute for, or mixed with, tea.

The moisture, ash, and tannin were estimated in each of the samples, with the following percentage results :—

| — | Moisture. | Ash in Dry Bark. | Tannin in Air Dry Bark. | Tannin in absolutely Dry Bark. |
|---|---|---|---|---|
| Abies dumosa | 12·24 | 4·83 | 10·26 | 11·69 |
| Abies Webbiana | 11·69 | 4·90 | 6·90 | 7·81 |
| Cryptomeria Japonica | 13·15 | 4·21 | 6·37 | 7·33 |
| Juniperus recurva | 11·25 | 4·96 | 8·23 | 9·27 |
| Pinus longifolia | 11·75 | 2·33 | 12·90 | 14·62 |
| Taxus baccata | 14·38 | 6·19 | 19·55 | 22·83 |

The *Pinus longifolia* is the only one of these about which I have found any statement of use in tanning, although *Abies dumosa* and *Taxus baccata* might also be advantageously employed for tanning purposes. The others could, perhaps, only be profitably used in the absence of richer material.

A study of the tannin of these barks was made by extracting each with acetone, purifying the resulting tannin, and submitting it to combustion. The following percentage results were obtained :—

| — | Carbon. | Hydrogen. |
|---|---|---|
| Abies dumosa | 61·32 | 4·89 |
| Abies Webbiana | 59·91 | 5·12 |
| Cryptomeria Japonica | 61·73 | 4·72 |
| Juniperus recurva | 59·99 | 4·71 |
| Pinus longifolia | 62·50 | 5·28 |
| Taxus baccata | 63·00 | 5·10 |

When we take into account that tannin is an amorphous substance and purified with great difficulty, we must conclude that the percentage composition of these tannins is sufficiently close to warrant the conclusion that they are identical; and when we further consider that this percentage composition averages very nearly to the percentage composition of oak bark tannin, and we find that the behaviour of all these tannins towards ferric salt, bromine water, and calcium hydrate is similar, and closely resembles the behaviour of oak bark tannin towards the same reagents, we are forced to the conclusion that these tannins are not only identical with one another, but that they are also identical with oak bark tannin.

Over four years ago (*The Tannins*, Vol. II.) I made the statement that no tannin had been studied by me that did not yield, on elementary analysis, results which classed it with the oak bark tannins containing about 60 per cent.

carbon, and 5 per cent. hydrogen, or else with nutgall tannin containing carbon 52·10 per cent. and hydrogen 3·52 per cent. Since then my list has been considerably extended, with results that strongly confirm my original opinion. This list now includes the tannins from several wattle barks, eucalyptus gum, the palmettos of southern United States, some plants from the natural order *Ericaceae*, and a large number of barks from coniferous trees. These tannins nearly all belong to the oak bark group, while the examination of a number of species of sumacs and a few ericaceae shows them to yield tannins which belong with the gall tannin group.

Another object of the present examination was geographical. These conifer barks from India have been found to contain tannins identical with those found in the barks derived from the same natural order in America; therefore, so far as studied geographically, distribution has caused no variation in the tannins of the conifers.

## Nottingham Section.

*Meeting held on Wednesday, May 25th, 1898.*

MR. JAMES O'SULLIVAN IN THE CHAIR.

### ON THE RATE OF ALCOHOLIC FERMENTATION.

BY JAMES O'SULLIVAN, F.I.C., F.C.S.

The few experiments which I purpose describing in this paper have particular reference to the rate at which alcoholic fermentation takes place. I am aware that this has already been investigated by Dumas and by Adrian Brown, who used varied strengths of solutions of dextrose and an excess of yeast in their experiments. The results arrived at by Brown corroborated those of Dumas. "That " under identical conditions of fermentation the time taken " to ferment solutions of dextrose was proportionate to the " quantity of dextrose present." Brown ("Influence of Oxygen and Concentration on Alcoholic Fermentation," Jour. Chem. Soc., 1892) showed that this rule held good for solutions of dextrose with nutrient matter up to 20 per cent., beyond which the rate was diminished.

I have most carefully made the following experiments to determine the rate of the fermentation of dextrose, and particularly that of maltose, which has not been determined, and, as the results will show, the rule laid down by Dumas and supported by Brown holds good also for maltose.

The apparatus employed in carrying out these experiments consisted of a constant temperature bath, in which the temperature did not vary from the beginning to the end of the experiment more than a quarter of a degree. All the fermentations were conducted at 22°. The bath was fitted with stirring gear, worked by small belts, so arranged that the contents of two glass cylinders placed in the bath could be kept equally agitated. A spindle reached to the bottom of each of the cylinders, and terminated in a screw-shaped fan. By means of an electric accumulator and motor the spindles were revolved. This kept the yeast in suspension, and the solutions undergoing fermentation in continuous and equal agitation.

*Influence of Agitation on Fermentation.*

I have already drawn attention to the necessity of keeping yeast in suspension during the progress of the hydrolysis of cane sugar (Jour. Chem. Soc. 1892), and with the accumulation of knowledge now to hand relative to yeast we must come to the conclusion that agitation is absolutely necessary in studying quantitatively its functions.

As the experiments I am about to describe were made with an excess of yeast, and chiefly without using nutrient matter in the solutions fermented, it is advisable for me to give an experiment to show the influence of agitation on fermentation.

Fifty grms. of maltose were dissolved in about 200 c.c. of water, and the solution was boiled for some time, in order to obtain normal rotation (this was also done where dextrose was employed); it was then made up to 250 c.c.

"*a*." 50 c.c. of this solution were made up to 100 c.c. with water.

"*b*." To 12 grms. of pressed young yeast in one of the cylinders 100 c.c. of yeast water (sp. gr. 1,005) were added, and, after well mixing, the solution "*a*" was poured into it. After a lapse of three minutes, this being taken as the start of the fermentation, a sample was drawn, the agitation going on all the while, and after shaking up with a small quantity of thick aluminium hydrate it was rapidly filtered, and the opticity of the filtrate determined.

"*c*." 100 c.c. of the maltose solution were added to a like quantity of yeast, and yeast water similarly treated. In the second cylinder, and as in "*b*," the opticity at the start was determined.

"*d*." To 6 grms. of the yeast, in a cylinder of slightly smaller diameter than that of the others, 50 c.c. of the yeast water were added, and after thoroughly mixing 50 c.c. of the maltose solution, were poured into it, and, as in "*b*" and "*c*," the opticity was determined. This cylinder was also placed in the bath.

At intervals of two hours samples were taken from "*b*" and *c*," and the amounts of maltose fermented determined by the loss in opticity, but as "*d*" was not disturbed until the seventh and ninth hour, it is only necessary to give the amount fermented in "*c*," the 10 per cent. solution, which should be compared with "*d*," also a 10 per cent.

The following are the quantities per 100 c.c. of the solutions:—

| | Grms. |
|---|---|
| *c* in 7 hours | 7·22 |
| *d* „ 7 „ | 5·11 |
| *c* „ 9 „ | 8·59 |
| *d* „ 9 „ | 5·86 |

It is almost unnecessary to mention that the yeast in "*d*" fell to the bottom of the cylinder in a very short time.

I wish to state that not more than 2 per cent. of the yeast cells which fermented these solutions were observed to make any attempt at budding, and it was only after the fourth hour that any were noticed, and observations often made after this showed that the small buds did not enlarge or separate from the parent cell. This observation applies to all the experiments described in this paper.

Experiments were made on the hydrolysis of cane sugar by yeast, and on the fermentation of malt extract, with and without agitation, in which the yeast was not in excess, and I found that in the former as in the latter case the action was accelerated by agitation.

*Rate of Fermentation of Dextrose without Nutrient Matter.*

Two experiments, "*e*" and "*f*," were made with dextrose after the manner of "*b*" and "*c*" for maltose, but water was used instead of yeast water, with the following results:—

| — | Fermented. | |
|---|---|---|
| | E. 5 per Cent. | F. 10 per Cent. |
| | Grms. | Grms. |
| 1st 2 hours | 1·62 | 1·41 |
| 2nd „ | 1·48 | 1·62 |
| 3rd „ | 1·35 | 1·41 |
| 4th „ | .. | 1·68 |
| 5th „ | .. | 1·35 |

*Rate of Fermentation of Maltose without Nutrient Matter.*

Two experiments, "*g*" and "*h*," were made with maltose after the manner of "*e*" and "*f*" and observations were made every hour, with the following results:—

| — | Grms. Fermented. | |
|---|---|---|
| | G. 5 per Cent. | H. 10 per Cent. |
| 1st hour | 0·473 | 0·374 |
| 2nd „ | 0·249 | 0·374 |
| 3rd „ | 0·523 | 0·324 |
| 4th „ | 0·386 | 0·488 |
| 5th „ | 0·386 | 0·488 |
| 6th „ | 0·374 | 0·520 |
| 7th „ | 0·370 | 0·500 |
| 8th „ | 0·530 | 0·510 |
| 9th „ | 0·530 | 0·500 |

### *Rate of Fermentation of Maltose with and without Nutrient Matter.*

"*i.*" 50 c.c. of water were added to 6 grms. of yeast, and, after mixing, 50 c.c. of a 20 per cent. solution of maltose were poured into it.

"*j.*" To the same quantity of yeast there were added 50 c.c. of yeast water, and, after mixing, 50 c.c. of the same maltose solution were poured into it. In four hours there were fermented in—

| | Grms. |
|---|---|
| "*i*" | 2·21 |
| "*j*" | 3·26 |

### *Rate of Fermentation of Dextrose with and without Nutrient Matter.*

"*k.*" To 6 grms. of yeast there were added 50 c.c. of water, and, after this was mixed, 50 c.c. of a 10 per cent. solution of dextrose.

"*l.*" To the same quantity of yeast there were added 50 c.c. of yeast water, and 50 c.c. of the same dextrose solution.

Two similar experiments, "*m*" and "*n*," were made under the same conditions, but not the same yeast.

The following are the quantities fermented in four hours in those experiments :—

| — | Grms. Fermented. | |
|---|---|---|
| Without yeast water | "*k*" 3·44 | "*m*" 4·32 |
| With yeast water | "*l*" 4·18 | "*n*" 5·00 |

### *Rate of Fermentation of Maltose compared with that of Dextrose.*

In the experiments described up to this the yeast was weighed for each one, but in those under this heading it was thought advisable to mix it in a volume of water, and to use equal volumes of the mixture.

Fifteen grms. of yeast were mixed in 125 c.c. of water, and 50 c.c. of this were employed in each experiment.

To one of the 50 c.c. of yeast mixture 50 c.c. of a 10 per cent. dextrose solution were added, and to the other 50 c.c. of a 10 per cent. maltose solution. Three pairs of fermentations, employing a different yeast for each pair, were carried out under these conditions, and the results were as shown in the table in the next column.

In the first pair, "*o*," the quantities fermented in four hours are given, but in the second, "*p*," and third, "*q*," I give the quantities fermented in intervals of two hours.

It is obvious that in all of these fermentations the quantities fermented are per 100 c.c. of the solution yeast-free, and not 100 c.c. of the mixture.

As we know that the body of a fermentable solution does not vary by being fermented, the sugar is the only varying factor in the fermentations described.

The conclusions I would draw from these experiments are :—

1. That agitation accelerates fermentation, whether it is carried on with or without an excess of yeast.

2. That the rate of fermentation of maltose and of dextrose is practically proportionate to the quantity present, approximately equal quantities being fermented in equal times.

3. That the rate of alcoholic fermentation of maltose and of dextrose differs from that of the hydrolytic action of yeast on cane sugar, as shown by me (Jour. Chem. Soc. 1892).

| — | Grms. Fermented. | |
|---|---|---|
| | Dextrose. | Maltose. |
| "*o*" in 4 hours | 3·84 | 3·41 |
| "*p*" in 1st 2 hours | 1·35 | 1·24 |
| 2nd „ | 1·62 | 1·44 |
| 3rd „ | 1·55 | 1·22 |
| In 6 hours | 4·52 | 3·90 |
| "*q*" in 1st 2 hours | 2·02 | 1·02 |
| 2nd „ | 1·84 | 1·52 |
| 3rd „ | 1·68 | 1·64 |
| In 6 hours | 5·54 | 4·18 |

In view of the fact established by Fisher that yeast contains an enzyme—beer yeast glucan—which hydrolyses maltose to dextrose, and what appears to be established by Buchner, that yeast also contains an enzyme which transforms dextrose into alcohol and carbonic acid, this difference between the rate of the hydrolytic action of yeast and of fermentation enzyme action it now appears to be reduced to is of great importance. My reason for working with maltose was that I believed it would be found that its ferment action would proceed in accordance with the rate of the hydrolytic action of yeast, but this, however, is not the case.

4. That maltose ferments at a slightly lower rate than dextrose.

5. That the presence of nutrient matter, under the conditions of the experiments, although there is no increase of yeast, yet the fermentation is accelerated. Here I wish to emphasise the fact that where nutrient matter was employed the difference between the appearance of the yeast cells and those in the solutions without nutrient matter was most noticeable. In the former case the cells remained much more transparent and healthy-looking, and a greater number showed an attempt to bud.

### DISCUSSION.

Mr. CAVEN pointed out that, according to the tabulated statement of the results of Mr. O'Sullivan's experiments, although the amount of material fermented kept decreasing, the amount fermented was about the same.

Mr. J. T. WOOD asked if Mr. O'Sullivan could draw a curve showing graphically the difference between the enzyme action and fermentative action. He also asked how the yeast was weighed.

Mr. O'SULLIVAN said, in reply, that although in two of the fermentations the amount fermented in equal intervals of time showed a decrease, this was not the rule. It was not to be expected when dealing with the action of yeast that the results would be invariable ; however, his experience with the hydrolytic action of yeast showed that a diminishing quantity in equal intervals of time was the rule. As in fermentation approximately equal quantities are fermented in an equal number of hours, the rate of fermentation would be graphically represented by a straight line, but a curve would represent its hydrolytic action. The yeast was prepared by suspending it in water, and, after allowing extraneous bodies to subside the supernatant liquid, with the yeast in suspension, was passed through three folds of a linen towel ; the yeast left in the towel was squeezed as much as possible by hand, and weighed quantities of it were employed.

## THE CAUSES OF "STENCH" IN BEER.

### (*Preliminary Note.*)

BY WILLIAM FREW, PH.D. (MUNICH).

"STENCH" in British beers may occur during the primary fermentation or after the beer has been stored for some time in cask or in bottle. This peculiar phenomenon is characterised by the development in the beer of a most disagreeable odour, resembling that of sulphuretted hydrogen or rotten eggs, and owing to this fact, the "stench" of the primary fermentation, and that showing itself during storage have, as far as I have been able to discover, been regarded as identical by all previous writers on this subject. Thus, in "The Microscope in the Brewery and Malthouse" (Matthews and Lott), the authors say, in speaking of Reess's *Saccharomyces Ellipsoideus* (p. 49): "The smaller form of *S. Ellipsoideus*, the one which more particularly concerns us, is not infrequently found associated with cloudy frets and sickness, and in such cases may grow pretty freely. It is probable that beers brewed from very hard waters are more open to this form of secondary fermentation than others. With the first-mentioned ales a decided stench often comes on, owing to the production of sulphuretted hydrogen, and from this traces of sulphur alcohols, a strongly unpleasant smelling class of substances. These effects wear off, however, and a beer which has been through a very bad *Ellipsoideus* fret may, if otherwise sound, become quite palatable. *S. Ellipsoideus* is frequently met with in bottled ales and forced samples, but is not always accompanied by smell, but so far as our experience goes, a bad stench is generally accompanied by *Ellipsoideus*." Their latest statements on this subject are to be found (so far as I know) in an article on "The Chief Causes of Irregular Fermentations" (Boakes Diary for 1894). There they say: "The typical irregularity in smell, excluding minor variations, is the 'stenchy' or sulphuretted hydrogen fermentation which comes on, as a rule, quite unexpectedly. The presence of the evil-smelling gas is easily verified by exposing strips of blotting paper dipped into a solution of acetate of lead to the gas coming off from the fermentation . . . . . Unpleasant and alarming as this phenomenon is, it is very seldom that the finished beer shows any signs of the peculiar phase of fermentation through which the wort has passed." Again: "iv. Stenchy Fermentations. Possible causes—Sulphured hops, impure yeast, excessive use of sulphites or bisulphites, presence of wild yeasts."

During the course of an investigation into the causes of stench in beers, which has already occupied me for a considerable time, and which, I hope, will be finished in the autumn of the present year, I have been able to prove that the two varieties of stench are perfectly distinct in their nature and cause. That occurring during the primary fermentation is due to the evolution of sulphuretted hydrogen caused by the reduction of some of the sulphur compounds in the wort. This may take place when only culture yeasts are present; it does not require the presence of wild forms to cause it. It is evidently due to some peculiar condition of the yeast cells, which causes the reduction of some of the sulphur compounds present. In this connection interest attaches to certain experiments on the action of animal tissues and of yeast on sulphur, made by J. de Rey-Pailhade (Compt. rend. 108, pp. 356—357, and 118, pp. 201—203. Compt. rend. Soc. biol. 1893, pp. 46—47). Sulphuretted hydrogen was given off when finely divided sulphur was suspended in alcohol along with certain animal tissues; when sulphur was suspended in water along with a large quantity of yeast; and when an alcoholic extract of yeast was allowed to act on finely divided sulphur.

The production of sulphuretted hydrogen is also said to be observed in the case of beers to which salts of sulphurous acid have been added as antiseptics. It is, therefore, probable that this phenomenon may occur more frequently in breweries where a water is used, containing large quantities of sulphates in solution. This variety of stench can hardly be called a disease, it is more of the nature of an irregularity, and no permanent harm (as far as I know) is done to the beer.

The second variety of stench is of quite a different nature, and occurs at a far later stage. After the beer has been stored for a longer or shorter time in cask or bottle, it developes a very unpleasant smell, resembling that of sulphuretted hydrogen to some extent, but being more akin to that of rotting organic matter; in fact, a beer seriously attacked in this way has, more or less, a putrid smell. This, the *real* Burton stench, comes on after the primary fermentation is finished, and during, or after, the secondary fermentation in cask or bottle; it is usually accompanied, as far as my experience goes, by a "fret" and by turbidity. The peculiar smell may become modified by longer storage, but the beer is never so good afterwards in taste or smell.

In the course of my investigation I found that the deposit of yeast from forced samples of beer showing this stench always contained quantities of a small yeast resembling the Ellipsoideus form described by Reess and by Matthews and Lott (*vide supra*). In no case where the deposit contained apparently only culture cells was there even a trace of stench. It was possible to have a beer whose deposit showed a considerable proportion of stench forms, and which did not exhibit a stenchy odour; in all such cases, however, the odour of the beer was not good, it was distinctly sickly.

I found next that the disease could be produced in sound beers by inoculating them with a trace of the deposit from a stinking beer, and that it was accompanied by a large development of the aforesaid small yeast. Within the last few months I have been able to isolate from the deposit of a stinking beer at least one variety of yeast capable of producing "stench" when inoculated into a sound and sterile beer.

Briefly summarised, the results of my work up to the present time are as follows:—

1. The real "Burton Stench" is caused by the presence and growth of one or more varieties of small wild yeasts in the beer affected, one of which I have been able to isolate in a state of purity. The disease is most apparent (or, probably, only occurs) during the after-fermentation, and is the result of an infection of the beer by the wild yeasts previously mentioned. The most dangerous time for infection is towards the end of the primary fermentation (at racking) or afterwards.

2. As far as my observations go, the peculiar odour of beers attacked by this disease is not due to sulphuretted hydrogen or to any sulphur compound, but is probably caused by the presence of certain fatty acids or higher alcohols produced by the stench yeasts. Any experiments I have made go to show that beers brewed from waters containing little or no sulphates afford a more suitable field for the development of the organism and the disease it produces than those brewed from waters containing large quantities of sulphates. The reasons why hard-water beers have been credited with a peculiar tendency towards this disease are (1) the belief that the evolution of sulphuretted hydrogen during the primary fermentation, which certainly may occur more frequently in worts prepared with highly sulphated waters, is only another phase of this disease,* and (2) that, as a general rule, only beers brewed from waters containing sulphates are stored long enough for the disease to show itself. To show how widespread this disease is, I may state that I have observed it in pale ales brewed in Scotland and England (Burton), in Scotch and Irish stouts, and in a bottled lager beer from Australia. In all those cases the deposited yeast contained the characteristic "stench" forms, and by inoculation from these deposits the disease could be produced, with accompanying growth of the "stench" yeasts, in sound sterile beers.

The accompanying drawing shows a characteristic deposit from a "stinking" beer. The cells may vary in size from 3—7$\mu$ in length and 2—4, 5$\mu$ in as breadth, compared with culture (top) yeasts of from 8—11$\mu$ in length. Like all other yeasts, however, their form varies so much with varying conditions of growth that no reliance can be placed on mere microscopic appearance.

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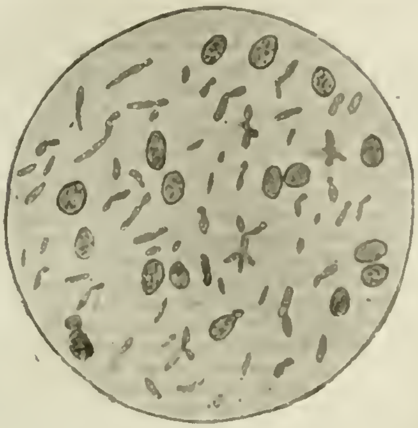
* To avoid confusion the term "stench" will in future be used only to designate the disease, not the evolution of sulphuretted hydrogen during the primary fermentation.



A detailed account of my work on the subject of "stench" and of the particular form of wild yeast which I have succeeded in isolating, will be published in the end of this year. The present note is published in order to secure priority, and also for the sake of obtaining the practical experience of any brewers who may have had this disease brought under their notice. I should feel much indebted to any brewer who may have had experience of this trouble and who would be good enough to send me any practical observations he may have made. Samples of beers affected by this disease would also be most welcome. I need hardly say that, of course, all such communications would be regarded as strictly confidential by me. Such information would very much increase the value of my paper in the end of the year from a practical point of view.

It is proposed to call this variety of "stench" yeast Saccharomyces foetidus I.

## Scottish Section.

### THE EDUCATION AND TRAINING OF BREWERS.

BY WM. FREW, PH.D. (MUNICH).

(*This Journal*, 1898, 222—227.)

ERRATUM.

On p. 225, 1st col., and last line, the word "served" should be followed by the words "in a brewery."

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## I.—PLANT, APPARATUS, AND MACHINERY.

*Raising Liquids, A New Apparatus for.* G. Trouvé. Comptes Rend. 1898, **126**, [15], 1097—1099.

The apparatus consists of a frustum of a cone which is rapidly rotated on its axis within a fixed body of the same shape. The smaller end of this body serves as the inlet, and a tangentially disposed opening at the wider end, as the outlet for the liquid. The apparatus works through centrifugal action, and the gyratory motion which is imparted to the stream of liquid. Its output increases with the square of the velocity, and hence rapid rotation of the axis affords large outputs and high pressure of the outflowing liquid. The addition of a screw to the interior surface of the cone increases the pressure, but does not affect the output. The apparatus would probably serve as a propeller similar to a ship's screw, as well as for raising liquids.
—J. A. B.

*Explosion of Cylinders containing Liquefied Ammonia; The Prevention of.* A. Lange. Chem. Industrie, 1898, **21**, [10], 191—198.

On the 17th of September 1897, a cylinder containing liquefied ammonia exploded at the North German Refrigerating Works in Rummelsburg, near Berlin. The valve of the cylinder was closed at the time, and the cause of the explosion was traced to the great rise of pressure which took place whilst the cylinder was being heated in a vessel containing hot water. This led the author to make some experiments on the pressure produced in cylinders containing liquefied ammonia, and the limiting temperature to which they may safely be exposed. The conclusions which he has arrived at are as follows:—

(1.) The presence of air in commercial liquefied ammonia does not exercise any great influence on the safety from explosion of the containing cylinders.

(2.) On heating liquefied ammonia in cylinders, the pressure rises gradually so long as the cylinders are not completely filled with the liquid. On further heating, there is then a rapid increase of pressure, which becomes gradually slower again as the temperature rises.

(3.) Cylinders containing liquefied ammonia, and which comply with the regulations of the German railways, can be safely heated to 65° C. At this temperature the pressure is only two-thirds of that at which they are tested. Provided that there are no flaws in the cylinders themselves there is no danger of them bursting until the temperature exceeds 68° C.—J. S.

### PATENTS.

*Distilling and Rectifying Processes, Impts. in, and in Apparatus therefor.* E. Guillaume, Paris. Eng. Pat. 4902, Feb. 23, 1897.

In this invention various improvements are introduced, both in the processes for effecting continuous distillation and rectification, and in apparatus therefor. There are 23 claims, for details of which, and also for the illustrations in connection with them, the original must be consulted.—A. S.

*Evaporating, Concentrating, Distilling, and Condensing Liquids; Impts. in and relating to Apparatus Applicable to.* J. Foster, Pollokshields, Glasgow. Eng. Pat. 8158, March 30, 1897.

The improvements are shown applied to an evaporator working under multiple effect. An upper horizontal feed pipe A is connected by short vertical tubes $A^1$ to a number of distributing chambers $A^2$, having each a central raised or conical part $a$, and short pipes or ducts $b^1$ leading from the lower parts to two rows of vertical evaporating tubes $b$, below. These preferably consist of film evaporating tubes as described in specification to Eng. Pat. 3118 of 1895. (See this Journal, 1896, 185.) A chamber, or vapour-escaping space, $B^2$, is formed between the top tube plate and the cover of the vessel forming the bottom of the liquor-distributing chambers, while a second separate chamber C is bolted to the vessel below the bottom tube plate. Both of these chambers are in connection, under vacuum, with the next evaporator or a condenser, the former by a branch connection $c$ past a vertical baffle plate $c^1$ in a "save all" vessel to the outlet $C^3$, and the latter by a branch $C^2$ to the same outlet. The concentrated liquor escapes by an outlet $C^1$, while steam enters at the inlet $B^5$.

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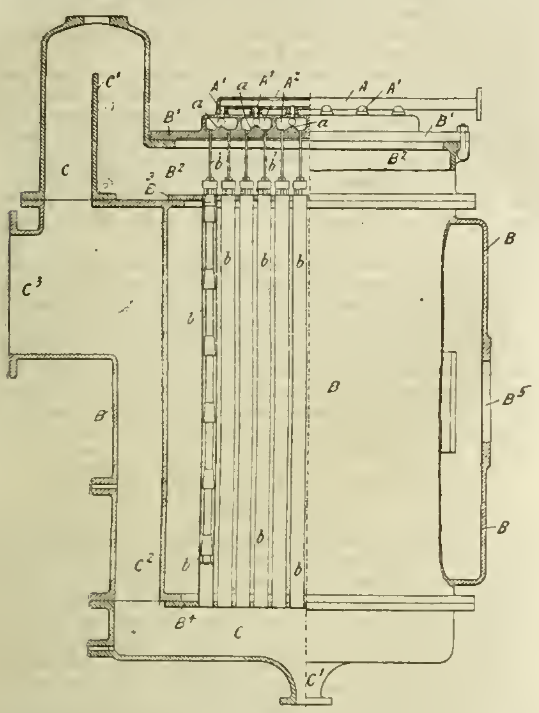
* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

—R. S.



*Drying Granular and Pulverulent Materials, An Improved Method of and Means for.* H. McPhail, Wakefield. Eng. Pat. 11,449, May 7, 1897.

An apparatus for drying materials such as grain, malt-seeds, coffee, tea, and the like by superheated steam, consists of an upright steam-jacketed cylinder with a contracted lower end having a lateral passage near the top for admitting the granular material.

The superheated steam is admitted at the base, and, after passing through the material, escapes past a perforated partition to the outlet pipe near the top. A suitable exit opening is provided at the bottom of the cylinder, and is closed by a suitable valve, as are also all other openings.

—E. G. C.

*Evaporating or Drying Viscous Substances, Semi-Liquids, and Pasty Materials; An Improved Method of and Means for.* H. McPhail, Wakefield. Eng. Pat. 11,450, May 7, 1897.

Relates to an apparatus for drying substances such as molasses, sugar, syrup, glycerin, tar, alkalis, and the like. It consists of a boiling pan provided with perforated heating pipes, which latter are arranged in the form of a cross near the bottom and fed with superheated steam. Openings are provided in the bottoms of the pans to allow of the withdrawal of the material after treatment, and a number of these pans may be placed in an apparatus of the type described with reference to Eng. Pat. 11,448 of 1897 (see page 574), or alternatively in Eng. Pat. 11,449 (preceding abstract) of even date therewith.—E. G. C.

*Drying Wood and other Vegetable Substances, An Improved Method of and Means for.* H. McPhail, Wakefield. Eng. Pat. 3690, Feb. 14, 1898.

A steam-jacketed cylinder, preferably horizontal, has one end closed by a removable cover, so that a truck or carriage holding the wood, roots, fruits, or the like substances to be treated, can be run into the chamber and there subjected to the drying action of superheated steam.

The chamber may also be provided with shelves upon which the substances can be laid, and the truck is preferably of the type described with reference to Eng. Pat. 11,448 of May 7, 1897, page 574.—E. G. C.

*Filter Presses and Similar Filtering Apparatus, Impts. in.* J. Williamson, Glasgow. Eng. Pat. 11,803, May 12, 1897.

Fig. 1.

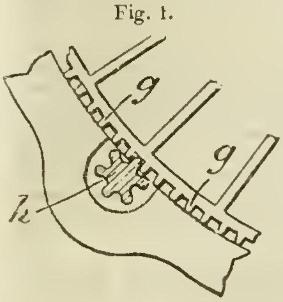
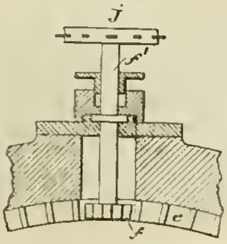


Fig. 2.



The invention relates to devices for altering the positions of the frames of a press, or the gratings in those frames, so that fresh areas of filtering cloth shall be presented to the liquid under treatment, and the life of the filtering medium lengthened. The invention is stated to be particularly applicable to filtering apparatus, in which practically continuous filtration is obtained by periodical brushing of the exposed surfaces of the filtering medium.

In plates of circular form, secured upon a central shaft, the rim, $g$, of the grating (Fig. 1) may be toothed to gear with an actuating pinion, $h$, running the whole length of the filter, or adjacent pairs of gratings $e$ (Fig. 2) may be moved in opposite directions by pinions, $f$, placed between them, and operated independently by hand wheels, $j$, or together by auxiliary gear.

Plates of rectangular form are provided with pinions, in a similar manner to the arrangements described above, or alternatively both forms may be operated by levers.

—E. G. C.

*Filtering or Absorbent Material, A Process for the Manufacture of.* W. Leuscher, Teutschenthal, Halle on S., Germany. Eng. Pat. 27,121, Nov. 19, 1897.

A plastic mixture is made of clay, porcelain earth, stony material, asbestos, and sawdust, or other organic and combustible material. This is then shaped, dried, and incinerated, or lime or cement is substituted for porcelain or stoneware and sawdust, in which case the mass does not require to be incinerated.—H. H. B. S.

*Reverberatory Furnace, with a New System of Retorts for Calcining Divers Products.* H. Mawhood, Antwerp. Eng. Pat. 14,778, June 18, 1897.

Reverberatory retort furnaces, in which the retorts are arranged in flues made of fireproof flags, and in these flues the combustion products circulate, so that the retorts are not heated directly by the flames. This results, as stated, in a longer life for the retorts, in a more regular calcination, and in economy of fuel.—R. S.

*Artificial Ice, Impts. in and in Apparatus for the Manufacture of.* A. M. Clark. Communicated by A. M. L. Osmund, Paris, France. Eng. Pat. 15,523, June 29, 1897.

Artificial ice is manufactured by the employment of metallic freezing surfaces disposed within a suitable container. The surfaces are covered on one side by the water to be frozen, and exposed on the other side to a refrigerating agent, preferably air, and when a sufficient thickness of ice has formed, a heating agent is introduced on this latter side to detach the ice shell from its freezing surface.

For the production of solid blocks of ice, the shells produced in this manner are filled with water, and exposed to the action of the refrigerating agent in a suitable chamber, while, in order to prevent the bursting of the shell during the process, a compressible object, such as a pig's bladder, is immersed in the water to be frozen, thereby leaving a sufficient space to allow of expansion as the freezing progresses.—E. G. C.

*Raising and Forcing Liquids by Means of Compressed Air or Gases; A New or Improved Method and Apparatus for.* J. G. Lorrain, London. Communicated by The Ingersoll-Sergeant Drill Co., New York, U.S.A. Eng. Pat. 836, Jan. 11, 1898.

The apparatus consists of a valveless delivery pipe surrounded by a submerged pressure chamber, the lower end of which is permanently open and immersed in the liquid. Air under pressure is supplied to the pressure chamber, thereby depressing the liquid column until the lower open end of the delivery pipe is exposed, when a rush of air takes place and the water column rises. In this manner the air and water are caused to pulsate about the lower end of the delivery pipe, and alternate masses of each will enter the pipe and be forced to the surface.—E. G. C.

*Detergent, An Improved; applicable also for Preventing Incrustation in Boilers, for Preventing Rust, and for similar Purposes.* E. J. Mills, Glasgow. Eng. Pat. 4762, Feb. 26, 1898.

The detergent paste is made by dissolving 24 parts of caustic soda and 8 parts of caustic potash in 20 parts of water, then stirring in 36 parts of slaked lime and adding 8 parts of rosin oil. The proportions may be varied. For cleansing purposes, the paste, mixed with water, is applied by means of a cloth or brush with friction. To prevent boiler scale, a little of the diluted paste is put into the boiler. To remove paint, resin, grease, tar, or varnish from a surface, the paste is spread in a thin layer and worked with a wire brush. The paste, applied in a thin layer, is also useful as an anti-rust.—E. S.

*Metallic Capsules for Storing Compressed or Liquefied Gases, Impts. in.* J. M. Thèvenet-Le-Boul, Paris, France. Eng. Pat. 7590, March 29, 1898.

The capsule is formed of two similar metal tubes, closed at one end and open at the other, and of such diameters that one tube can slide within the other. An obturating washer, having neither odour nor taste, is placed in the closed end of the larger tube, and the open end of the smaller tube is afterwards inserted. The capsule is now placed in a closed filling vessel having a movable cover, the joint being made by a hollow rubber ring. Gas is forced into this vessel, passing into the capsule by the capillary space between the tubes. As soon as the filling operation is completed, the removable cover is pressed down, so that the edge of the inner tube is forced into the obturating washer, and an effectual closing of the capsule is obtained.

In a modified arrangement there is a second obturating washer placed upon a shoulder near the closed end of the inner tube.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

[*Petroleum.*] *Burning Oil: Influence of Paraffin Content on Illuminating Power.* R. Kissling. Chem. Zeit. **22**, [21], 223—225.

Representations have lately been made to the German Chancellor that numerous grades of American petroleum, although passing the 21° C. Abel test prescribed by law, are both deficient in illuminating power and dangerous by reason of their explosibility. Consequently, it is said to be desirable to fix a standard candle-power for these oils, because "insufficient illuminating power or too high a solidifying point may result in damage to sight, and, under certain circumstances, be the cause of accidents."

Commenting on this statement the author mentions that there is no known connection between illuminating power and explosibility in petroleum, nor is there any apparent support for the alleged existence of any risk of explosion beyond that revealed by the Abel test.

Concerning a minimum limit of candle-power, this is a difficult point on which to legislate, owing to the unreliability of photometric tests—a circumstance due to the impossibility of obtaining absolute uniformity in lamp wicks. The only accurate measure available is by fractional distillation in the manner prescribed by the author (Chem. Zeit. **19**, 778), but this is too complicated to be fit for general use as a companion to the Abel flash-point apparatus.

With regard to the influence on sight of the height of the setting point (paraffin content) of the oil, no connection appears to be traceable, the quantity of paraffin present being so slight as to have practically no effect on the burning quality of the oil. Besides, experience has shown that the addition of spermaceti or paraffin increases the illuminating power of mineral oils; only in the exceptional cases when the weather is so cold as to cause a deposition of paraffin in the oil, can there be any deterioration of its lighting powers.—C. S.

*Calcium Carbide.* J. Sieber. Chem. Zeit. 1898, **22**, [31], 308.

The published yields of calcium carbide on the manufacturing scale vary from 2 to 15 kilos. per electrical horse-power per 24 hours. The figures over 10 kilos. must be received with great suspicion, and probably refer to a product very far from pure. The thermal figures calculated from the equation $CaO + 3C = CaC_2 + CO$ give a possible yield of 9·38 kilos., assuming that there is no evolution of heat in the formation or decomposition of calcium carbide itself (a question which appears not to have been solved yet, though there should be no difficulty in arriving at the answer). If we assume that the whole of the heat needed to raise the materials to the temperature of the reaction (say 3,000° C.) is wasted, then the yield can only reach about 3·44 kilos. per electrical horse-power per 24 hours, and the actual yield will therefore vary between 3·44 and 9·38 kilos., as this heat is less or more perfectly economised. As a pound of coal used first to generate steam and then to produce electric current will only furnish in the electrical furnace about 11 per cent. of its theoretical calorific power, the author suggests that it would probably be economical to have a preliminary heating of the materials carried out directly, though of course the electric heating cannot be dispensed with for the final attainment of the requisite temperature. He also draws attention to the calorific value of the carbon monoxide which is produced in the reaction.—J. T. D.

*Water-Gas and its Applications.* V. B. Lewes. J. Soc. Arts, 1898, **46**, [2373], 583—591.

Ibbetson in 1824 attempted to increase the volume of gas from a charge of coal by passing steam through the hot coke in gas retorts after the completion of carbonisation, but the temperature of the coke was too low for the production of water-gas of good quality. The large proportion of carbonic acid in Ibbetson's water-gas ruined the illuminating power of the coal-gas with which it was mixed. In 1849, Gillard raised fuel to incandescence by an air blast, and at intervals passed steam through the fuel, and thus obtained water-gas. He introduced the "blow and run" method of making water-gas, as distinguished from earlier methods in which the carbonaceous matter was contained in a retort heated by external firing. In 1875, Lowe and Tessié du Motay inaugurated systems of making carburetted water-gas in America. Water-gas produced in cupola furnaces on the "blow and run" principle was used for welding, &c. at works at Essen, and was introduced at the Leeds Forge in 1888. Economic conditions, which will now be considered, prevented the spread of pure water-gas, while carburetted water-gas was adopted to such an extent that it now forms one-fifth of the gas supply of London north of the Thames.

In order to make on the "blow and run" system, 1,000 cb. ft. of water-gas, containing a little over 15 lb. of carbon, 4,000 cb. ft. of producer-gas, containing over 44 lb. of carbon, must first be made. Therefore, about 60 lb. of carbon are expended in producing 1,000 cb. ft. of water-gas, and practically it has been found that 1 ton of coke yields on an average 34,000 cb. ft. of water-gas. The combustibles in 1,000 cb. ft. of the gas are 2·75 lb. of hydrogen and 30 lb. of carbon monoxide, and their thermal value is 155,600 thermal units, whereas the thermal value of the 60 lb. of carbon used in the manufacture of the gas is 484,800 thermal units. The manufacture therefore entails a loss of over 65 per cent. of the calorific value of the fuel, and this loss has prevented the general use of water-gas. Where, however, the producer gas can be utilised while hot, another 223,328 thermal units will be secured, and the calorific values of the water-gas and of the producer-gas will amount to 80 per cent. of that of the fuel used. Intermittent production and the large volume of producer-gas have, however, prevented the general use of pure water-gas as fuel. In carburetted water-gas plant, however, the producer-gas is applied to the heating of chambers in which oil is decomposed, and the oil-gas obtained is used to render the water-gas luminous. The practical loss of heat is thus reduced to a minimum.

In the ordinary water-gas process, the fuel is raised to incandescence by blowing air through so deep a bed of fuel that the chief products are carbon monoxide and nitrogen. One pound of carbon develops only 2,400 heat-units when burnt to carbon monoxide, but 8,080 when burnt to carbon dioxide. Carl Dellwik has devised a process by which the products of the blow are carbon dioxide and nitrogen, and thus the additional heat-units are made available in raising the temperature of the bed of fuel in place of passing into the producer-gas. The result is that twice as much water-gas is obtained per pound of fuel, and the extra heat generated reduces the period of blow, and allows the run to be prolonged. By the old process, "blows" of nearly 10 minutes' duration were necessary to raise the fuel to the proper temperature for a "run" or make of four to five minutes' duration. By the Dellwik process, "blows" of two minutes and "runs" of seven to ten minutes are possible. The average percentage of carbon dioxide in the water-gas produced in a series of runs with the Dellwik plant was 4·58, which is quite as low as is common in water-gas made by the old process. The first products of the decomposition of steam by carbon are carbon monoxide and hydrogen, but if the fuel is cooled so far that only a portion of the steam is decomposed, the excess of steam oxidises the carbon monoxide to carbon dioxide. In the Dellwik process the flow of steam to the fuel is regulated so that even at the end of a run there is no excess of steam to oxidise the carbon monoxide to dioxide. Combustion during the period of blowing in the Dellwik plant is shown to have been complete by two analyses of the products given off during a blow. These show 17·9 and 18·8 per cent. of carbon dioxide, and only 1·8 and 1·0 per cent. of carbon monoxide.

The Dellwik process makes available in the water-gas about 82 per cent. of the calorific value of the coke consumed in both generator and boiler. A ton of good coke yields 70,000 cb. ft. of water-gas, in place of 34,400 by the old process. Under the old process the cost of 1,000 cb. ft. of water gas was from 4*d.* to 6*d.*; by a large Dellwik installation it should be below 3*d.*; the items being approximately:—coke, at 9*s.* per ton, 1·50*d.*; gas maker, 0·23*d.*; engine driver and boiler man, 0·23*d.*; coke man. 0·17*d.*; purifying, 0·15*d.*; wear and tear, 0·10*d.*; interest, 0·20*d.*; water, 0·10*d.*; and oil, waste, &c., 0·07*d.*; making a total of 2·75*d.* Water-gas may safely be assumed therefore to be obtainable in the holder at 3*d.* per 1,000 cb. ft., whereas coal-gas costs 1*s.* to 1*s.* 3*d.* For use for illuminating purposes with the incandescent mantle, water-gas must be freed from a gaseous compound of carbon monoxide and iron, which, during combustion, deposits oxide of iron on the mantle. Though water-gas has only half the thermal value of the same volume of coal-gas, it develops a higher illuminating power, because it gives a non-luminous flame in an Argand burner without previous admixture of air, and an intense combustion takes place on the surface of the mantle where the gas and air meet.

The mantles for water-gas are made of the Auer mixture, but are of larger and thicker mesh than ordinary mantles. 7 cb. ft. of water-gas at eight-tenths pressure gave a light of 137 candles, or 19·8 candles per cubic foot, at a price which other means could not equal. A larger field for water-gas, however, is in the development of power by gas engines. A consumption of 44 cb. ft. develops one brake horse-power per hour, at a cost of 0·12*d*, and the adaptation of the gas engine to the use of water-gas is expected to reduce this consumption by 40 per cent.

Carburetted water-gas is largely used as an enricher of coal-gas, and its employment has only been checked by alarm at the poisonous properties of the carbon monoxide which it contains. There is practically no danger when more than its own volume of coal-gas is mixed with it. Three analyses of carburetted water-gas of 22 to 23 candle-power show an average of 29·03 per cent. of carbon monoxide in the gas, while coal-gas from Durham coal contains only about 4 per cent. of carbon monoxide.

Haldane breathed air containing 0·05 per cent. of carbon monoxide for four hours without experiencing marked symptoms of poisoning, and this percentage may be taken as the margin of safety. The air of a bedroom of 1,000 cb. ft. air-space is probably changed twice in the hour, and if gas escape from a tap at the rate of 6 cb. ft. per hour, the percentage of gas in the air will in three to four hours rise to 0·3 per cent., and then, owing to the changing of the air, remain constant. The proportion of carbon monoxide in the gas may therefore be one-sixth before the air of the room will contain over 0·05 per cent. of that constituent, and such a proportion is arrived at by a mixture of coal-gas and carburetted water-gas in about equal volumes. These figures refer to coal-gas from Durham coal such as is used in London; Edinburgh gas, on the other hand, has an initial proportion of 10 to 11 per cent. of carbon monoxide. The mixture of gas distributed should not contain more than 16 per cent. of carbon monoxide. The percentage of that constituent in the gas supplied to London north of the Thames during the past few years has varied from 7 to 10, and once was as high as 12.—J. A. B.

### [*Gas*] *Purifying Materials, The Relative Activity of Bog Oxide and Weldon Mud as.* J. Gas Lighting, 1898, **71**, [1823], 877—879.

One hundred parts of bog oxide containing 50·34 per cent. of anhydrous ferric oxide, would be completely fouled by 32·09 parts of sulphuretted hydrogen; 100 parts of Weldon mud containing 34·0 per cent. of manganese dioxide by 26·6 parts. In the case of oxide, 290 cb. ft., and in the case of Weldon mud, 240 cb. ft., of purified gas should be delivered per pound of purifying material fouled. If these results are not attained in practice, the active ingredient may be suspected of inertness on account of its physical state, and means of altering the latter may advantageously be reviewed.

The avidity of each material for sulphuretted hydrogen is indicated by the thermic effects attendant on fouling. 34,386 calories in the case of oxide, and 22,467 calories in the case of Weldon mud, are liberated on the fouling, and the fouled materials attain temperatures of 155° F. and 132° F. respectively, provided evaporation of water has not occurred. The heat liberated would, however, be nearly the same in both cases if the materials contained the same proportion of their active ingredients. Weldon mud contains more water than does oxide, and hence should not exhibit so great a rise in temperature on fouling. In the revivification of the completely fouled materials, oxide liberates 25,664 calories and Weldon mud 27,321 calories, and temperatures of 603°F. and 640° F. respectively would be attained by the materials if evaporation of water did not occur. For a given proportion of active ingredient the heat liberated on revivification by Weldon mud is, however, nearly twice that which would be liberated by oxide. The fact that bog oxide usually contains a greater amount of ferric oxide than the amount of manganese dioxide in Weldon mud obscures the fact that the energy of revivification is greater with Weldon mud than with oxide, as does also the greater proportion of water in Weldon mud. The presence, formation, and evaporation of water are the factors which control the temperature of oxide and Weldon mud during fouling and revivification. The amount of water present may be even less than 18 per cent. of the oxide or 40 per cent. of the Weldon mud, and the temperature attained during fouling will not be too high, provided air is excluded from the purifiers. But fouled oxide should contain not less than 25 per cent., and fouled Weldon mud not less than 42 per cent. of water, if the temperature attained during revivification is to be kept within safe limits. After the first revivification the water present may be reduced by one part for every five parts of free sulphur in the material. The amount of water should not greatly exceed the proportions stated, or the revivification will be sluggish. Weldon mud will revivify satisfactorily when it contains an amount of free sulphur which would render oxide useless.

The figures given above are based on theoretical considerations. Practically the full theoretical efficiency of Weldon mud is attainable in actual use, but this is not generally the case with oxide, because particles of the ferric oxide are physically inaccessible to sulphuretted hydrogen. Calcined ferric oxide, even though finely ground, is useless for gas purification. Natural Irish bog ore is the best ferric oxide in common use, and owes its superiority to its light porous structure. Nevertheless, its theoretical duty is not achieved in practice, since some particles of ferric oxide in it are not attacked by sulphuretted hydrogen. Ferric hydrate may be prepared artificially, and has a higher efficiency as a purifying agent than bog oxide. A suitable artificial ferric hydrate is not produced as a by-product in any industry, but Weldon mud is a by-product, and owes its great efficiency chiefly to its physical state. Under conceivable circumstances the production of artificial ferric hydrate suitable for the purification of gas might be remunerative.—J. A. B.

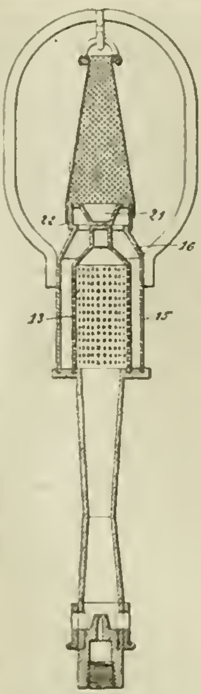
*Coal Analysis; Preliminary Report of the Committee of the American Chem. Soc. on.* J. Amer. Chem. Soc. 1898, **20**, [4], 281.

*See under* XXIII., *page* 608.

## PATENTS.

*Incandescent Gas Burners, Impts. in.* O. Kern, Paris. Eng. Pat. 291, Jan. 5, 1897.

This burner is to be used with gas at the ordinary pressure, and without any chimney or artificial draught. It is of the Bunsen type, having the ordinary gas inlet and nozzle screwed into the Bunsen pipe, with air inlets and throttle ring as usual. The "distinguishing feature" of the pipe is that it consists of two tubular truncated cones, joined at their smaller ends, the lower cone being shorter than the upper. The angle of divergence of the upper cone should be from 5° to 7° C., and certainly not more than 10° C. The angle of convergence of the lower cone may vary more widely, but preferably does not exceed 10°. The length of the lower cone must be such as to permit the air and gas to be well mixed before they reach the point of junction with the expanding cone, and the upper cone should be nearly twice as long as the lower. Thus, in a burner consuming 80 litres per hour, the section of the pipe at the junction of the cones should be 7 mm., the length of the lower cone 3 cm., and that of the upper cone 5—6 cm. For the more perfect mixing of the gas and air, the pipe is surmounted by two concentric cylinders, 15 and 13, the latter being closed at the top and perforated laterally with small holes which preferably flare inwards. When the air inlets are properly adjusted, the burner produces a gaseous mixture in which the gas and air are so proportioned that it can burn completely without the flame deriving a further quantity of oxygen from the air, an intensely hot flame resulting. Upon the top of the cylinder 13 there is fixed a conically flaring cup, 21, while upon the cap 16 is fixed a cylindrical tube, 22, the upper edges of these forming an annular nozzle from which the gas mixture issues in such direction that, when ignited, the outer surface of the inner zone of the flame will be nearly vertical. The mantle is supported over the burner as shown, by a copper loop which conveys heat to the cylinder 15 and so pre-heats the combustible mixture. A duplex burner constructed on the above principles, and alternative constructions of parts, are described. There are six claims.—H. B.



*Incandescing Media for Lighting Purposes, Impts. in the Manufacture of.* R. Langhans, Berlin. Eng. Pat. 13,899, June 5, 1897.

Reference is made to Eng. Pat. 14,845 of 1896, and 501 of 1897 (this Journal, 1897, 601, and 1898, 37). The patentee believes that the decrease in light emission which incandescence bodies suffer after a certain period of use is due to the "fluctuating oxide" (*e.g.*, ceria, oxide of vanadium—the oxygen carriers) becoming indifferent by formation of an intermediate compound or complex oxide, which is incapable of giving off and reabsorbing oxygen in the flame. He aims at producing a vanadium compound which will act as an oxygen carrier, and will be incapable of forming such indifferent compounds. In the case of vanadiferous zirconia, for instance, enough calcium oxide is employed to form zirconate and vanadate of calcium. "The percentage of earth-alkali oxide must be up to the limit of the 'binding' between that oxide and the zirconia or thoria—*i.e.*, up to the formation of a neutral zirconate or thorate R $(ZrThO_3)$, R 1 (CaSrBa)O—but must not surpass that limit." According to one method, to a vanadiferous solution of zirconium nitrate or (and) thorium nitrate is added the required amount of alkaline earth, as hydroxide, in small quantities while the liquid is stirred, and the hydrate of zirconium or (and) thorium produced, readily redissolves. For impregnating a fabric, this fluid should contain about 30 per cent. of dry salts, and an example of the proportions is: zirconium nitrate, 68·2 per cent; strontium hydroxide, 30·0; vanadium pentoxide, 0·12; nitrate of alkali, 1·68 per cent. Instead of the hydroxide, the carbonate or formate of strontium may be used. Instead of using the alkaline earth as hydroxide, carbonate, &c., it may be used as nitrate, the formation of zirconium hydrate being effected by adding ammonia, aniline, &c. According to another method the nitrate of the alkaline earth is dissolved in vanadium pentoxide solution, and to this is added oxychloride of zirconium or (and) thorium. Here it is "the elimination of chlorine from . . . the oxychloride in the calcining step which enables the formation of the alkali-earth zirconate to take place." An example given is: zirconium oxychloride $(Zr_3O_4Cl_4)$, 62 per cent.; calcium nitrate (dry), 36 per cent.; vanadium pentoxide, 0·12; nitrate of alkali, 1·88 per cent.

Instead of vanadium pentoxide or vanadyl salts there may be used vanadic acids, vanadates, &c., and the complex acids, "such as phosphor-, niob-, bismuth-, and chrom-vanadium acids." There are nine claims for processes and substances.—H. B.

*Apparatus for Carburetting Air: Impts. in or relating to.* A. J. Boult, London. From A. Lefebvre, Paris. Eng. Pat. 10,261, April 24, 1897.

The carburetter is an oblong metal box divided horizontally into compartments by partitions fixed alternately at the opposite ends. The lowest compartment is provided with a tube through which air is forced, and with a tube leading from the reservoir of liquid hydrocarbon. On the latter tube is a special float valve for indicating the amount of liquid in the carburetter.

A modification of the carburetter is shown applied to a motor-cycle or other road vehicle, the vehicle having also an air pump, a compressed air reservoir, a motor, a lamp, valves, and connections. The motor is driven and the lamp lit by the carburetted air.—R. S.

*Petroleum or other Inflammable Liquids for Illuminating Purposes; Impts. in the Means or Apparatus for Supplying and Burning.* J. B. I. Julhe, Paris, France. Eng. Pat. 14,594, June 16, 1897. (Date under Inter. Convention, March 24, 1897.)

Relates to arrangements for supplying and burning petroleum after the manner of gas, that is, through a system of pipes connected with burners placed at the desired points. The oil is contained in a reservoir (which may be in an elevated position), the pipe union at its lower part having a filtering plug of wool or wadding. The pipes are of very small section. Each burner is provided with two needle regulating valves, one a little distance off, the other immediately beneath the burner, while between the two is fixed a short pipe forming an air chamber, into which some petroleum passes when the light is turned out. The burner is annular, and is connected by two tubes to the supply pipe just above the last regulator. Within this annular burner is placed a tightly fitting wick, formed of tightly coiled cotton impregnated with silicate of potash, then dried at 150° C., and finally calcined so as to destroy the organic matter to about the depth of 1 mm.—R. S.

*Gas for Lighting, Heating, and Motive Power Purposes; Impts. in the Manufacture of, and in Apparatus therefor.* C. M. Johnson, London. Eng. Pat. 14,273, June 11, 1897.

Gas is obtained from oil by heating the latter in a vaporising vessel, (*a*) by a water-jacket, through which an exhaust pipe from an engine is passed; (*b*) by a water-jacket supplied with hot water from the cooling jacket of an engine; (*c*) by a lamp heating a layer of water. above which is a layer of oil; or (*d*) by a hot-water coil from the cooling jacket of an engine. An automatic feeding apparatus, acting without a valve, may be employed for maintaining the level of the oil in the evaporator.—R. S.

*Laboratory Gas Furnace, A New or Improved.* G. T. Holloway, London. Eng. Pat. 21,311, Sept. 17, 1897.

The claim is for "A laboratory gas furnace in which the base and the body are made separate, but preferably secured together by dowel pins. or otherwise, the base extending beyond the body to form a stand for the door or doors when the same are drawn forward."—R. S.

*Supply of Combustible Liquids at a Constant Pressure to Lighting or Heating Appliances, Impts. relating to the.* H. H. Lake, Southampton Buildings. From La Compagnie Continentale d'Incandescence et de Chauffage (systemes Franck et Poitrimol), Brussels, Belgium. Eng. Pat. 5489, March 5, 1898.

A regulator for establishing and automatically maintaining at a predetermined value the air-pressure upon the combustible liquid contained in a feed-vessel is formed by a vertical pump, open above to the atmosphere, and communicating below with the surface of the liquid. In this pump works a piston which is loaded to the required amount. On the tube leading from the pump to the top of the liquid is a three-way valve, allowing communication to be made (*a*) between the pump and the liquid, (*b*) between the pump (below the piston) and the air, or (*c*) between the air and both the pump (below the piston) and the liquid. The loaded piston is raised by a rack actuated by a toothed pinion and a handle or key. The arrangement is shown connected to an incandescent lighting burner; and in a lyre-shaped form of this, the weight of the apparatus itself, suitably loaded if need be, produces the constant pressure on the liquid.—R. S.

*Incandescent Light or Bunsen Flame Wick Burners for Liquid Fuels, Impts. in or connected with.* J. Spiel, Berlin. Eng. Pat. 13,113, May 27, 1897.

The supply of air from the exterior to the top of the wick is to be permanently regulated according to the requirements of the burner by narrowing the cross-section of the passage for the air. Thus the burner cap surrounding the wick tubes may have an annular constriction near its upper part, so that the distance between the constricted part and the wick tube is only, say, 3 mm.; or the cap may simply be narrowed down considerably at its upper part. Another arrangement is to have the outer wick tube stamped with an annular protuberance, or it may be fitted with an annular collar or a plate.—H. B.

*Incandescent Bodies for Gas and other Lighting, Impts. in the Manufacture of.* H. Kayser, Berlin. Eng. Pat. 14,259, June 11, 1897.

A fabric is first impregnated as usual with a solution of, say, thorium nitrate, then burned off, and the skeleton is dipped into a solution containing 0·1 to 5·0 per cent. of "suitable binding salts"—say nitrate of potassium. After drying and exposing to a flame for a short time, the mantle is again dipped into thorium nitrate solution, dried, ignited, dipped into potassium nitrate solution, and these operations are repeated till the mantle has attained the desired strength, when it may be treated with cerium solution. Instead of thorium and cerium, other combinations of earths may be used, and as "binding" materials there may be used salts of the alkalis, of alkaline earths, of aluminium, chromium, zinc, &c. There is claimed the process of dipping alternately into solutions of the incandescence and binding materials, and mantles so manufactured are stated to be very strong.—H. B.

*Acetylene Gas Generator, An Improved; chiefly designed for Application to a Portable Lamp, but also applicable for other Purposes.* C. Crastin, Holloway, and G. Baldwin, West Dulwich, formerly of Holloway. Eng. Pat. 9928, April 20, 1897.

A number of generating chambers are placed around a water tank and superposed gas holder, and each chamber contains carbide insufficient to yield enough gas to fill the gasholder, which is of bellows or other collapsible pattern. The waterways from the tank to the chambers are normally closed by valves, which may be opened by means of a key when water is to be admitted to a chamber. The generated gas passes through a non-return valve to the interior of the collapsible holder, which it extends. Indicators, safety devices, and cleansing cocks are provided.—J. A. B.

*Acetylene or other Gas generated by the Addition of a Liquid to a Solid, Impts. in Apparatus for Generating.* E. Evans, Llanrwst, Denbigh. Eng. Pat. 10,508, April 27, 1897.

Beneath a gasholder is a chamber in which are trays having a large number of compartments containing carbide, the compartments being arranged so that water entering the first flows on from one compartment to the next as the carbide in each is exhausted. The water is supplied from the gasholder tank through a pipe having a valve, which closes when the gasholder rises above a certain height.
—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* J. J. Patterson, Batavia, State of New York, U.S.A. Eng. Pat. 10,686, April 29, 1897. (Under Internat. Convention.)

Water passes from a closed reservoir, through an intermediate chamber, into a generating vessel containing carbide. The gas generated, passes through a cooler into a gasholder the movements of which control cocks in such a manner that only the water in the intermediate chamber is discharged on to the carbide at one time, and that when, owing to exhaustion of the carbide, the generation of gas ceases, the supply of water to the generator is cut off. A device for bringing automatically a second generator into action when the first is exhausted is described.—J. A. B.

*Acetylene Gas Plant, Impts. in.* D. Whalley, J. Hacking, and The Ideal Gas Company, Ltd., all of Blackburn, Lancs. Eng. Pat. 13,667, June 3, 1897.

Water is supplied to carbide in a generator at sufficient pressure to flood that vessel and expel all gas from it. The gas evolved passes through a pipe carried upwards to a height above that to which the water will ascend, to a cooler, and thence, by a similar pipe, to a gasholder. Water cannot therefore be carried forward from the generator to the cooler, and from the cooler to the gasholder.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Automatically Generating, Storing, and Supplying.* F. H. Haviland, Bournemouth, Hants, and W. H. Murch, Cradley Heath, Staffs. Eng. Pat. 14,208, June 11, 1897.

A cistern is placed above, and communicates with, a closed vessel, which serves as a relief gasholder. Water is supplied to carbide in a perforated vessel within a generating chamber, through a pipe which enters the relief gasholder at a certain height, so that when excess production of gas has depressed the level of the water in the relief gasholder below the mouth of the pipe, water ceases to pass to the carbide. The generated gas passes through a cooling coil in the cistern to the point of combustion; the surplus temporarily passes into the relief gasholder.—J. A. B.

*Gas, Impts. in Apparatus for Generating; by the Addition of a Solid to a Liquid.* G. W. Gaskell and R. F. Reeve, Liverpool. Eng. Pat. 14,313, June 12, 1897.

Carbide, sulphide, or carbonate is put in a box attached to the crown of the bell of a gasholder. The bottom of the box is hinged on one side, and is attached to a lever, the end of which supports a weight by means of a chain. When the bell descends, the weight strikes the bottom of the holder and releases the lever, so that the contents of the box are discharged on to a perforated diaphragm in the bell, and come in contact with the liquid in the tank of the gasholder. As the bell rises, the weight closes the bottom of the box, which may then be recharged without risk of escape of gas.
—J. A. B.

*Acetylene Gas Burners, Impts. in and relating to.* C. Kaestner, Halle-on-Saale, Germany. Eng. Pat. 15,060, June 23, 1897.

A capsule rests, by means of an internal shoulder, on the head of an ordinary burner. The capsule terminates above in two circular hollow cheeks, between which is the lower part of the flame. In the hollow cheeks are lateral openings through which air is drawn in quantity proportioned to the gas burned. Complete combustion of the gas is said to be obtained.—J. A. B.

*Acetylene Gas, Impts. in Means Employed in the Manufacture of.* O. V. Sigurdsson, Hammersmith. Eng. Pat. 16,793, July 15, 1897.

At the base of a hopper on the crown of a gasholder bell is a chamber in which a hollow cylinder is rotated by means of a lever engaging with a pin on one of the guide rods of the holder. Beneath the cylinder is a feeding chamber, the bottom of which is opened when a weight attached to an extension of it comes in contact with a bar or grating within the holder. As the bell descends, the hollow cylinder is first rotated so that carbide passes from the hopper into it. By further rotation the carbide is passed from the cylinder to the feeding chamber, the bottom of which opens on the continued descent of the bell, and allows the carbide to fall into the water. On the rise of the bell the bottom of the chamber and the hollow cylinder are restored to their former positions.—J. A. B.

*Acetylene Gas Producer.* H. de Thiersant and W. A. Coulson, both of London. Eng. Pat. 29,571, Dec. 14, 1897.

A receptacle containing carbide is placed within a receiver having lateral apertures and an outer casing which provides an annular space closed at top, in which gas, passing through the apertures, may collect. The whole is surrounded by a casing provided at top with a pipe for the escape of surplus gas. The apparatus is contained in a vessel into which water is put. The gas generated, passes from the carbide receptacle, through bent condensation tubes, to a tap and outlet, but if the production exceed the consumption, the surplus gas escapes through the escape pipe provided.
—J. A. B.

*Acetylene, Impts. in Apparatus for Generating.* H. Berger, Berlin. Eng. Pat. 4113, Feb. 18, 1898.

A cylinder extending upwards from the base of a bell gasholder to above the water level, has a top which closes automatically when the pressure in the bell exceeds that within the cylinder. A column of carbide holders is placed in the cylinder and water is supplied to them through a pipe having a cock controlled by the movements of the bell. The pressure of the generated gas lifts the lid of the cylinder, and the gas escapes to the bell; but when the carbide is exhausted, the holders may be removed without risk of escape of gas.—J. A. B.

*Acetylene Gas, New or Improved Apparatus for Generating and Utilising.* L. P. Hvid, Copenhagen. Eng. Pat. 5498, March 5, 1898.

The apparatus, which is portable, comprises a perforated receptacle, in the upper part of which is a funnel, the bottom of which is closed by an india-rubber membrane having a small fissure. In the funnel is a grid on which carbide is placed, and to the bottom of the funnel is attached an india-rubber bag. In the top, common to the funnel and receptacle, is the outlet gas tube, with cock and safety valve. Water is poured over the carbide, the top is screwed on, and the pressure of the generated gas forces the water into the india-rubber bag, whence it rises as the pressure within falls owing to consumption of gas. 15 lb. per sq. in. is stated to be the most practicable working pressure in the generator.—J. A. B.

*Acetylene, Improved Apparatus for Generating.* C. H. P. Schlüter and C. L. F. Lüdemann, both of Hamburg. Eng. Pat. 5594, March 7, 1898.

Water flows into a container when the bell of a gasholder descends. It raises a float in the container, and thereby liberates in succession a series of carbide vessels, which discharge their contents into the water as they are liberated by the float. The gas generated passes from the top of the container, through purifying material, to the gasholder.—J. A. B.

*Carbide Non-Hygroscopic, Process for Rendering.* P. Jensen, London. From J. von Orlowsky, St. Petersburg. Eng. Pat. 4298, Feb. 21, 1898.

There is claimed—"A process for producing non-hygroscopic carbide, for instance calcium carbide, consisting in saturating the carbide with liquid hydrocarbons, as for instance petroleum." The carbide, immediately after the usual crushing process, as it comes from the furnace, and while still having a temperature of 100° to 120° C., is conveyed into a bath of petroleum residues, where it remains about 5 to 10 minutes. It is then withdrawn and allowed to drain. It is stated that the moisture of the air does not decompose the carbide so treated, whereas with a body of water it decomposes very slowly at first and with increasing rapidity "as the particles are laid bare."—H. B.

*Calcium Carbide, An Improved Receptacle for the Storage of.* L. Rumelin and H. Auer, Gratz, Austria-Hungary. Eng. Pat. 4159, Feb. 22, 1898.

The receptacle consists of a box having an opening in the bottom for filling or emptying it, the opening being closed by a cap, which can be screwed on to the threaded rim which surrounds the opening. The sides of the box are continued downwards to a plane somewhat below that of the screw cap. The top of the receptacle is provided with a handle. The damp air which is enclosed when the box is filled liberates some acetylene, which prevents "the penetration of air or liquids."—H. B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Paraffin Oil.* P. Soltsien. Zeits. für öffentl. Chem. **4**, 223. Chem. Centr. 1898, **1**, [14], 869.

Although up to the present dextro-rotation of mineral oils has seldom been observed, it is stated that all commercial paraffin oils exhibit a small dextro-rotation. The extent of the rotation increases with the density of the oil. In a 200 mm. tube, the dextro-rotation amounts to from 1° 14′ to 4° 11′; with thick "paraffinum liquidum D A B," from 3° 23′ to 4° 11′.—A. S.

*Ozokerite, Valuation of.* E. von Boyen. Zeits. angew. Chem. 1898, **17**, 383.

*See under* XXIII., *page* 609.

*Paraffin, Commercial, Estimation of the Setting Point of.* L. Singer. Chem. Rev. Fett- u. Harz-Ind. **5**, [4], 65.

*See under* XXIII., *page* 609.

*Oil of Turpentine, The Custom House Technical Examination of; and a Simple Method of Distinguishing Pure Oil of Turpentine from "Patent" Oil of Turpentine, or Mixtures of Both.* F. Evers. Zeits. für öffentl. Chem. **4**, 211.

*See under* XXIII., *page* 607.

### PATENTS.

*Shale, The Destructive Distillation of; Impts. in and relating to Retorts for.* W. Young, Peebles, and J. Fyfe, Glasgow. Eng. Pat. 13,665, June 3, 1897.

This invention comprises improvements in retorts for the destructive distillation of shale in accordance with the principles described in Eng. Pat. 1377, March 21, 1882, granted to W. Young and G. T. Beilby. The objects are to reduce labour, save fuel, and increase the yield of products, and to enable existing but worn-out retorts, which have been erected in accordance with the above invention, to be economically replaced upon existing foundations by similar retorts, provided with improved and enlarged multiple hoppers, built of steel plates and bolted at the bottom to flanges on the upper ends of a number of retorts, and permitting of differential expansion of the retorts and hoppers. To ensure that the shale may regularly pass to the retorts from the hoppers, each hopper is provided with a rocking shaft or similar device, to which are attached rods or chains suspended in the mouths of the retorts, and capable of being reciprocated or raised and lowered therein. The chambers for the reception of the spent shale are similar in construction to those in ordinary use, but are greatly enlarged. This is accomplished by thinning the division walls and replacing the front walls by iron plates. The lower end of the retort is provided with a mechanical device, consisting of a rocking shaft furnished with prongs in part extending through a grating, the prongs being oscillated to effect the disengagement and discharge of the spent shale. When the ash is soft and free from grit, it suffices to provide a screw shaft with right- and left-handed threads under each retort, the shaft being rotated in order that the screw threads may effect the discharge of the spent shale. These improvements enable most of the work to be done during the day, the multiple hoppers and spent shale chambers being of such dimensions as will supply fresh shale and receive spent shale, during the night shift, the only labour then required being that of supervision, regulating the temperature of the retorts, and seeing that the mechanical arrangements are working properly. —D. B.

*Asphalt, Impts. in the Refining of.* W. Scott Wilkinson, Baltimore, U.S.A. Eng. Pat. 3793, Feb. 15, 1898.

The methods hitherto adopted for refining asphalts, particularly such as are obtained from the Pitch Lake in the Island of Trinidad, are said to have given a product inferior in all essential qualities to that produced by this invention. The purification is effected by subjecting crude asphalt in an open tank to the action of steam heat, disseminated throughout it. The tank contains sets of steam pipes, the steam employed being preferably conducted through jacketed pipes to the heating pipes. After passing through them, and in its passage distributing its heat thoroughly and evenly through the mass of asphalt, the steam is condensed and returned to the boiler. During this operation the asphalt is agitated by jets of compressed air or by other suitable means, in order to bring almost every portion of the mass into contact with the heated surfaces, thereby breaking down the emulsion, setting free and producing a rapid evaporation of the water, and simultaneously keeping the mass so mixed as to prevent the inorganic matter from settling and carrying down with it valuable asphalt.—D. B.

## IV.—COLOURING MATTERS AND DYES.

*Tetramethyldiamidobenzophenone, Nitro- and Bromo-Derivatives of.* E. Grimaux. Comptes Rend. **126**, [16], 1117—1118.

With a view of determining the effect of the introduction of the nitro group or a bromine atom into tetramethyldiamidobenzophenone upon the dyestuffs formed from these products by condensation with substituted aromatic amines, the author has prepared them by the following methods: To the ketone dissolved in 20 parts of ordinary sulphuric acid are added 2 mols. of potassium nitrate; after vigorous agitation the mixture is allowed to stand 24 hours, and is then poured into water, and neutralised with a solution of sodium carbonate. The precipitate after crystallisation from acetone, forms orange crystals, melting at 166° C. The nitration being performed in presence of a large excess of sulphuric acid, the nitro groups probably enter each nucleus in a position ortho to that of the ketone group. On reduction with stannous chloride, an amorphous base is obtained melting at 69° C., and giving a crystalline picrate. The di-bromo derivative is obtained by dissolving the ketone in 10 parts of chloroform and slowly adding 2 mols. of bromine. After removing the chloroform on the water bath, the residue is taken up with hydrochloric acid, diluted with its own volume of water, and the base is precipitated by ammonia. It is crystallised from 25 parts of boiling alcohol and forms hard, shining prisms, melting at 131° C. The di-nitro and the di-bromo ketones both gave condensation products with dimethylaniline and phenyl-α-naphthylamine in presence of chloride of phosphorus. The new dyestuffs give, however, duller shades than the non-substituted ones, although they appear to be somewhat faster to soap. It would thus appear that the introduction of nitro groups or of bromine into dyestuffs derived from tetramethyldiamidobenzophenone is not advantageous.—T. A. L.

*Azo-Dyestuffs in Calico Printing, Phenolic Compounds for the Production of Insoluble.* J. Brandt. Bull. Soc. Ind. Mulhouse, 1898, **68**, 43—49.

β-naphthol is practically the only phenol employed in the production of insoluble azo dyestuffs on cotton: α-naphthol, amidonaphthols, β-hydroxynaphthoic acid (m.p. 216°), and other compounds, being of less importance for the purpose.

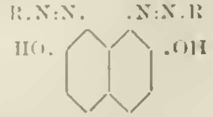
Experiments were made with the view of discovering whether there were any compounds other than the above-mentioned which might with advantage find technical application, attention being restricted to those phenols which, like β-naphthol, give *o*-hydroxyazo combinations.

2.2′-*Dihydroxynaphthalene*, although easily soluble in water, will not react with diazo salts, except in the presence of alkalis, which cause it quickly to turn brown on exposure to air. An addition of glucose to the solution prevents this, and thus enables clear and regular results to be obtained in printing.

A suitable solution is composed of 60 grms. of 2.2′-dihydroxynaphthalene, 85 grms. of caustic soda at 40° B., 100 grms. of glucose, 100 grms. of Turkey-red oil, 20 grms. of sodium aluminate, and 2 litres of water. The tissues prepared with this, and dried at a low temperature, are printed with the customary, thickened solutions of diazo salts, and are then washed and soaped at 50° C.

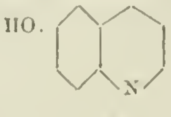
The colours of the compounds produced are as follows :—With diazotised *a*-naphthylamine, *p*-toluidine and a copper salt, *o*-nitraniline, and *p*-nitraniline, browns ; benzidine, dark brown ; dianisidine and a copper salt, blue-purple.

They are as fast to soap as the corresponding *β*-naphthol derivatives, from which it would appear that they are disazo compounds, of the type



the reaction being of interest, as it has not hitherto been found possible to effect a combination of 2.2′-dihydroxynaphthalene with more than one molecular proportion of a diazo salt.

*p-Hydroxyquinoline*



in alkaline solution is very slightly affected by atmospheric oxygen. The padding solution employed, contained 30 grms. of the phenol, 30 grms. of caustic soda at 40° B., 50 grms. of Turkey red oil, and 10 grms. of sodium aluminate, per litre. The following colours are obtained :—From *a*-naphthylamine, dull red ; *p*-nitraniline, bright orange ; *o*-nitraniline, yellow-orange ; benzidine, chocolate ; *p*-toluidine, with a copper salt, brown ; dianisidine, with a copper salt, blue-purple.

The diazo solutions may be printed without the usual addition of sodium acetate, rather deeper colours being thus produced. This is probably to be ascribed to the slightly basic character of the phenolic compound.

*p-Cresol* gives compounds of the following colours :—With diazotised *a*-naphthylamine, buffy-yellow ; *p*-nitraniline, yellow mode ; *p*-amidobenzaldehyde, mode ; the last-named base, together with a copper salt, a darker mode ; dianisidine and a copper salt, grey-purple ; Magenta, mode ; benzidine, brown-yellow. The tissues were padded with a solution of 60 grms. of *p*-cresol, 70 grms. of caustic soda at 40° B., and 2 litres of water.

The colours obtained from *p*-cresol being very distinct from those given by *β*-naphthol, striking conversion effects may be produced with the two. Thus, by padding the tissue in an alkaline solution of *p*-cresol and printing across it in fine stripes a thickened solution of *β*-naphthol (30 grms. of *β*-naphthol, 30 grms. of caustic soda at 40° B., 0·5 litre of water, and 0·5 litre of gum tragacanth, 120:1000), and then a two-colour pattern with diazotised *a*-naphthylamine, and Fast black (Noir réduit) (steaming 3 minutes, washing, and soaping at 50° C.), there is produced a pattern in black on a ground of claret and buff. Or, again, by preparing with *p*-cresol and glucose (200 grms. per litre), printing with *β*-naphthol as described, then with diazotised *a*-naphthylamine and precipitated sulphur (200 grms. per litre), and finally "covering" with an alkaline indigo mixture (steaming, &c., as usual), a reserve pattern in claret and buff on a blue ground is obtained. If, in the last example, diazotised benzidine be taken in place of diazotised *a*-naphthylamine, and be printed side by side with indigo, a pattern in puce, brown, and blue results.

The azo compounds from *p*-cresol, although fast to soap and to rubbing have the defect of volatilising and staining yellow contiguous parts of the tissue, packing papers, &c. Moreover, *p*-cresol has a disagreeable smell, which it is difficult to entirely remove from the printed tissues. The compounds from benzidine and Magenta volatilise the least, and it is considered that those from the first named base may have a technical value.—E. B.

### *Dimethylaniline, Action of Carbon Bisulphide on.* J. Weinmann. Bull. Soc. Ind. Mulhouse, 1898. 68, 40—43.

Carbon bisulphide is without action on dimethylaniline, even at high temperatures, in the absence of metallic salts. In their presence the compounds react with ease, the action under the influence of zinc chloride being quantitative below 100° C. According to the temperature and duration of the action, there are formed dimethylamidothiobenzoic acid, tetramethyldiamidodiphenylthioacetone, hexamethylparaleucaniline and tetramethylthioaniline.

Dimethylamidothiobenzoic acid is chiefly produced on heating in a closed vessel carbon bisulphide (4 kilos.), dimethylaniline (30—40 kilos.), and zinc chloride (20 kilos.), at 60°—70° C. for 70—100 hours. The product is washed with dilute acetic acid to remove the zinc chloride, the excess of dimethylaniline which separates as an oily layer, containing in solution the dimethylamidothiobenzoic acid. The latter is extracted with caustic soda and precipitated by either hydrochloric or acetic acid.

Tetramethyldiamidodiphenylthioketone is formed when a mixture, such as that described, is heated, while well agitated, at 80° C. during 100°—120° hours. After the separation of dimethylamidothiobenzoic acid the dimethylaniline is removed by a current of steam, leaving the ketone as a brown crystalline powder. This is purified from hexamethylparaleucaniline by means of dilute hydrochloric acid. The ketone finds use in the manufacture of Auramine, the base of which it gives in quantitative amount when it is heated under pressure with alcoholic ammonia. Aqueous ammonia at 140° C. converts it into tetramethyldiamidobenzophenone.

Hexamethylparaleucaniline is the principal product of the reaction, which takes place when the above compounds are heated at 100°.

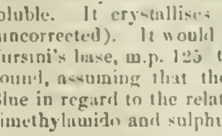
*Tetramethylthioaniline*, $S[C_6H_4N(CH_3)_2]_2$, is also obtained with the above quantities of compounds, at 130°—140° C. The reaction in this case proceeds abnormally, the carbon bisulphide in its action resembling carbon tetrachloride or chloroform. Tetramethylthioaniline and thioformaldehyde are first produced. The latter then reacts with dimethylaniline, forming tetramethyldiamidodiphenylmethane and hydrogen sulphide.

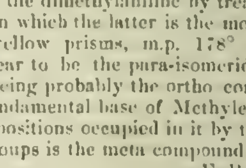
The tetramethylthioaniline is extracted from the mass produced by treatment with caustic soda, filtration from the zinc sulphide, removal by distillation of the dimethylaniline, and crystallisation of the crude residue from alcohol. It may also be separated from the dimethylaniline by treatment with hydrochloric acid, in which the latter is the more soluble. It crystallises in yellow prisms, m.p. 178° C. (uncorrected). It would appear to be the para-isomeride, Tursini's base, m.p. 125° C., being probably the ortho compound, assuming that the fundamental base of Methylene Blue in regard to the relative positions occupied in it by the dimethylamido and sulphur groups is the meta compound.

—E. B.

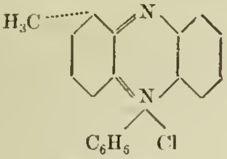
### *Tolusafranine, Aposafranine and Azonium Compounds therefrom.* F. Kehrmann and A. Wetter. Ber. 31, 966—977.

With a view of obtaining information with regard to the composition and constitution of several of the safranines on the market, the authors have removed the amido groups from tolusafranine in a manner similar to that employed in the case of phenosafranine (Ber. 29, 2316 ; this Journal, 1896, 802). They find that the well-crystallised tolusafranine obtained from Geigy yields, after elimination of one amido group, a mixture of two aposafranines, which can be separated by means of their hydrochlorides, the one being a mono- the other a di-methylaposafranine. On removing the second amido group, two different azonium salts are formed, and, in the mother liquors, a third very easily soluble aposafranine was found, which could not be obtained in a crystalline form. The mono- and di-methyl derivatives have apparently the formulæ:—

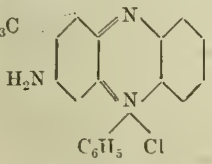


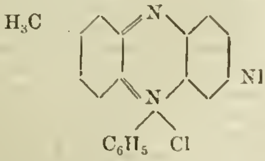


The monomethyl derivative was obtained by diazotising tolusafranine sulphate with sodium nitrite, boiling with alcohol, converting the sulphate into the chloride, and crystallising this latter. It forms long, light-brown needles, tolerably soluble in pure water and easily in alcohol, with a blood-red colour, and in concentrated sulphuric acid with a yellowish-green colour, changing through emerald-green to blood-red on dilution. The alcoholic solution shows a strong, bright-red fluorescence. The platinum double salt and the nitrate are sparingly soluble. The acetyl derivative (yellowish-brown needles) differs from aposafranine in the behaviour of its chloride towards alcoholic ammonia. As already described (Ber. **30**, 1565; this Journal, 1897, 793), acetylaposafranine chloride when treated with ammonia is converted into acetylphenosafranine chloride, whereas this homologue under the same treatment is simply hydrolysed, only very small quantities of tolusafranine chloride being formed. This behaviour is still more pronounced in the case of acetylrosinduline, which can be boiled with aniline without change, whilst the acetylrosinduline of Nietzki-Otto's isorosinduline is more easily substituted even than acetylaposafranine. In order to remove the amido group from methylaposafranine, the purified chloride is dissolved in concentrated sulphuric acid, and so much water is added that the solution just remains clear. Sodium nitrite solution is then run in until the green colour turns blood-red. The whole is then poured into twice its volume of alcohol, and precipitated after standing two hours with a saturated ferric chloride solution. The double salt forms thick brownish-red prisms, melting at 150° C. The compound has the formula—*



and on treatment with ammonia in alcoholic solution is converted into the two following isomeric methylaposafranines.



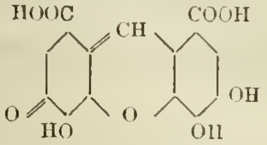


As theory predicts, the first predominates. Its chloride is sparingly soluble. The amido groups in both these compounds can be again removed when the same azonium compound is obtained from each, and it is thus possible to convert the two isomeric apotolusafranines into one another.

The dimethylaposafranine was obtained by recrystallising the commercial tolusafranine chloride until the crystals consisted entirely of uniform greenish needles. On removal of one amido group, an easily soluble magenta red aposafranine chloride was obtained, which consisted of a mixture containing principally the dimethyl derivative. This was diazotised and precipitated with ferric chloride, the iron double salt of the dimethyl derivative being much less soluble in glacial acetic acid than its lower homologue. The salt forms light brown plates, melting at 190° C., very easily soluble in water, hot alcohol, and acetic acid, and sparingly soluble in cold alcohol or acetic acid. The solution in concentrated sulphuric acid is greenish-brown, turning golden-yellow on dilution with water. A concentrated solution of the salt in 80 per cent. alcohol turns magenta red on adding a sufficiency of concentrated ammonia. After adding water and evaporating to expel ammonia, the filtrate is treated with concentrated hydrochloric acid, when the chloride of dimethylaposafranine crystallises out. No isomeric compound could be detected, from which it is assumed that the substance has the formula given above. The chloride of the acetyl derivative forms thin reddish-brown needles, which dissolve easily in water and alcohol with a yellowish blood-red colour, the solution in alcohol having a slight brick-red fluorescence. Amines such as ammonia, dimethylamine, and aniline react easily, forming tolusafranine derivatives. It might have been expected that this would be the case, and that it would behave similarly to acetylaposafranine (which by the action of amines yields phenosafranine derivatives) on account of its symmetrical constitution.—T. A. L.

*New Blue Mordant Dyestuff* [*Hydroxyfluorone Carboxylic Acid*]. R. Möhlau. Rev. Gén. des Mat. Col. 1898, **2**, 129—131.

A DYESTUFF of the constitution—



is produced by adding the calculated amount of nitrous sulphuric acid to a cold concentrated sulphuric acid solution (300 grms.) of methylenedigallic acid (10 grms.), leaving the mixture at rest for 24 hours, and diluting first with fairly concentrated (1:1), then with dilute (1:10) sulphuric acid. The compound is thus obtained in combination with sulphuric acid in large, transparent, red prisms, which become opaque and irregular in shape when treated with water; the yield is about 70 per cent. of the methylenedigallic acid taken. It dissolves sparingly in hot water and in the ordinary organic solvents, but freely in concentrated sulphuric acid, with an orange-red colour. Alkali carbonates, borates, and acetates dissolve it with a purple, ammonia and caustic alkalis with a blue or greenish-blue colour; none of these solutions exhibits dichroïsm.

It combines with metallic hydrate mordants giving insoluble or sparingly soluble lakes, those of uranium, cerium, nickel, cobalt, tin, aluminium, iron, and chromium being blue or green-blue. The chromium lake becomes dark green in colour when it is treated with potassium bichromate and acetic acid.

Acetyl (m.p. 140·5°—141·5°), benzoyl, and leuco-compounds were prepared.—E. B.

*Indigo, Analysis of.* A. Brylinski. Bull. Soc. Ind. Mulhouse, 1898, **68**, 33.

*See under* XXIII., *page* 609.

*Substantive Dyestuffs with p-Nitraniline and Copper Sulphate, Development of.* E. Grossman. Färber Zeit. **9**, [10], 151.

*See under* VI., *page* 576.

*β-Naphthol, Detection of α-Naphthol in.* A. Dubose. Bull. de la Soc. ind. de Rouen, 1897, 434.

*See under* XXIII., *page* 607.

## PATENTS.

*Colouring Matters* [*Blue*], *Manufacture of Substantive.* J. Imray, London. From "La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis," Paris, France. Eng. Pat. 11,997, May 14, 1897.

AN extension of Eng. Pat. 5736 of 1890 (this Journal, 1891, 358). According to the present specification, nitro-azo colouring matters are condensed by means of caustic alkali, with derivatives of a diamine, one $NH_2$ group of which is free or acetylated, and the other is combined as an azo group with a phenol, naphthol, or sulphonated amine. The following is a typical example of the method employed:—The diazo compound from 13·8 kilos. of *p*-nitraniline is combined with 30·4 kilos. of R salt, and the resulting nitro-azo derivative is converted into the amido compound. This, in the form of paste, is mixed with the azo dyestuff obtained by combining 27·6 kilos. of *p*-nitraniline with 60·8 kilos. of R salt, and stirred into 5,000 litres of water, together with 500 litres of caustic soda-lye of 10 B. The mixture is heated for 24 hours to 75°—85° C., and the dyestuff produced is precipitated with 5 per cent. of salt. It forms a reddish-brown powder soluble in water, and dyes unmordanted cotton blue. By substituting 1·4-naphthol sulphonic acid for the R salt, a violet dyestuff is obtained, whilst if the nitro-amine be combined with 2·3′-naphthol sulphonic acid and this is condensed with the amido compound mentioned in the first example, a violet-blue dyestuff is formed.—T. A. L.

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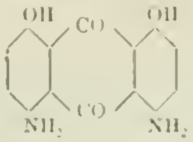
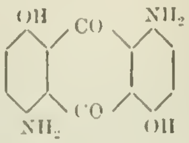
* It is uncertain which of the two positions indicated by the dotted lines is occupied by the methyl group.

*New Anthraquinone Dyestuffs, The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 12,011, May 14, 1897.

The specification refers to the production of mono- and disulphonic acids of the two following anthraquinone derivatives—





of which the first may be termed *p*-diamidochrysazin, the other being known as *p*-diamidoanthrarufin. These dyestuffs give very valuable blue shades on unmordanted wool from an acid-bath, and on chrome-mordanted wool, greenish-blue shades. In order to produce these substances, chrysazin or anthrarufin is sulphonated, dinitrated, and the compounds so obtained are then reduced. According to another method, the diamidodihydroxyanthraquinones are sulphonated according to Eng. Pat. 14,345 of 1893 (this Journal, 1894, 723), that is, with sulphonating agents in presence of boric acid. The first method yields disulphonic acids, whilst monosulphonic acids are produced according to the second method, if the sulphonation is effected quietly. The following is an example of the first method :—A solution of 20 kilos. of chrysazin, $1.HO.C_6H_3:C_2O_2:C_6H_3.OH.1'$, in 80 kilos. of fuming sulphuric acid (20 per cent. $SO_3$) is heated at 100°—120° C. until a sample is soluble in water. The melt is then mixed with 120 kilos. of sulphuric acid (66° B.) and 52 litres of a mixture of sulphuric and nitric acids (containing 10·4 kilos. of pure nitric acid) are gradually added at 20°—30° C. After standing some time until a sample neutralised with caustic soda shows a pure blue colour with sodium stannite, the reaction is finished, and the greater part of the dinitrochrysazin disulphonic acid separates as a crystalline precipitate. It is filtered off on an asbestos filter, dissolved in 1,500 litres of hot water, to which 50 kilos. of potassium chloride are added, and, on cooling, the potassium salt crystallises out. In order to reduce it, 5 kilos. of the salt are dissolved in 250 litres of water, and mixed with a solution containing 12 kilos. of stannous chloride, 30 litres of water, and 30 litres of concentrated hydrochloric acid. The colour of the mixture turns blue, and after a short time *p*-diamidochrysazin disulphonic acid separates as a gelatinous mass, which becomes crystalline on warming. This reduction may also be effected on the fibre. As an example of the second method, 10 kilos. of *p*-diamidoanthrarufin—

$$(1.4)OH.NH_2.C_6H_2:C_2O_2:C_6H_2.NH_2.OH(1'.4')$$

(Ber. 29, 2937 and 2940) are heated with 200 kilos. of fuming sulphuric acid (30 per cent. $SO_3$) and 10 kilos. of crystallised boric acid at 100°—130° C., until a sample, after washing free from acid, is readily soluble in dilute soda-lye. The melt is then poured into water, and the new dyestuff, after boiling up, is filtered off. It dissolves in caustic ammonia with a blue, and in soda-lye with a greenish-blue colour, whilst it is sparingly soluble in boiling water, with a violet colour. As in the first example, the chrysazin derivative may be employed, or mixtures of diamidoanthrarufin and diamidochrysazin, but the patentees disclaim the use in example II. of the diamidodioxyanthraquinone of Eng. Pat. 974 of 1894 (this Journal, 1895, 30).
—T. A. L.

*Brown to Black Dyestuffs, Process for the Manufacture of.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Höchst a M., Germany. Eng. Pat. 13,104, May 27, 1897.

The sulphonic acids of amidonaphthols, dihydroxynaphthalenes, their nitroso derivatives and azo dyestuffs derived from them, yield brown to black dyestuffs when heated for some time with sodium sulphide and sulphur. In the case of the dihydroxy sulphonic acids it is best to add ammonia salts. The products are easily soluble in water and dye unmordanted cotton. The following typical example is given :—1 kilo. of sodium 2.1′.3′-amidonaphthol sulphonate is heated for one hour to 100°—120° with "several times the quantity of sodium sulphide and double the quantity of sulphur," with constant agitation. The temperature is gradually raised and kept at 200°—250° C. for several hours. The melt, after cooling, forms a black mass soluble with a brownish-green colour in water, and dyeing cotton a greenish-black. Acids precipitate the dyestuff from the solution, whilst it is soluble in alkalis. Other acids suitable for this process are the 1.1′.3.3′ and the 1.1′.3.4′-amidonaphthol disulphonic acids and the 1.1′.3.3′ and the 1.1′.2.4′-dihydroxnaphthalene disulphonic acids.
—T. A. L.

*Aromatic Aldehydes, Impts. in the Production of.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 13,709, June 3, 1897.

By passing a mixture of hydrochloric acid and carbon monoxide into aromatic hydrocarbons of the benzene series in presence of cuprous chloride and aluminium chloride, aldehydes of the benzene series are produced, the mixture of hydrochloric acid and carbon monoxide acting as the hypothetical formyl chloride ClCHO. For example, a mixture of gaseous hydrochloric acid and dry carbon monoxide is passed into a mixture of 10 kilos. of toluene, 10 kilos. of aluminium chloride, and 1 kilo. of cuprous chloride at a temperature of 40°—50° C. for about six hours, when 5 kilos. of aluminium chloride and 0·5 kilo. of cuprous chloride are added, and the mixed gases are passed through for another six hours. The whole melt is then poured into ice water and steam-distilled, when the aldehyde and unaltered toluene pass over and are separated by means of sodium bisulphite. The aldehyde produced, is the para compound and has the formula $1.4.C_6H_4.CH_3.CHO$. Similarly, from *m*-xylene is produced $1.3.4.C_6H_3.(CH_3)_2.CHO$, and mesitylene yields $1.3.5.2.C_6H_2.(CH_3)_3.CHO$, all of which are known ; whilst *o*-xylene gives $1.2.4.C_6H_3(CH_3)_2CHO$, boiling at 226° C., and *p*-xylene gives $1.4.2.C_6H_3.(CH_3)_2CHO$, boiling at 220° C., both of which were previously unknown.
—T. A. L.

*Naphthylamine Derivatives and Colouring Matters [Red, Bluish-Black] therefrom, Impts. in the Manufacture of.* R. Holliday and Sons, Ltd., J. Turner, and H. Dean, Huddersfield. Eng. Pat. 13,999, June 8, 1897.

By nitrating 1.4-chloronaphthalene sulphonic acid, two new chloronitronaphthalene sulphonic acids are produced, which, on reduction, yield the corresponding naphthylamine derivatives and probably have the constitution 1.4′.1′ and $1.1'.4'\text{-}C_{10}H_5.NH_2.Cl.SO_3H$. Dyestuffs are obtained from these acids by employing them in place of the well-known 1.1′ or 1.4′-naphthylamine sulphonic acids, the resulting products having different properties from those already known. For the production of the new acids, "100 kilos. of 1.4-chloronaphthalene sulphonic acid are dissolved in 5—6 parts of sulphuric acid (66° B.)" and nitrated with a mixture of 50 kilos. of sulphuric acid and 46 kilos. of nitric acid (70 per cent.) the temperature being kept below 25° C. The mixture is then diluted with water, reduced with iron borings, and converted into the sodium salt. The two isomers may be separated by the difference in solubility of their sodium salts, that of No. I. acid being much less soluble. A bluish-red dyestuff for unmordanted cotton is obtained by converting 18·4 kilos. of benzidine into the tetrazo compound and adding it to a solution of 56 kilos. of the sodium salt of chloro-naphthylamine sulphonic acid II. in presence of sodium acetate. After a few days, the mixture is made alkaline with sodium carbonate and the dyestuff is separated in the usual manner.—T. A. L.

*Colouring Matters [Black], Impts. in the Manufacture of.* H. R. Vidal, Paris, France. Eng. Pat. 14,132, June 10, 1897.

The patentee states that the action of sulphur on a mixture of two disubstituted *p*-derivatives of the benzene series, such as hydroquinone and *p*-amidophenol or *p*-amidophenol and *p*-phenylene diamine (see Eng. Pat. 5690 of of 1897; this Journal, 1898, 344) results in thiazines which are fugitive dyestuffs, and not directly suitable for cotton, whilst if *m*-di-derivatives be employed, the colours obtained dye cotton, but only in light-brown and yellow shades. By combining these two methods, that is, by reacting with sulphur on mixtures of para- and meta- derivatives of the same series, black dyestuffs of great intensity are produced which are stable to light, and can be used directly for dyeing vegetable fibres. Some of the products are soluble in acids, and can be applied for dyeing wool and silk. For example, on heating together, at about 200° C., 11 kilos. of hydroquinone, 11 kilos. of *m*-phenylene diamine, and 3 kilos. of sulphur, or 11 kilos. of *m*-amidophenol, 11 kilos. of *p*-phenylene diamine, and 3 kilos. of sulphur, or 11 kilos. of *m*-amidophenol, 11 kilos of *p*-amidophenol, and 3 kilos. of sulphur, in each case, until the evolution of sulphuretted hydrogen has ceased, a black mass is obtained, soluble in alkalis and alkaline sulphides, which dyes vegetable fibres black.—T. A. L.

*Asymmetric Diamidophenylacridines [Leather Yellows] and their Alkyl Substitution Products, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 14,920, June 19, 1897.

The specification relates to the production of dyestuffs analogous to the phosphine obtained from magenta residues. This is usually considered as consisting mainly of an asymmetric diamidophenylacridine, together with homologous substances. The phosphine-like bodies now described are obtained by condensing *p*-amidobenzaldehyde or its derivatives substituted in the amido group, with alphyl derivatives of *m*-tolylene diamine described in the following example. Heat together in an enamelled vessel 3 kilos. of dimethyl-*p*-amidobenzaldehyde and 5 kilos. of phenyl-*m*-tolylene diamine hydrochloride at 140°—150° C. until the melt becomes brittle on cooling. Break up and extract with 2,000 litres of water containing 25 kilos. of hydrochloric acid (30 per cent. HCl), cool, filter, and precipitate some of the impurities by adding a little sodium nitrate. Filter and then salt out the dyestuff by cooling with ice and adding sodium nitrate. A yellowish-red precipitate is obtained, which is filter-pressed and dried at 30°—40° C. It is sparingly soluble in cold water, but dissolves easily in hot water with a yellowish-red colour, and when pure separates from the aqueous solution in orange needles. The product consists principally of the dimethyl derivative of an asymmetric diamidomethylphenylacridine, and gives reddish-yellow shades on leather, as commercial phosphines does.—T. A. L.

*Colouring Matters from Oxazines, Manufacture of New.* J. Imray, London. From La Société L. Durand, Huguenin et Cie., St. Fons, Rhône, France. Eng. Pat. 6055, March 11, 1898.

The process consists in treating oxazines with sulphurous acid or sulphites under pressure in a suitable medium. Leuco compounds are obtained which oxidise easily in the air, and almost instantaneously in the presence of alkalis. The new products differ in appearance from the original oxazines and yield stronger, bluer, and purer shades. About 20 kilos. of ordinary gallocyanine, 100 litres of distilled water, and 25 kilos. of normal sodium sulphite are heated to 100° C. for 4—5 hours in an enamelled or lead-lined autoclave with agitation. The leuco derivative obtained, gives on chrome-mordanted wool bluer and brighter, and on chrome-mordanted cotton, bluer shades than those produced with the ordinary Gallocyanine bisulphite compound (Gallocyanine B S). The gallocyanine and water may be replaced by 200 kilos. of commercial Gallocyanine D H (10 per cent. paste), and, in place of the normal sodium sulphite, 18 kilos. of sodium bisulphite solution (40° B.) may be employed. Other oxazines suitable for this process are Gallamine Blue, Prune pure, Muscarine and Meldola's Blue.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Viscose and Viscoid.* L. Lefèvre. Rev. Gen. Mat. Colorantes, 1898, Feb.; Leipzig. Monatschr. für Textil-Ind. **13**, [3], 159—160.

The aqueous solution of the xanthogenate obtained by treating alkali-cellulose with carbon bisulphide in the cold, is employed under the name of "viscose" for sizing and facing paper, chiefly of a coarse quality, *e.g.*, packing paper, the attempts made to apply the treatment to white papers having hitherto proved unsatisfactory. For the former purpose the viscose is added to the pulp, and is then precipitated on the fibres, preferably by zinc sulphate, the presence of the gelatinous material during rolling increasing the tensile strength of the paper by as much as 80 per cent. In facing paper, the viscose is applied to the surface, then decomposed by heat, and calendered. This not only improves the paper in appearance, but also enables it to resist boiling water, and facilitates staining.

Textiles can also be finished with viscose, and thereby become coated with an elastic layer of cellulose, which, by embossing, pressing, &c., can be made to resemble leather for bookbinding and similar purposes. Fine effects may be produced on thin linen and cotton material by impregnation with viscose and china clay, followed by heating and pressing—a durable finish capable of resisting washing, bleaching, and hot soaping, and therefore superior to the ordinary starch or china clay finish, being thus produced. Damask effects are also obtainable by printing designs with viscose, and then drying, or *vice versa*.

When coagulated by a temperature of 70°—80° C., or by spontaneous evaporation, viscose yields "viscoid," which corresponds, in all but physical (finely divided) condition, with cellulose, except that it has a greater affinity for dyestuffs. Prepared in thin films it exhibits great tensile strength and elasticity; in thicker blocks it forms a blackish mass like horn, and can be coloured white by the incorporation of zinc oxide, brown or red with iron, and ebony with carbon. With cork dust or wood it forms a mass specifically lighter than water, and being impervious to the latter, is suitable for a variety of purposes—tubes, electrical apparatus, surgical instruments, &c.—C. S.

### PATENTS.

*Mercerising Vegetable Fibres or Textile Fabrics, Impts. in or relating to the Process of.* R. Thomas and E. Prevost, Crefeld, Germany. Eng. Pat. 9517, 2nd Edit., April 14, 1897.

"The fibre or fabric is submitted to simultaneous stretching and strong pressing action whilst it is undergoing Mercer's treatment or washing out by any suitable means, such as calenders, beating or beetling mechanism, rollers, or the like." An almost equally high gloss may be produced when the pressure is exerted after the removal of the lye, or even of the acid washing, but before drying, as the mercerised material still remains soft and susceptible to pressure. In either of these ways a gloss, fast to subsequent washing, is obtained, whereas if the material be allowed to dry before applying the pressure, the effect is only transient.—H. I.

*Cellulose, The Manufacture of Crimped Fabrics, such as Crape, and Articles ordinarily made of Crimped Human or Horse Hair, such as Wigs and the like, from.* W. P. Dreaper, Braintree, Essex, and H. K. Tompkins, West Dulwich, Surrey. Eng. Pat. 10,487, April 27, 1897.

Cellulose is dissolved in basic zinc chloride, with or without the addition of other metallic salts or phosphoric acid, or after treatment in a suitable manner with sulphuric acid in water alone, and the solution "is squirted through a die" into alcohol or acetone, which dissolves the reagent but

not the cellulose. The threads thus obtained are washed and dried. By introducing dyestuffs into the cellulose solution, or by dyeing the threads when made, coloured threads are obtained. They may be waterproofed by passage through solutions containing resins, or varnish, or gelatin. For crape, and as a substitute for hair, the threads are woven, or made into the desired articles and crimped as usual.
—H. I.

*Drying Fabrics, Yarns and Threads, and Fibres; An Improved Method of and Means for.* H. McPhail, Wakefield. Eng. Pat. 11,448, May 7, 1897.

An apparatus for quickly drying yarns and the like, without impairing their quality, consisting of a drying chamber A, heated by superheated steam circulating in the pipe system B. Steam is also admitted directly into the chamber by the perforated pipes C, while the exhaust escapes through an opening A³. The material to be treated is fed on to the topmost travelling band D by rollers D, D², and, after traversing the length of the chamber in one direction, falls upon the band immediately below, by which it is traversed in an opposite direction. The material is finally conveyed by the lowest band to an outlet A², and is deposited in a dried condition in a suitable receptacle. The rollers D¹ may be hollow perforated drums connected to the superheated steam supply, and in modified arrangements, where the material under treatment takes the form of a roll of fabric, this latter is itself led over the drums in succession. In a further arrangement the material may be spread out on the shelves of a truck or carriage, which is run into the drying chamber.—E. G. C.

*Conditioning Spun Cotton, Process for.* T. E. Wilson and P. Kestner, Lille, France. Eng. Pat 18,347, Aug. 6, 1897.

The cotton thread, wound on bobbins or cops, is introduced into a closed vessel, which is then evacuated of air, and steam admitted so as to maintain a partial vacuum. In this way the temperature at which the conditioning takes place, is kept below 85°, the melting point of the resin or wax contained in the cotton, and the weakening of the fibre, are thus avoided. A quarter of an hour treatment is found to suffice to set the twist on the thread.—H. I.

*Sizing Yarn, Impts. in or applicable to the Process of and Means for.* W. Baldwin, Tardeo, Bombay, India. Eng. Pat 19,256, Aug 20, 1897.

The yarn, immediately after passing through a pair of rollers, the lower one of which is immersed in the size, before it has time to reabsorb the air, enters the sizing trough. After passing through the trough, where it is supported and protected from the disturbing effects of the ebullition of the size by a roller, the yarn passes through a similar pair of rollers, which deprive it of its surplus size, and thus it leaves the trough. "By this means the yarn is completely filled with size, and, in consequence, has a smoother appearance and is more fit for weaving."

A passage through a steaming chamber immediately before sizing takes the elasticity from the fibres and causes them to conform to the twist.—H. I.

*Fabrics and Clothing Materials Acid-proof, Process for Rendering.* J. V. [illegible]sop, Hamburg, Germany. Eng. Pat. 29,766, Dec. 16, 1897.

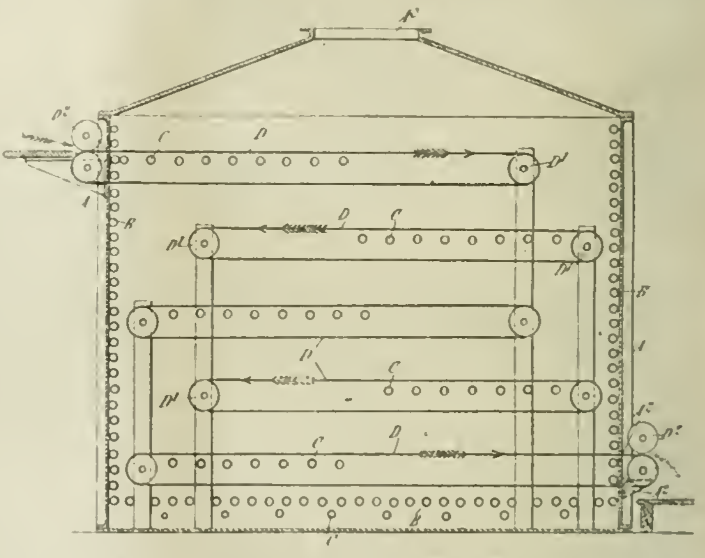
The process consists in coating fabrics or clothing with a layer of acid-proof vulcanised rubber or other acid-proof material, such as rosin, gum lac, varnish, and the like.
—H. I.

*Artificial Silk, Impts. in or in the Manufacture of.* H. C. Woltereck, New York, U.S.A. Eng. Pat. 3898, Feb. 16, 1898.

Cotton or other suitable thread is passed through a solution of cellulose in one of its solvents, and then through a coagulating solution, which takes up the solvent and leaves the cellulose adhering to the thread, to which it is fixed by passage through dilute acid, or, where nitrocellulose is used, through a solution of an alkaline sulphydrate or iron salt. Threads obtained in this way have all the desirable characteristics of artificial silk, and, in addition, a much greater tensile strength and cheapness.—H. I.

*Industrial [Waterproofing] Compounds involving the use of Gluten, Impts. in.* W. Painter, Baltimore, U.S.A. Eng. Pat. 4532, Feb. 23, 1898.

As ordinarily prepared from cereals, gluten soon ferments, loses its adhesive qualities, and becomes partially soluble in water. If, however, it be partly or completely dried at atmospheric temperatures, it no longer returns to the plastic condition; while if it be heated to 90° or 120° C., it is rendered much harder and less permeable to moisture according as the time and temperature of drying are increased. The inventor's composition, therefore, consists of a solution of gluten in glycerin, with or without the addition of ground cork; the ingredients being subjected to artificial heat before, during, or after incorporation. Convenient proportions for certain purposes are: gluten, 65 per cent.; glycerin, 5 per cent.; cork, 30 per cent. The product can be rolled into sheets and applied to fabrics, &c.—F. H. L.



## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Cotton Bleaching at* 140° C. A. Scheurer and A. Brylinski. Bull. Soc. Ind. Mulhouse, 1898, 68, 29—32.

Fents of unbleached calico were smeared with tallow, then immersed in dilute sulphuric acid (1 : 100) at 60° C. for an hour, washed, and saturated with the alkaline solutions employed in the experiments. They were then wrapped in bleached calico and placed in iron tubes, which were thereupon filled with the alkaline solutions, closed, and heated at 140° C. for four hours.

Experiments were made with mixtures of caustic soda, sodium carbonate, and resin, and of caustic soda and resin, and with caustic soda alone. The results showed that the influence of the resin was very considerable. It was found that a solution containing per litre 23 grms. of caustic soda at 38° B., 4 grms. of sodium carbonate, and 2½ grms. of rosin, was more effective in its action than one of 30 grms. of caustic soda at 38° B. The best result was obtained with a solution of 30 grms. of caustic soda at 38° B. and 2½ grms. of rosin per litre. With this the colouring matters present in the cotton were removed along with the fatty compounds.

It would thus appear that a complete bleaching of cotton in a single boiling operation, without mechanical circulation of the solutions used, is possible of attainment.—E. B.

### *Cotton Bleaching, Origin of Use of Soda Ash and Hydrochloric Acid in.* A. Scheurer. Bull. Soc. Ind. Mulhouse, 1898, **68**, 24—28.

An alkali carbonate, namely potassium carbonate, would appear first to have been employed in America for cotton bleaching (Dana, Bull. Soc. Ind. Mulhouse, 1837).

In the process originally adopted, it was applied three times in the course of the bleaching operations, its first application immediately following the lime-boil, and replacing the souring operation recommended by A. Scheurer-Rott (report on E. Schwartz's Memoir, Bull. Soc. Ind. Mulhouse, 1835). In this connection, Scheurer-Rott pointed out that alkali carbonates act on lime-soaps and transform them into calcium carbonate and alkali soaps, and that they thus differ essentially from caustic alkalis, which have no action upon them, the lime-boil forming, therefore, the basis of the process of bleaching with alkali carbonates.

The substitution of hydrochloric for sulphuric acid, previously suggested in 1834 by Gréau (Soc. d'Agric. &c. du département de l'Aube) in the processes of linen bleaching, was effected as regards cotton bleaching by A. Scheurer-Rott.

E. Schwartz and A. Scheurer-Rott recommended that the operations of "chemicking" between the ley-boils should be suppressed, and that the tissues should be thoroughly freed from fatty matters before being treated with bleaching powder.—E. B.

### *Carbon Tetrachloride, Application of,* S. Sommerfeld. Leipziger Färber- u. Zeugdrucker-Zeit. **47**, [5], 223—224.

Carbon tetrachloride, which for years has been made only for laboratory use, is now brought into the market at a relatively low price. One valuable property of carbon tetrachloride rests on the fact that it can be heated and distilled without any danger, which is more than can be said of petroleum spirit.

It dissolves fats, oils, resins, &c., and hence, like petroleum spirit, it can be used for removing grease stains; but, unlike petroleum spirit, there is, after using carbon tetrachloride, no halo left after the stain has disappeared.

It dissolves fatty colour lakes easily—a property which the author has endeavoured to utilise by employing the solutions of colour lakes in carbon tetrachloride for dyeing silk goods according to the so-called dry-process, *e.g.*, gauzes, laces, ribbons, &c.

Basic dyes are used in the formation of the fatty colour lakes, of which the author gives the following as an example:—

Take 400 grms. of resin, 50 grms. of caustic soda, 100 grms. of sodium carbonate, 3 litres of water; add a solution of 25 grms. of basic dyestuff dissolved in 2 litres of water, heat gently, add a solution of aluminium sulphate (100 grms. per litre), allow to stand, filter off the precipitate, dry, and dissolve in a warm solution of carbon tetrachloride.

Goods dyed with this colour lake solution are fast to washing, but possess a rather harsh feel; a softer "handle" and particularly brilliant shades are obtained with olein lakes.

These olein lakes are made by adding an olein soap to the solution of the basic dyestuff, and precipitating as above with aluminium sulphate.

An addition of stearin or paraffin in the formation of an olein lake might possibly make the lake applicable for waterproof fabrics. Thus, the two operations of dyeing and making the waterproof could be combined into one.

—J. E. H.

### *Indigo, Synthetical and Natural.* Chem. Ind. 1898, **21**, [8], 149—150.

Berzelius showed that Indigo constantly contains, besides Indigotin and mineral matters, indirubin, indigo-brown, and Indigo-gluten ("Indigleim").

He observed that Indigo-gluten is dissolved by acids, and resembles in its properties vegetable gluten, to which he assumed it to be related; that indigo-brown is a nitrogenous compound, combining with bases and giving an insoluble compound with lime, in which form it is commonly found in indigo; and that indirubin dyes wool brownish-red, gives a colourless reduction compound, and under reduced pressure sublimes unchanged.

Schützenberger has also asserted (Traité des Matières Colorantes, 563) that indigo containing indirubin, yields, in dyeing, a shade of blue which is more purple than that which is obtained from indigotin alone.

These statements would appear to have led to the opinion that the above compounds are of importance in cotton-dyeing, in spite of the fact, which seems hitherto to have been overlooked, that P. Schwarzenberg and G. Schwartz (Bull. Soc. Ind. Mulhouse, 1837, **10**, 413, 425) have shown that the contrary is the case.

The conclusions of these chemists, deduced from an exhaustive series of trials on both the small and large scale, are as follows:—

1. The greater part of the Indigo-gluten passes into solution in the Indigo vat; a small quantity remains in the vat-sediment. This compound is without influence in dyeing.
2. Indigo-brown forms an insoluble compound with lime and remains in the sediment. It, too, has no effect on Indigo dyeing.
3. Indirubin dyes to some extent, but it increases neither the fastness nor the brilliancy of shade of the dye; the effect, indeed, of a large proportion of it, is to give a shade which is dull and unpleasing.

These conclusions have recently been confirmed by the chemists of the Badische Anilin und Soda Fabrik, who find, moreover, that the fastness to rubbing and washing of their new synthetical indigotin, when dyed on cotton fabrics in the ordinary manner, is equal, if not slightly superior, to that of natural indigo. The shade and fastness of indigo dyes, they state, are not influenced by indirubin, Indigo-gluten, and other organic substances present in indigo, but simply by the methods of setting and working the vats; further, the effect upon dyeing of the glue, &c., recently recommended (this Journal, 1898, 41), is as great with ordinary, natural indigos as it is with "Indigo pure."

Incidentally it is mentioned that the proprietors of the Badische Anilin und Soda Fabrik are in a position to manufacture, by a synthetical process, indirubin, but that for the present they are refraining from doing so.—E. B.

### *Paranitraniline Red, Improved Method of Dyeing.* A. G. Green. J. Soc. Dyers and Colourists, **14**, [4], 1898, 95—98.

In Eng. Pat. 28,499 of 1896 (this Journal, 1897, 1014) the author claimed the method of employing mercerised cotton in dyeing Paranitraniline Red, stating that much fuller shades are thereby obtained, the colour produced also being much faster to light. Further details in connection with this process are given in the present paper, and experiments are adduced showing that after mercerisation the cotton acquires a decided affinity for $\beta$-naphthol, and, in fact, better shades are obtained with 1 per cent. of $\beta$-naphthol on mercerised cotton than with 2 per cent. when dyed in the ordinary way. Similarly, too, a smaller quantity of *p*-nitraniline is required, the theoretical amount of *p*-nitraniline for 1 per cent. of $\beta$-naphthol is 0·96 per cent., but allowing for that removed by the cotton, about $1\frac{1}{3}$ per cent. is required in actual practice. In order to mercerise the cotton, it is immersed for one hour in a cold caustic soda lye, maintained at 30° Tw. During this operation the weight of solid caustic soda taken up by the cotton is about 30 per cent. of the weight of the latter, the fibre contracting about 17 per cent. of its length, and becoming proportionately thicker. After washing and hydro-extracting, the cotton is padded, 2 lb. at a time, in a bath containing 0·25 gall. of a $\beta$-naphthol solution in 2·25 galls. of cold water, the bath being refreshed for each 2 lb. of cotton by the addition of 2·75 oz. of the naphthol solution. The yarn is then hydro-extracted and dried upon a Haubold's rotatory drying machine. The developing bath is prepared as follows:—Stir together 2 lb. of *p*-nitraniline and 0·5 gall. of cold water to a paste, then add 0·4 gall. of hydrochloric acid (35° Tw.) or 0·5 gall. of 28° Tw., stir for five minutes, and add 1 gall. of cold water. To this mixture is then added, with continuous stirring, 1·125 lb. of sodium nitrite in 0·5 gall. of water, and stirring is continued for 10 minutes. This solution should turn starch iodide paper blue, and is then made up to 6 galls. In this form, the diazo solution is stable and will keep for 1—2 weeks. When required for use, mix 0·5 gall. of the diazo solution, 2·5 galls. of cold water, and 0·5 gall. of acetate solution (3·625 lb. of sodium acetate and 0·625 lb. of soda ash, made up to 6 galls. with cold water). Each 2 lb. bundle of dry grounded yarn is worked for half a minute in this bath and squeezed off over the bath, refreshing the latter each time with 0·75 lb. of diazo solution and 0·75 lb. of acetate solution. The cotton, after developing, is allowed to lie for 10 minutes, and is then washed off in cold water, and finally soaped at 60° C. in a 0·25 per cent. soap bath, rinsed, and dried. With grey cotton, the brilliancy can be improved by a subsequent bleach with chloride of lime. Exclusive of labour, the cost of dyeing amounts to 0·9 of a penny per lb., against 1·0 penny by the old process.—T. A. L.

*Affinity of Wool for Colouring Matters, New Method of Increasing the.* E. Knecht. J. Soc. Dyers and Colourists, **14**, (4), 1898, 98—100.

According to an observation of Kertesz, wool, when treated with a very strong solution of caustic soda, followed by a treatment with ammonia, acquires a greatly increased affinity for dyestuffs, whilst not materially suffering in strength. The goods are printed with a mixture of 100 parts of soda-lye (75° Tw.), 100 parts of tragacanth solution (1 : 100), 75 parts of British gum, and 150 parts of glycerin. They are then passed through a bath charged with 40—50 lb. of ammonia per 100 galls., washed, and dyed. The author was more successful by substituting for the former bath equal parts of caustic soda (80° Tw.) and British gum (1 : 1), and obtained excellent results with such colours as Acid Green, Acid Magenta, and Naphthol Yellow S. Bontrock (this Journal, 1898, 452) has shown that strong caustic soda increases the breaking strain of woollen yarn. A yarn breaking at 610 grms. was immersed for 10 minutes in caustic soda of different strengths, washed in water, then in acid, dried, and tested again. At 4° B. the strength had decreased to 570, at 10° to 430, at 16° to 180, and at 20° to 95, whilst at 30° it rose to 335 grms., at 40° it was 770, and at 42° B. it attained a maximum of 815 grms. As a matter of fact, wool is more readily affected by weak than by strong alkalis; a yarn heated with caustic soda (40°—50° Tw.) on the water-bath for several hours, did not dissolve, but when the solution was diluted to 10° Tw., the action was completed in a short time. It is well known that wool, when chlorinated, treated with dilute sulphuric acid, or steamed, has an increased affinity for dyestuffs, and the author suggests, in view of his previous work on the subject, that a partial disintegration of the wool fibre takes place, forming products which are chemically more active towards dyestuffs. (See also this Journal, 1898, 242.)—T. A. L.

*Dyestuffs, The Mixing or Combination of, in the Dyebath.* Leipziger Färber u. Zeugdrucker-Zeit. **47**, [5], 201—204.

In the mixing or combination of dyestuffs in the dyebath, only very few dyers make use of any scientific knowledge. Most dyers use a mixing so long as it gives the correct tone of colour in dyeing, but they pay little attention to the results which the succeeding baths give, or to the effects which the finishing processes that follow, exercise upon the shade produced.

It is a well-known fact that basic and acid dyes, or basic and substantive dyes, may not be mixed together in the dyebath. To obtain the best results therefore, the effects of the different dyestuffs upon each other must be carefully noticed. Under no circumstances must dyes be used for a compound shade, which have the slightest tendency to precipitate each other in solution. In very few cases only, do the acid dyes precipitate each other in solution.

A very important point to be considered, and one which is usually overlooked, is the relative proportions in which the different dyes are exhausted from a mixture of their solutions. Hence it is best to select these dyes for a certain mixture, which are all practically uniformly exhausted in the dyebath, that is, which are all equally attracted by the fibre.

This question of exhaustion is especially important in the case of cotton dyeing. Here, in dyeing with substantive dyes, the baths are not exhausted, and hence are preserved for further use. It is therefore quite evident that a dyestuff which does not exhaust so quickly as the others in the same dyebath, accumulates in the dyebath, and consequently the dyeings of the different batches differ in shade and approach gradually to the shade of the dyestuff which is only slowly exhausted.

Again, attention must be paid to the method of dyeing. Only such dyes as are similar in their method of application must be selected.

For instance, in cotton dyeing, some substantive dyes require the presence of a salt of phosphoric acid for the best results, whilst the presence of this salt is decidedly injurious for other substantive dyes.

Further, dyers give too little consideration to the fastness of the colour produced by the dyestuff which they employ only in small quantities for obtaining a compound shade. But, as the author remarks, if the presence of a dyestuff in such small proportions is actually necessary in order to get the desired result, then particular attention ought to be paid to its relative fastness towards light, washing, milling, steaming, &c.: in short, the colour must possess the same fastness as the other constituents of the mixed shade.—J. E. H.

*Substantive Dyestuffs with p-Nitraniline and Copper Sulphate; Development of.* E. Grossman. Färber-Zeit. **9**, [10], 151.

Certain colours, which are fast to washing, can be produced on cotton by developing substantive dyes with diazotised *p*-nitraniline. This process is simple and inexpensive, and, indeed, produces deeper shades. It possesses, however, the disadvantage that it is applicable only in the case of very few substantive dyes, whilst the fastness to light of the colours produced by this method is diminished almost without exception.

The author modifies and improves the above method by adding 3 per cent. of copper sulphate to the solution of the diazo-*p*-nitraniline. The advantageous effect produced by this addition is especially prominent in the case of Congo Brown G, Chromanil Brown R and 2 G, Chrysamine G. Chicago Blue B, Zambesi Black B R and D, and Columbia Black B.

Also such dyes as Columbia Brown R and Toluylene Orange R, which, on developing with diazo-*p*-nitraniline alone, give colours comparatively fast to washing, are still further improved by the addition of copper sulphate. Further, dyes like Congo Brown G, Chromanil Brown 2 G and R, Chicago Blue B, Zambesi Black B R, which, by the after-treatment of the direct dyeings with certain metallic salts, are already fast to light, also retain this superior fastness to light when treated according to the author's method.

For yellow shades, Chrysamine G is said to be much preferable to Primuline, as it is very fast to light and washing.

The author's process is carried out by developing the dyed material (which has been dyed directly after the manner of substantive dyes) for half an hour in the cold in a bath containing 1 per cent. of diazotised *p*-nitraniline, to which is added the requisite amount of sodium acetate and 3 per cent. of copper sulphate.

The developing proceeds quite smoothly and evenly without the observance of any special precautions; indeed, the developing bath may be used even when its temperature is at 25° C.—J. E. H.

*Paranitraniline Red and White Discharge Prints on Indigo-dyed Calico.* L. Bloch, E. Zeidler, and E. Brandenberger. Bull. Soc. Ind. Mulhouse, 1898, **68**, 50—56 and 56—57.

The destruction of the Indigo is effected by the oxidising action of alkali chlorates and ferricyanides under the influence of steam.

Tissues dyed pale shades of Indigo are padded with a solution of 1,700 grms. of "$\beta$ naphthol R," 1,700 grms. of caustic soda at 40° B., 40 litres of water at 40° C., 5 litres of Turkey-red oil, 10 kilos. of sodium chlorate dissolved in 40 litres of water at 40° C., 2 litres of ammonia solution, and 2 kilos. of sodium acetate, the whole being diluted to 100 litres. They are then printed with the following colour-mixtures :—*White Discharge.*—640 grms. of thickening (starch and gum tragacanth), 150 grms. of sodium chlorate, 80 grms. of sodium ferrocyanide, and 130 grms. of tartaric acid. *Red Discharge.*—4¼ litres of "diazo solution" (see below), 4,330 grms. of thickening (starch and gum tragacanth), 1,000 grms. of sodium chlorate, 260 grms. of potassium ferricyanide, and 210 grms. of tartaric acid. "*Diazo Solution.*"—1,320 grms. of Azophor Red P N (Höchst Farbwerke), 3½ litres of water, 600 c.c. of caustic soda at 22° B.

The printed pieces are passed two or three times through the steam aniline ageing machine, the steam being as dry as possible, and then for a minute, in the open width, into dilute caustic soda solution (1 per cent.) at 60° C. Then they are washed, soaped at 40°—50° C. for 20 minutes, again washed, and, if necessary, finally, "chemicked" slightly.

For darker shades of Indigo, more concentrated mixtures than the above are required.

The white discharge mixtures, in virtue of the large proportion of tartaric acid they contain, are able to resist Paranitraniline Red. When employing them for this purpose, however, it is advisable to increase the proportion of tartaric acid (in the mixture used for medium shades of blue, for instance, from 200 to 250 grms. per kilo.). By this means the mixtures resist the red discharge perfectly, at the same time effecting the discharge of the Indigo.

Dianisidine and Alizarin Blues may be resisted in a similar manner. Pale Indigo-dyed pieces are prepared and printed with the mixtures ordinarily used for dark Indigo shades, with the addition to the red discharge mixture of 20 grms. per litre of ammonium oxalate, when the tetrazotised dianisidine mixture is used, to prevent the copper salt present therein from injuriously affecting the red.

Brandenberger's method (Sealed Note, deposited June 30, 1897) is essentially the same as that above-described. The principal differences, apart from those in the proportions of the several reagents employed, consist in the employment of gum-arabic as thickening agent, of *p*-nitraniline, diazotised in the usual manner, in place of Azophor Red, and of sodium acetate in the red printing mixture—an addition which Bloch and Zeidler prefer to make to the prepare, on account of the rapid decomposition of the printing mixture which takes place in the presence of this salt.—E. B.

*Paranitraniline Red and White Discharge Prints on Indigo-dyed Calico.* Report on the foregoing two papers by E. Grandmougin. Bull. Soc. Ind. Mulhouse, 1898, **68**, 57—58.

The only method hitherto known for the production of red and white discharge-patterns on Indigo-dyed calico, is that of Kurz and Kunert (this Journal, 1897, 438; see also this Journal, 1898, 151). This method requires very careful attention to details to secure satisfactory results.

The method devised by Bloch and Zeidler, and Brandenberger, can only be carried out with medium shades of indigo blue. It also requires a careful adjustment of the proportion of oxidising agents employed, to the depth of engraving of the printing rollers and to the intensity of the shade of blue to be discharged, an excess of the agents being very injurious to the red. Good results may also be obtained by substituting Nitrosamine Red for Azophor Red and making other changes in the colour-mixtures used.

As regards the question of priority of discovery of the ferricyanide-chlorate method of discharging indigo, it would seem that the discovery was independently made at about the same time by different persons, the firm of L. Cassella and Co., having, perhaps, drawn attention to the possibility of the method, when they published, in May 1897, a circular showing the application of such a discharge in the case of Diamine Blue dyes.—E. B.

*Cerium Salts: Application in Dyeing and Calico Printing.* R. Goehm. Rev. Gén. des Mat. Col. 1898, **2**, [16], 135—136.

According to B. Kosmann (Ger. Pat. 15,509 of 1897), the nitrates and sulphates of cerium, lanthanum, and didymium, may be employed in textile colouring either as mordanting salts or in the production of mineral colours. The latter are formed on the fibre by impregnating with solutions of the salts named, and then passing into a bath of hydrogen dioxide rendered alkaline with ammonia or other alkali. The cerium salts may be used alone or in admixture with salts of copper, iron, &c., or tannic acid.

The author in 1878 made use of cerium salts to fix Alizarin Blue on cotton, employing for padding purposes a mixture of 500 c.c. of chromic acetate at 15° B. and 10 grms. of "cerium bisulphate," thickened with 50 grms. of dextrin, and, for printing, a thickened mixture of "cerium bisulphate" (20 grms. per litre) and sodium acetate (30 grms. per litre). The fabric was dried and passed through a fixing bath, to fix the mordant, and then dyed. Fairly bright, but pale, colours were obtained.

In a note subjoined to the original paper, L. Lefèvre states that Horace Koechlin has left some notes regarding the utility of cerium salts in dyeing and printing, which will probably be published.—E. B.

*Direct Dyestuffs, Discharge of, by Sodium Hyposulphite (Hydrosulphite).* E. Blondel. Rev. Gén. des Mat. Col. 1898, **2**, 134—135.

Azo dyestuffs are reduced and removed from dyed fabrics, without injury to the latter, by treatment with a hot, dilute solution of sodium hydrosulphite.

Yellow, blue, and black dyestuffs are decomposed easily. The reds—Congo, Benzopurpurin 4 B, and Paranitraniline Red—offer more resistance.

Aniline and Vidal Blacks are reduced to leuco compounds, which are readily oxidised, so that, by the use of sodium hydrosulphite, it is possible to approximately determine the respective proportions of these dyestuffs and of colouring matters, such as Diamine Blacks, in certain composite black dyes, and in black-dyed yarns spun from variously dyed fibres.—E. B.

## PATENTS.

*Damask and the like Fabrics, An Improved Process for Imitating, principally intended for Wall Coverings.* K. Gebhardt, Munich, Germany. Eng. Pat. 9560, April 14, 1897.

To produce an imitation damask, a fabric of jute, linen, cotton, or other suitable cheap material is dyed or painted in a bright ground shade, and a design in a strong bright colour is then printed on it. After drying, the same design is again printed, but this time in a dark colour, the pattern being slightly displaced from its former position in order to produce the effect of light edges. These light edges are, of course, broader or narrower according to the amount of displacement of the pattern.

Similar effects may be produced by double printing in the same manner on coloured fabrics having a brilliant surface—*e.g.*, satin or sateen—or on paper which has been previously printed with a suitable ground.—R. B. B.

*Chlorination of Wool, A Process for.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 11,917, May 13, 1897. (Compare Knecht and Milnes, this Journal, 1892, 131.)

Chlorine in excess, acts very energetically on wool, converting it into a white, pulpy substance, and evolving hydrochloric acid. Dry wool is attacked slowly, but the moister the wool the more violent is the reaction. If the quantity of chlorine, its concentration, and the amount of moisture on the wool be carefully regulated, its action can be controlled, so that the fibre is not in any way injured, but acquires those properties characteristic of wool which has been treated with hypochlorites, namely, a greater affinity for dyestuffs, greater transparency, gloss, and scroopy feel, while it loses its power of felting, and becomes slightly whiter in colour. The maximum quantity of chlorine required per kilo. of wool is 25 litres, and the minimum 5 litres.

By printing wool with thickened hygroscopic substances, such as calcium, zinc, or magnesium chloride, and exposing to the action of chlorine, only the printed parts will, under appropriate conditions, be attacked. Similarly, by printing wool with substances which, after drying or steaming, are difficult to wet—*e.g.*, thickened aluminium acetate—damping the goods, and chlorinating, the wool beneath the printed parts is protected, and thus resist styles may be obtained.

During the chlorination, which takes place in a gas-tight chamber, the goods are kept in constant motion, by a mechanical arrangement situated outside the chamber, to ensure the uniform action of the gas. After treatment, they are washed and soaped as usual.—H. I.

*Printing or Colouring Yarn and Similar Material, Impts. in Methods of and Apparatus for.* P. A. Newton, London. From The Alexander Smith and Sons Carpet Company, Yonkers, New York, U.S.A. Eng. Pat. 26,667, Nov. 15, 1897.

This patent describes a method of printing yarn in which a rubber works in combination with the colour wheel, and is caused to rub simultaneously portions of the stripe of yarn being printed and the next previously printed stripe. The rubber is of such a width that it acts upon more than one-half and less than the whole of each of the two stripes which are being simultaneously rubbed.—R. B. B.

*Application of Colours to Fabrics by Printing, Impts. in and connected with the, and in Apparatus to be employed therein.* H. Giesler, Molsheim, Alsace. Eng. Pat. 2198, Jan. 27, 1898.

Several improvements are described in the apparatus for printing colour on both sides of a fabric, the improvements being as follows:—

In order to obviate any difference between the colours of the two sides of the fabric, the two printing cylinders are separated to such an extent that a drying apparatus may be placed between them, and the fabric is dried after printing the first side and before printing the second side.

Instead of surrounding each cylinder with linen or cotton to form an elastic cushion for the fabric to be printed upon, an endless band of felt or like material is arranged on each cylinder, and is carried along by the cylinder.

After the fabric has been treated with tin, chlorine, &c., before printing, it is exposed in the damp state to the action of steam, to render the fabric smoother and better able to absorb the colour.

To produce different depths of the same colour, the number of rollers is varied, but the concentration of the printing colour is kept always constant for the same fabric.
—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Hydrofluoric Acid: Aqueous, Percentage Content of HF in.* Chem. Zeit. 22, [24], 225.

The existing lack of a table showing the percentage of hydrogen fluoride in aqueous hydrofluoric acid of various densities is remedied by the following, drawn up in the laboratory of J. L. C. Eckelt, of Berlin:—

| B. | Sp. Gr. | HF. | ° B. | Sp. Gr. | HF. | ° B | Sp. Gr. | HF. |
|---|---|---|---|---|---|---|---|---|
| | | Per Cent. | | | Per Cent. | | | Per Cent. |
| 1 | 1·0069 | 2·32 | 16 | 1·1239 | 32·78 | 31 | 1·2716 | 66·61 |
| 2 | 1·0139 | 4·04 | 17 | 1·1326 | 35·15 | 32 | 1·2828 | 68·76 |
| 3 | 1·0211 | 5·76 | 18 | 1·1415 | 37·53 | 33 | 1·2943 | 70·91 |
| 4 | 1·0283 | 7·48 | 19 | 1·1506 | 39·91 | 34 | 1·3059 | 73·06 |
| 5 | 1·0356 | 9·20 | 20 | 1·1598 | 42·29 | 35 | 1·3177 | 75·21 |
| 6 | 1·0431 | 10·92 | 21 | 1·1691 | 44·67 | 36 | 1·3298 | 77·36 |
| 7 | 1·0506 | 12·48 | 22 | 1·1786 | 47·04 | 37 | 1·3421 | 79·51 |
| 8 | 1·0583 | 14·04 | 23 | 1·1883 | 49·42 | 38 | 1·3546 | 81·66 |
| 9 | 1·0661 | 15·59 | 24 | 1·1981 | 51·57 | 39 | 1·3674 | 83·81 |
| 10 | 1·0740 | 17·15 | 25 | 1·2080 | 53·72 | 40 | 1·3804 | 85·96 |
| 11 | 1·0820 | 18·86 | 26 | 1·2182 | 55·87 | 41 | 1·3937 | 88·10 |
| 12 | 1·0901 | 21·64 | 27 | 1·2285 | 58·02 | 42 | 1·4072 | 90·24 |
| 13 | 1·0983 | 24·42 | 28 | 1·2390 | 60·17 | 43 | 1·4211 | 92·39 |
| 14 | 1·1067 | 27·20 | 29 | 1·2497 | 62·32 | 44 | 1·4350 | 94·54 |
| 15 | 1·1152 | 29·98 | 30 | 1·2605 | 64·47 | 45 | 1·4493 | 96·69 |

—C. S.

*Perchloric Acid and its Salts, The Electrolytic Production of.* F. Foerster. Zeits. f. Elektrochem. 4, (16), 386—388.

The author refers to a previous paper by Mewes and himself upon the electrolytic production of iodoform (this Journal, 1898, 66), in which the reduction of iodates to iodides at the cathode had received notice. Since this reduction is in opposition to the results obtained by Oettel in his experimental work with chlorates (this Journal, 1895, 492), the author has carried out some investigations upon the electrolysis of chlorates. He finds that in neutral or in alkaline solution, potassium chlorate is not appreciably reduced to chloride at the cathode when the latter is of platinum, lead, copper, zinc, or nickel. This reduction is, however, very strong when the cathode is of soft iron or cobalt.

He also noted that in all cases, in neutral or in alkaline solutions, the chlorate could be oxidised to perchlorate at the anode. In order to determine the most favourable conditions for the production of perchlorates, the author used an experimental cell which permitted the gases liberated at both anode and cathode to be measured and analysed, and their volume and constitution compared with that of the gases liberated in a detonating voltameter included in the cell circuit. In this manner the deficiency in the amount of oxygen liberated from the experimental cell could be determined, and the oxidation effected at the anode ascertained. Neutral solutions of sodium and potassium chlorate were used. The former was found to be the more suitable for the purpose, since it is the more soluble of the two in cold water, and concentrated solutions yield the best results. The experimental data are given in tabular form, and cover variations in the temperature, time, concentration, and current density.

The best results were obtained with a 17 per cent. solution of sodium chlorate, which yielded a mean current efficiency of 89½ per cent. when a current density of 156 ampères per sq. ft. was employed. A current efficiency of 98 per cent. was obtained in another experiment with a 50 per cent. solution of sodium chlorate, when a current density of 78 ampères per sq. ft. and an E.M.F. of 4½ volts, with the electrodes 1·5 cm. apart, were used. The temperature of the electrolyte in these experiments was 17—20° C.; with a heated solution the results were less satisfactory.

The sodium perchlorate can be converted into potassium perchlorate by the addition of potassium chloride to the electrolysed solution. The potassium salt separates out in small crystals. A sample of the product yielded 45·6 per cent. of oxygen, as against 46·2 per cent.—the theoretical figure.

Using potassium chlorate in place of sodium chlorate, a current efficiency of 82 per cent. was obtained, but after one-half of the chlorate had been oxidised, the efficiency quickly fell away.

The production of perchlorates can occur equally well in acidified solutions of the chlorates, but in this case the reducing action at the cathode is more vigorous.

The direct production of perchlorates from chlorides is not considered by the author to be practicable, as special conditions are requisite to obtain the best yield of the intermediate products—sodium or potassium chlorate—from their respective chloride salt solutions.—J. B. C. K.

### *Hyperborates and Hypertitanates.* P. Melikoff and L. Pissarjewsky. Ber. **31**, [7], 953—956.

In this paper the authors give further particulars of the salts of hyperboric acid ($NaBO_3 + 4H_2O$) and hypertitanic acid (Ber. **31**, [4], 678). The ammonium salt of the former—$NH_4BO_3 + \frac{1}{2}H_2O$—is prepared by dissolving the boric acid in a 2·5 per cent. aqueous solution of hydrogen peroxide, adding ammonia, and precipitating the salt with alcohol. It forms white crystals, and when dry is very stable. Like the sodium salt described in the former paper, it evolves hydrogen peroxide on treatment with dilute sulphuric acid, ozone with concentrated sulphuric acid, and chlorine with hydrochloric acid. It dissociates in aqueous solution at the ordinary temperature and more rapidly at 45° C., with the liberation of oxygen and formation of ammonium nitrate. The freshly precipitated salt contains 3 mols. of water of crystallisation, but on standing for 24 hours over sulphuric acid it only retains a half-molecule. The barium salt [$Ba(BO_3)_2 + 7H_2O$] is prepared by double decomposition of a saturated solution of the sodium salt (cooled to zero) with barium chloride. It is with difficulty soluble in water, and when treated with acids, behaves in a similar manner to the ammonium salt. It slowly undergoes decomposition at the ordinary temperature. The calcium salt, obtained in like manner, is much more unstable in water than the barium salt. It is equally insoluble. The insoluble nickel and copper salts, obtained by the action of their respective sulphates on the sodium salt, decompose rapidly with the liberation of oxygen, and this fact, as also in the case of the calcium salt, prevented the authors from obtaining an accurate analysis of their composition.

By the action of cobalt chloride on the sodium salt, the higher oxide of cobalt ($CoO_2.CoO$) is formed with the liberation of oxygen, and a similar reaction takes place between the sodium borate and manganese salts. All attempts to form compounds of hyperboric acid with metallic peroxides were unsuccessful.

Hypertitanic acid forms with metallic peroxides, not only basic salts of the type $(R_2O_2)_2.TiO_3$, but also neutral salts, $R_2O_2.TiO_3$. Normal sodium peroxide hypertitanate [$Na_2O_2.TiO_3 + 3H_2O$] is obtained either by the action of hydrogen peroxide and caustic soda solution on the titanic acid, and decomposition of the resulting solution with alcohol, or by decomposition of the basic salt with water. In both cases a thick oily liquid separates, which changes to a yellow powder on drying over sulphuric acid. Dilute sulphuric acid liberates hydrogen peroxide from this salt, and hydrochloric acid is decomposed by it with the liberation of chlorine. Ammonium peroxide hypertitanate [$(NH_4)_2O_2.TiO_3.H_2O_2$] is prepared by pouring a solution of hydrogen peroxide over hypertitanic acid, adding ammonia, and precipitating the salt with alcohol. It crystallises in thin yellow prisms, and when dry is tolerably stable. It dissociates in aqueous solution with the evolution of oxygen. On warming over a naked flame it gives feeble explosions. Dilute sulphuric acid and hydrochloric acid act upon it in the same way as on the sodium salt. Normal barium peroxide hypertitanate ($BaO_2.TiO_3 + 5H_2O$) is prepared by double decomposition of the sodium salt with barium chloride. When dried over sulphuric acid it is a yellow powder.—C. A. M.

### *Soda Ash and Hydrochloric Acid: Origin of use in Cotton Bleaching.* A. Scheurer. Bull. Soc. Ind. Mulhouse, 1898, **68**, 24.

*See under* VI., *page* 573.

### *Cylinders containing Liquefied Ammonia, The Prevention of Explosion of.* A. Lange. Chem. Ind. 1898, **21**, [10], 191.

*See under* I., *page* 562.

### *Boric Acid Determination.* T. S. Gladding. J. Amer. Chem. Soc. 1898, **20**, [4], 288.

*See under* XXIII., *page* 606.

### *Tartaric Acid in Crude Materials, Estimation of.*

*See under* XXIII., *page* 609.

### *Sulphurous and Thiosulphuric Acids, The Detection and Estimation of.* W. Autenrieth and A. Windaus. Zeits. Anal. Chem. **37**, [5], 290.

*See under* XXIII., *page* 606.

### *Fuming Sulphuric Acid and Sulphuric Anhydride, Estimation of.* R. Rosenlecher. Zeits. Anal. Chem. **37**, [4], 209.

*See under* XXIII., *page* 606.

### *Phosphoric Acid, Estimation of, by Titration.* A. Hebebrand. Zeits. Anal. Chem. **37**, [4], 217.

*See under* XXIII., *page* 607.

### *Chili Nitre, Perchlorates in, Microchemical Detection of.* M. van Breukelween. Rec. Trav. Chim. Pays-Bas, **17**, 94; Chem. Centr. 1898, **1**, [16], 960.

*See under* XXIII., *page* 602.

## PATENTS.

### *Product, A New or Improved, adapted for use in Dyeing Wool, in Aerating Bread or Pastry, and for other Purposes.* G. Wendler, Manchester. Eng. Pat. 10,933, May 3, 1897.

The product is prepared, either by acting upon sodium or potassium acid sulphate or acid phosphate with boric acid with the aid of heat; or by acting upon sodium or potassium borate with sulphuric or phosphoric acid. Preferably, 100 parts of calcined sodium sulphate are added to 60 parts of "concentrated commercial sulphuric acid" which has been heated to the boiling point of the acid, and when complete solution is effected, about 15 parts of boric acid are added. After cooling, the mass may be broken up and packed for use as described in the title.—E. S.

### *Sodium Chlorate, Impts. in the Manufacture of.* A. R. Davis, Manchester. Eng. Pat. 12,534, May 20, 1897.

Dry sodium carbonate is caused to travel through a mechanical apparatus, whilst a current of dry chlorine is passed in an opposite direction. The resulting mixture of sodium chloride and chlorate is methodically lixiviated with hot water in jacketed tanks, the further separation being effected by concentration and crystallisation.—E. S.

### *Acetic Acid, Impts. in the Manufacture of.* E. G. Scott, Liverpool. Eng. Pat. 12,952, May 25, 1897.

Crude acetic acid, containing about 10 per cent. of pure acid, is heated in a still, provided with steam coils, to about 100° in a vacuum. The effect of this moderate heat is, that the acid and water distil, the tar, at the low temperature, remaining behind. The strengthening of the acid is effected as follows:—The vapours coming over from the still enter a vessel containing a coil, in which a portion of the vapours is condensed, either by radiation from the vessel or by cold water passing through the coil. Or, if radiation produces too much cooling, steam may be put through the coil. The uncondensed portions enter a second vessel, where a further condensation takes place, and so on, the vapours passing through a series of vessels. The last vessel is kept cold, so that everything not previously condensed, condenses there before reaching the vacuum pump. The vapour passing from one vessel to another, bubbles through the liquid, the effect being that more acid is condensed than steam, and so the first vessel is the strongest in acid, and the others proportionately weaker, and so on, until the last one is reached, which contains practically only water. When the liquid in the first vessel is strong enough (say 25 per cent.) for use, it is removed, and the contents of the second vessel are put into the first, and so on. By means of this arrangement, the use of lime is avoided, together with the subsequent evaporation and admixture of mineral acid.—D. B.

*Barium Oxide and Barium Hydroxide, Process and Apparatus for Obtaining, from Barium Carbonate.* W. Feld, Linz, Rhein, Germany. Eng. Pat. 13,519, June 1, 1897.

In converting barium carbonate into oxide, a difficulty is said to arise, due either to the access of matters capable of fluxing barium oxide, thus forming an impervious superficial coating on the lumps to be converted, or to admission of aqueous vapour to the kiln, whereby a coating of fusible barium hydroxide is formed. According to one process the barium carbonate is to be burnt in a kiln having a basic lining in the zone of greatest heat, and protected from leakage of air through the walls by an air-tight iron casing. Coke or anthracite, free from moisture, is used as fuel, air being supplied through tubes to the lower part of the kiln, where it is dried by contact with the barium oxide which has reached that part. The waste gases resulting from the heating of the barium carbonate are utilised in drying fresh barium carbonate and fuel. The fourth claim is for an apparatus "consisting of a chamber-kiln with a generator next to each chamber, and so arranged that the waste gases from the burning process shall heat the material to be burnt, and the fuel in other chambers, in order to dry them, and that the air for the combustion of the fuel shall be dried by being passed through already burnt barium oxide." It is stated that about 96 per cent. of the barium carbonate used may be decomposed by this process, as against a yield of from 40 to 50 per cent. obtained by the processes formerly used.—E. S.

*Sulphur Baths, Impts. in or relating to the Production of.* E. G. Ballard, Chester, and D. Herman, Prescot. Eng. Pat. 13,623, June 2, 1897.

The bisulphate and hyposulphite (thiosulphate) of an alkali metal are brought in contact with each other and the water of the bath, immediately before or at the time when the bath is being used, sulphur in a state of fine division and sulphurous acid being thus liberated. Two ounces of each salt may be dissolved in from 10 to 20 galls. of water. For local applications stronger solutions may be used. The two salts may be separately put up in the required quantities in suitable packages as an "article of manufacture."—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Pottery Decoration by the Resist Method.* W. Schiller. Thonwaaren-Ind. 12, [13], 196.

Instead of merely applying coloured glaze to the articles previously coated in places with a fatty resist, it is proposed to employ solutions of metallic salts, the articles being dipped therein and then fired. In this manner all the portions left uncovered by the resist are coloured, and the whole can then be glazed, thus producing a smooth surface and obviating the risk of melting in the final firing, the glazes usually applied in the first operation.—C. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Lime Burning, Chemical Reactions occurring in.* — Herzfeld. Thonind. Zeit. 22, [31], 374—376.

The author finds that calcium carbonate can be converted into hydrate in presence of superheated steam at a temperature of 700° C., whilst, with admission of air alone, a heat of 1,000° C. is required. Slaked lime can also be requickened by being steam-heated to 800° C. Unfortunately, no means of successfully applying this method in practice has yet been discovered, owing to the rapid corrosion of iron pipes and even firebrick in the superheating apparatus at those temperatures. The employment of ordinary steam with a view to disintegrating the contents of the kiln is unadvisable, the same effect being more cheaply accomplished by mechanical means.

In investigating the cause of so-called "dead burning," he finds that this result is chiefly due to silica, the presence of 8 per cent. of this substance sufficing to make the lime burn dead when subjected to a temperature of about 1,000° C. for some time. Alumina is less harmful in this respect, requiring much greater heat when alone; though in association with silica it gives rise to hydraulic lime. Iron greatly facilitates this latter reaction, so that the kiln temperature necessary for the formation of hydraulic cement varies inversely with the amount of iron present.

Pure calcium carbonate, on the other hand, does not burn dead under the influence of overheating. At 1,600° C. it sinters and becomes useless for the purposes of the sugar manufacturer, but only by reason of its diminished capacity for slaking rapidly; however, when sufficient time is allowed, it absorbs water and slakes perfectly.—C. S.

*Cement, Considerations regarding the Hardness of, and the Method for its Determination.* Eug. Ackermann. Chem. Zeit. 22, [40], 403.

In judging of the value of a cement, the author lays stress upon the consideration of the final hardness of the mortar, rather than upon the degrees of hardness attained at intermediate periods. A case is cited of a cement supplied to the French Government which failed to set sufficiently hard in seven or 28 days to satisfy the official tests, and yet showed excellent results at the end of three and six months. In this particular case also the minimum proportion of sulphuric acid allowed by the French Government—1 per cent.—was exceeded. The author observes that many excellent English and German cements contain as much as from 2 to 3 per cent. of calcium sulphate.—H. H. B. S.

### PATENTS.

*Paint or Composition to be used as a Coating, more especially in the Manufacture of Artificial Slates, and for Coating Tiles, Plates, and other Articles.* C. A. Hakanson and J. H. Hakanson, both of Kalmar, Sweden, and A. R. Hakanson, Karlshaum, Sweden. Eng. Pat. 8510, April 2, 1897.

The composition consists of finely powdered sandstone, sand, or similar material, linseed oil, boiled glue, turpentine, and a suitable drier, such as cerotine, with or without the addition of special colouring matters. It may be applied to stiff materials, such as pasteboard, or to pliable materials, such as leather, cloth, &c., in the production of plates or tiles to be used for inlaid work, or for making imitation marble, parquet flooring, &c. A priming is first put on and afterwards a second coat, which is worked up to a glossy surface by the application of suitable pressure.
—H. H. B. S.

*Plastic Materials, Impts. in the Manufacture of.* A. G. Hæhre, Hadeland, Oestre Lunner, Norway. Eng. Pat. 9105, April 13, 1897.

This is a plastic composition consisting of wood-pulp and hydraulic cement or similar materials mixed with solution of water-glass. It rapidly hardens, and is both fire- and waterproof, and is suitable for moulding building materials and for a variety of other purposes.—H. H. B. S.

*Porous Substances, Impts. in or relating to the Production of.* W. P. Thompson, High Holborn, Middlesex. From M. Rosenbaum, Stassfurt, Germany. Eng. Pat. 15,170, June 24, 1897.

The object of this invention is the production of porous substances, by mixing cement or other material with a substance capable of being afterwards sublimed. Naphthalene has been found suitable for the purpose.—H. H. B. S.

*Artificial Stone Compositions, Impts. in ; applicable also to Cements or Plasters.* W. F. Dyson, Clapham Common, Surrey. Eng. Pat. 10,317, April 26, 1897.

This invention consists in the production of an artificial stone in imitation of natural red sandstone by mixing a red aggregate with cements or plasters. The aggregate is prepared by impregnating sand with a solution of iron sulphate and heating to redness. The colour may be varied, if required, by the addition of iron oxide.—H. H. B. S.

*Cement, Impts. in the Manufacture of.* C. E. Lee and C. F. Lawton, both of New York, U.S.A. Eng. Pat. 24,179, Oct. 19, 1897.

To 3 galls. of a 17 per cent. solution of silicate of soda are added, in the form of powder, 3 lb. of calcium carbonate, 12 lb. of quartz, and 3 lb. of a compound made by fusing a mixture of 60 parts of sand, 28 parts of lime, 34 parts of alumina, and 5 parts of calcined carbonate of soda. After the foregoing ingredients have been mixed together and ground, 1 lb. of ground anhydrous antimonic oxide in 10 oz. of water is stirred in. The product is to be used for cementing sand, gravel, &c. to bricks, or for giving a smooth finish to brickwork.—H. H. B. S.

## X.—METALLURGY.

*Stamp Mills [Gold], The Minimum Amount of Water required for.* Eng. and Mining J. 1898, **65**, [16], 459.

Two samples of pulp were tested with a view of ascertaining the amount of water required for complete saturation. The samples had been ground to pass a 60-mesh screen in a dry process mill, and after treatment, had remained some months under cover, and were to all appearance free from moisture. They were each treated with water to such an extent that when placed in a pile on a slightly inclined surface, a little water would drain from the mass. The first sample, which was ground from a very soft ore, probably talc, required 96 galls. of water per ton. The second sample was of a red-brown colour and much harder than the first sample, and required 66 galls. of water per ton. —A. S.

*Gold Ore Slimes, Filter-Press Treatment of.* W. McNeill. Inst. Mining and Metallurgy, May 18, 1898.

The author advocates the use of the filter press in the extraction and washing of slimes, as opposed to the coagulation and decantation process, especially in cases where water is scarce and where the ores, when crushed, produce an excessive proportion of slimes, such as in the case of the soft talcose clayey ore to be met with in the oxidised zones in the West Australian goldfields. He briefly describes the treatment of the slimes at the Hannan's Brownhill Mine, where they are obtained by the dry crushing of the ore and dry separation from the sands in a Mumford and Moodie separator. The advantages of the dry separation, beyond the saving of water, are the assurance of the fine state of division of the gold and the retention of the float-gold in the slime.

The next treatment consists of cyaniding, and running the sludge into a receiver, whence it is forced by compressed air, at a pressure of 60 to 80 lb. per sq. in., into a filter-press so constructed that the complete percolation of the slimes with water and washing is successfully accomplished without the formation of channels. The cake produced retains only about 20 per cent. of water, as compared with 50 per cent. in the decantation method, and furthermore the washing is effected with a comparatively much smaller quantity of water. The time taken in filling the press, washing the cake, discharging and closing the press again, is 50 minutes, and the cost is considered less than that for working chemical precipitates, sewage sludges, &c. to a corresponding extent in the filter-press, owing to the greater weight per cb. ft. of the resulting slime-cake. —A. W.

*[Gold.] Slime-Pulp Formulæ.* W. A. Caldecott. J. Chem. and Met. Soc. of South Africa, **1**, [3].

| —— | Percentage (by weight) of Dry Slimes in Pulp. | Percentage (by weight) of Solution in Pulp. | Ratio (by weight) of Solution to 1 of Dry Slimes. | Volume (in cb. ft.) of 1 ton of Pulp. | Weight (in lb.) of 1 cb. ft. of Pulp. | Weight (in lb.) of Dry Slimes in 1 cb. ft. of Pulp. | Volume (in cb. ft.) of Pulp containing 1 ton of Dry Slimes. | Weight (in tons) of Dry Slimes in Charge of Pulp. |
|---|---|---|---|---|---|---|---|---|
| When sp. gr. of dry slimes = 2·5. | $\frac{1{,}000\,(x-1)}{6\,x}$ | $\frac{200\,(2{\cdot}5-x)}{3\,x}$ | $\frac{1-0{\cdot}4\,x}{x-1}$ | $\frac{32}{x}$ | $\frac{1{,}000\,x}{16}$ | $\frac{10{,}000\,(x-1)}{96}$ | $\frac{19{\cdot}2}{x-1}$ | Cb. ft. pulp × $\frac{x-1}{19{\cdot}2}$ |
| When sp. gr. of dry slimes = S. | $\frac{100\,S\,(x-1)}{x\,(S-1)}$ | $\frac{100\,(S-x)}{x\,(S-1)}$ | $\frac{S-x}{S\,(x-1)}$ | $\frac{32}{x}$ | $\frac{1{,}000\,x}{16}$ | $\frac{62{\cdot}5\,S\,(x-1)}{S\,1}$ | $\frac{32\,(S-1)}{S\,(x-1)}$ | Cb. ft. pulp × $\frac{S\,(x-1)}{32\,(S-1)}$ |

(1.) Let sp. gr. of slime-pump = $x$.
(2.) Let per cent. (by weight) of dry slimes in pulp = $y$.
(3.) One cubic foot of water or solution = 62·5 lb.
(4.) One ton of water or solution = 32 cb. ft.
(5.) One gramme per litre = 1 oz. (av.) per cubic foot.
(6.) Capacity (in cb. ft.) of circular vat = (diam. in ft.)$^2$ × (depth in ft.) × $\frac{11}{14}$.
(7.) Sp. gr. of slime-pulp = $\left\{\frac{\text{Wt. of (flask + pulp)} - \text{Wt. of flask.}}{\text{Wt. of (flask + water)} - \text{Wt. of flask.}}\right.$
(8.) Sp. gr. of dry slimes = $\left\{\frac{\text{Weight of dry slimes in air.}}{\text{Wt. of dry slimes in air} + \text{Wt. of (flask + water)} - \text{Wt. of (flask + slimes + water.)}}\right.$
(9.) $= \frac{xy}{xy-100\,(x-1)}$

Notes.—In sampling a charge of slime-pulp undergoing agitation, a narrow-necked vessel fastened to a rod should be employed, so that the sample may be taken not only from the surface of the charge, but also from as far below it as possible.

A 500 c.c. flask is a convenient size to employ in determining the sp. gr. of the pulp; the vessel containing the sample should be well shaken in the intervals of pouring into the flask, so as to prevent settling of slimes during the operation.

(3.) This statement and the formulæ derived from it are not absolutely accurate, but sufficiently so for practical purposes.

(5.) The use of this for simplifying calculations is obvious. For instance, the solubility of a substance expressed in grammes per litre can be read off at once in oz. (av.) per cb. ft.; similarly, the weight of dry slimes in grammes yielded by a litre of pulp can be readily converted into lb. per cb. ft.

(9.) By use of this formula, preliminary drying of slimes for sp. gr. determination may be avoided. The sp. gr. ($x$) and per cent. by weight of dry slimes ($y$) may be determined from 500 c.c. of slime-pulp of any consistency, and the sp. gr. of the dry slimes calculated from these results.

*Cadmium, The Electrolytic Estimation of.* E. Rimbach. Zeits. Anal. Chem. **37**, [5], 284.

*See under* XXIII., *page* 601.

*Gold: Minerals which accompany it in Ore Deposits, and their Influence on the Richness of such Deposits.* T. A. Rickard. Inst. of Mining and Metallurgy, March 23 1898.

This paper is a discussion on the false idea that certain minerals always indicate the presence of gold, especially when applied to localities in different parts of the world. Whilst in some districts a certain mineral might indicate the presence of gold and silver, in others its presence is of no use as a guide; consequently no reliance should be placed on these so-called indicative minerals when opening up a new country. More fallacious still is the idea that the associated mineral can be used as the basis for the prediction that an auriferous lode will become richer in depth. In this direction the finding of tellurides in the gold ores of Western Australia is generally supposed to ensure continuity in depth.

The presence of tellurium has generally been considered baneful to the metallurgist, but its combinations with gold and silver in certain localities in America and Australia have proved so valuable that it is now looked for as a hopeful indication. The author has found it in several ores, and it is possible that many unexplained low extractions in the mill might be traced to tellurium. The presence of this element appears thus to be associated with increased cost or trouble in metallurgical treatment.

In Western Australia it is still asserted that veins become richer below the water level, owing to gold being leached out of the oxidised zone, the ore below retaining its normal richness. Evidence of this is supposed to be derived from the fact that gold is sometimes present and sometimes absent in the cavities in the quartz remaining after the removal of the iron pyrites. In support of this it is believed that gold is soluble in sulphuric acid in the presence of tellurium oxide, and it is a fact that with chlorides also present, it will dissolve, as chlorine is then formed. As the mine water is salt, the theory seems at least tenable; nevertheless it forms an improbable explanation. There are many geological explanations.

In the Kalgoorlie district the gold exists in a chloritic and magnesian matrix, which is softer and more susceptible to erosion than the adjacent poor quartzose outcrops. The erosion of this matrix and the winnowing by the wind has caused a thick deposit or cement to form on its surface, the gold gravitating to the bottom of this cement and giving very little indication at the top of the rich lode lying underneath. The unsuccessful working of the quartz, followed by the discovery of these richer deposits without any surface indication, is the probable origin of the idea that Australian lodes become richer with depth.—A. W.

*Brass, Influence of Antimony on the Cold-shortness of.* E. S. Sperry. Trans. Amer. Inst. Mining Eng., Feb. 1898.

The occurrence of cracks during the rolling of brass is due, in some instances at least, to the impurities of the copper used. The author added to an alloy of 60 per cent. of purest lake copper and 40 per cent. of zinc, quantities of antimony varying from 0·01 to 0·65 per cent., and tested the behaviour in the rolling mill of the alloys so obtained. Brass containing 0·006 per cent. of antimony can be rolled without cracking, but when the percentage reaches 0·02, the fracture indicates the presence of the antimony. The occurrence of antimony in electrolytic copper no doubt accounts for this substance not being reliable as a constituent of brass which has to be rolled.—J. T. D.

*Antimony and Tin, Separation of, in Antifriction Metal and Similar Alloys.* Prost. Bull. Assoc. Belge des Chim. **11**, [11], 337.

*See under* XXIII., *page* 604.

*Uranium, Cadmium, &c., Electrolytic Determination of.* E. F. Smith and D. L. Wallace. J. Amer. Chem. Soc. 1898, **20**, [4], 279.

*See under* XXIII., *page* 601.

*Steel, Carbon in, Determination of, by the Moist Combustion Method.* G. Auchy. J. Amer. Chem. Soc. 1898, **20**, [4], 243.

*See under* XXIII., *page* 601.

PATENT.

*Linings of Metallurgical Vessels* [*Bessemer Converters*], *Impts. in.* J. Colley, Hillingdon, Bilston, Stafford. Eng. Pat. 10,103, April 27, 1897.

Special bricks for lining Bessemer steel converters, &c. are made, having dovetails or projections on their sides or ends, giving them a more secure hold and therefore enabling a safer and better lining to be constructed.—H. H. B. S.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (A.)—ELECTRO-CHEMISTRY.

*Aluminium as an Electrode.* G. J. Hough. J. Amer. Chem. Soc. 1898, **20**, [4], 302—303.

Aluminium may be safely used as a substitute for platinum for cathodes with solutions of nitric acid, oxalates, or cyanides, but not with solutions of sulphates or chlorides. It cannot, however, be used for anodes. Although commercial aluminium cathodes very gradually lose in weight, they may with great advantage be employed, especially for copper determinations. Besides being cheaper, the deposited copper is more readily removed from them, and the weight of the electrode is less out of proportion to that of the deposit than is the case with platinum.—W. G. M.

*Cadmium and Iron, The Electrolytic Separation of.* W. Stortenbecker. Zeits. für Elektrochem. **4**, [17], 409.

*See under* XXIII., *page* 604.

*Perchloric Acid and its Salts, The Electrolytic Production of.* F. Foerster. Zeits. für Elektrochem. **4**, [16], 386.

*See under* VII., *page* 578.

*Calcium Carbide.* J. Sieber. Chem. Zeit. 1898, **22**, [31], 308.

*See under* II., *page* 564.

PATENTS.

*Carbides, An Improved Method of Producing.* J. Landin, Stockholm, Sweden. Eng. Pat. 1033, Feb. 17, 1898.

The pulverised raw materials of the carbide which it is desired to produce (oxide or carbonate of lime and carbon in making carbide of calcium, silicic acid and carbon for carbide of silicon, and so on), are mixed together with the addition of a proportion of tar or other raw material containing hydrocarbon, and are formed into briquettes by heating and pressure, with or without the addition of some flux containing the substance the carbide of which is to be produced; for instance, calcium chloride in the case of calcium carbide. The uniform mass thus produced is introduced, with or without preliminary heating, between the carbons of an electric furnace, the electrodes of which may be made movable about their longitudinal axes to obviate their slagging together with the bloom formed.
—G. H. R.

*Batteries, Impts. in Electrical Galvanic.* [*Impermeable Diaphragm.* G. Laura, Turin, Italy. Eng. Pat. 5912, March 10, 1898.

In this cell, which is an improvement on that described in Eng. Pat. 1245, 1895 (this Journal, 1895, 585), instead of the powder of marsh mallow therein employed, powders made of any of the following plants may be substituted:—

(1.) From the tuberous roots of the *Iris Florentina*;

(2.) From the leaves and stems of various lichens, especially *L. Islandicus* and *L. Caragaen*;

(3.) Of the sphagnum of the family of mosses;

(4.) From the tuberous roots of the various dahlia;

(5.) From the flowers of the *crysanthem, leucanthemum, crysanthemum*;

(6.) From the flowers of *Matricaria Chamomilla*;

(7.) From the roots of the *licorice*, *Glycyrrhiza Glabra.*

The cell is modified by the introduction of a second inner vessel having no bottom, and provided with a turned-in flange which supports the diaphragm. Vertical instead of horizontal diaphragms may also be employed, and they may be used in different forms of batteries in which the elements are zinc and carbon. Hydrochloric acid may be added to the depolarising liquid previously described, and dilute hydrochloric acid or other suitable conducting material may be substituted for sodium chloride in the formation of the diaphragm.—G. H. R.

*Batteries, Primary, Impts. in.* W. Rowbotham, London. Eng. Pat. 9276, April 12, 1897.

An improved construction of liquid-feeding arrangements of batteries of the kind described in Eng. Pat. 2378 of 1897 (this Journal, 1898, 465). Claim: "A primary battery of the kind described, having a closed compartment fed with acid from a reservoir having at its upper part a gas trap so arranged that the action of the battery generates nitrous or other depolarising fumes, which produce pressure within the closed part of the cell, and thus cause acid to pass through the porous partition into the active part of the battery."—J. C. R.

*Batteries, Impts. in Primary. [Circulation.]* W. Rowbotham, London. Eng. Pat. 10,719, April 30, 1897.

This battery, which is an improvement on Eng. Pat. 2378, 1897 (this Journal, 1898, 465), consists of two compartments placed side by side, one of which is gas-tight, and which are supplied continuously with electrolyte through the intermediary of small reservoirs arranged in steps from two large vessels containing respectively water and an aqueous solution of sulphuric and nitric acids. The anodes consist of turnings or scrap metal, preferably iron, contained in a casing of some metal, such as aluminium, which is not appreciably acted on by the electrolyte, while the cathodes, which are formed of carbon rods, are contained in porous pots.—G. H. R.

*Electric Batteries [Carbon Fuel], Impts. in or connected with.* J. L. Dobell, Harlesden, Middlesex. Eng. Pat. 10,484, April 27, 1897.

Relates to batteries in which carbon or carbonaceous matter is oxidised by fused lead oxides. A porous pot containing an electrolyte is suspended within a cell containing another electrolyte, and heated by a suitable furnace, one of such electrolytes being fed with carbonaceous matter and the other with oxygen; a compound, consisting of bichromate of potash, chromium trioxide, and caustic soda or potash, being used for the oxygen-fed electrolyte. The carbon is oxidised by molten lead carrying oxygen from this electrolyte.—J. C. R.

*Batteries, Electric Accumulators or Storage; Impts. in or relating to.* O. Lindner, Brussels. Eng. Pat. 11,861, May 13, 1897.

"Lead electrodes, provided with a spongy surface, obtained mechanically, formed of small irregular torn cavities with no even or regular surface space between same," &c. . . . "retaining in its irregular torn cavities, lead oxides artificially applied," &c.—J. C. R.

*Batteries, Impts. in Galvanic. [Double Cell.]* R. Krayn and Co., Koenig, Berlin, Germany. Eng. Pat. 12,675, May 22, 1897.

A zinc rod stands in a solution of common salt contained in a carbon porous pot, surrounding which is a paste of ammonium chloride enclosed within a zinc outer cell, both the zinc and the carbon being provided with terminals, so that the arrangement forms a complete double cell possessing a common electrode.—G. H. R.

*Batteries, Impts. in and relating to Electric Accumulators or Storage. [Dry Cells.]* A. Heinemann and W. Schafer, Berlin, Germany. Eng. Pat. 15,047, June 23, 1897.

The electrolyte is absorbed by a filling of chemically pure wood charcoal which surrounds the electrodes, and which is either in the form of powder, or is compressed into casings or plates which protect the electrodes and prevent the gradual falling out of the active material.—G. H. R.

*Electric Batteries, Impts. in and connected with Substances to Prevent Polarisation in.* G. Platner, Witzenhausen-on-the-Werra, Germany. Eng. Pat. 23,329, Oct. 11, 1897.

I. "Composition for filling electric battery cells for depolarisation purposes, composed of chlorates of potassium or sodium combined with the peroxides of the heavy metals, their acids and salts, the peroxides of the heavy metals freeing the oxygen of the chlorates by catalytic action." . . . II. . . . . . "composed of chlorates, metallic salts," iron, chromium, &c., "which are capable of readily forming basic salts, and the peroxides of the heavy metals," lead, manganese, &c.—J. C. R.

*Compositions, Acid-Proof and Electric Insulating; Impts. in.* W. Morison, Montclair, New Jersey, U.S.A. Eng. Pat. 9290, April 12, 1897.

The composition, which is used for battery cases and the like, is prepared by melting asphalt and intimately mixing asbestos fibre therewith whilst hot; the mass is then spread out on a hot surface, and the moisture and foreign substances are removed by thoroughly working, beating, and pounding the material. To make the composition more elastic and tenacious, a small quantity of gutta-percha may be worked in. The addition of sulphur makes the material spongy, and gives tenacity to the mass. This is worked in after the composition has been spread out. It is then pounded whilst hot to expel all foreign matter.

The above operations are performed at a moderate temperature (not less than 150° F.). When ready, the compound is put into a mould hot, and it can be taken out after 20 to 40 seconds, forming a hard and smooth material, having a polished surface and requiring no seasoning or further treatment.—D. B.

*Electrically Decomposing Solid Substances, A Process of and Apparatus for.* W. S. Romme, New Rochelle, State of New York, U.S.A. Eng. Pat. 11,402, May 7, 1897.

Particularly intended for the decomposition of salts of the alkalis, such as the chlorides of sodium and potassium, and the production of hydroxides and chlorine therefrom. With the object among others, of minimising the volume of electrolyte acted upon, the solid, as common salt, is dissolved by water percolating through layers of it arranged above and between horizontal electrodes fitted in a tank, the resulting solution being run off from below the electrodes. Five claims. I. ". . decomposing solid substances electrically, which consists in placing the substance to be acted upon between electrodes, and causing a liquid solvent of the substance treated, to percolate through the mass in such quantity as suffices merely to keep the mass moist without submerging the mass in said fluid, and passing an electric current through the mass."—J. C. R.

*Insulating Material, Impts. in the Manufacture or Production of an; and the Application thereof to the Insulation of Electrical Conductors.* H. Edmunds, London. Eng. Pat. 14,439, June 15, 1897.

Paper or other fibrous material is coated on both sides with a solution of vulcanised or unvulcanised rubber of such "density" that it shall not penetrate into the substance of the paper, but shall leave the pores full of air. The solvent is evaporated as usual, and the product is wound, spirally or otherwise, round the conductors whilst the rubber coating is still tacky. The whole may then be painted with another solution of rubber, if desired, and finally vulcanised or united by heat.—F. H. L.

*Electric Accumulators, Impts. in and relating to.* C. Junge, London. From R. Knoschke, Leipsic, Germany. Eng. Pat. 4825, Feb. 26, 1898.

Spongy lead of fine texture is produced by boiling lead in dilute nitric acid, 10 to 20 per cent., the boiling being stopped before the formation of crystals begins, cooling the solution, immersing strips, rods, or bars of zinc, coke, or coal therein, and adding pulverised manganese peroxide to the spongy lead precipitate, mixed with minium or litharge. This mixture is used for active material.—J. C. R.

## (*B.*)—ELECTRO-METALLURGY.

*High Temperatures, New Process for the Production of, and for the Preparation of Metals Fusible with difficulty, and Free from Carbon.* Goldschmidt. Zeits. f. Elektrochem. 1898, **4**, [21], 494—498. (See also pp. 543 and 612.)

The author shortly reviews previous work on the use of aluminium in producing high temperatures, and then describes the method he adopts. As is well known, an extremely violent reaction takes place when aluminium unites with oxygen, the estimated temperature of the reaction being about 3,000 C. In order practically to make use of the high temperatures thus obtained, the chief difficulty is so to moderate and control the reaction that it will perform the required work.

The first point to be noted is that it is not necessary to heat up the whole of the reacting mass to the temperature of ignition. It is sufficient to start the combustion at a single point only, but this in itself presents some difficulties. In the preparation of metallic chromium from a mixture of its oxide with powdered aluminium, these difficulties may easily be overcome by applying at any convenient point a small quantity of a mixture of aluminium with a more easily reducible oxide, or preferably a peroxide. Lead oxide, copper oxide, potassium permanganate, and many other substances may be employed for this purpose.

One great advantage of this process is that it admits of the preparation of pure metals free from aluminium; the only precaution which requires to be observed is that the oxide to be reduced must be present in slight excess.

The process is capable of two main applications. In the first place the heat given off by the combustion of a mixture of aluminium with any convenient oxide may be utilised for heating purposes only (welding, &c.), or, secondly, the reducing power of aluminium at a high temperature may be used for the preparation of pure metals or alloys. In either case only slight alterations in the mode of working are necessary. If a moderate heat be required, the reacting mass is diluted by the addition of some inert substance, which, at the same time, prevents the whole mass from melting. A convenient mixture of this sort is composed of aluminium and the cheapest available oxide (*e.g.*, iron ore, sand, &c.), which is then diluted either with a large excess of the same oxide or with magnesia, lime &c. If, on the other hand, the metal itself be required, a large excess of the oxide must be avoided, so that the heat which is generated will not only melt the metal, but also the slag (corundum), which then protects the regulus from the action of the air.

The following experiments illustrate the applications of the process:—A rivet weighing 3 kilos., such as is used in the construction of bridges, is surrounded by a mixture of oxide of iron, sand, &c., and powdered aluminium, and almost wholly embedded in sand contained in a wooden box. On the top of the small exposed portion of the aluminium mixture is laid a small ball prepared by mixing aluminium powder with a more easily reducible oxide, and into which is inserted a short length of magnesium ribbon. The reaction is started by setting fire to the magnesium ribbon, and as soon as this is done more sand is placed on the top in order to keep in the heat. If the contents of the wooden box are emptied out after a short time it will be found that the rivet is white hot and ready for forging.

In a somewhat similar way 1-in. iron pipes may be hard-soldered at an estimated cost of about 2*d.*; or, two pieces of wrought iron may be fused (welded) together. The joint so obtained is satisfactory, as can be shown by sawing the pieces through at the joint. It is claimed that such a joint is better than that produced by electric welding, owing to the greater uniformity of heat. By diminishing the quantity of inert material, the iron in the above experiment may easily be melted. Holes may be made through $\frac{1}{2}$-in. wrought iron plates by igniting some of the reacting mixture on them, and adding more, if necessary, as soon as the reaction has started.

In order to reduce chromium from its oxide, a mixture of this and powdered aluminium is made and introduced into a crucible lined with magnesia. The reaction is started as before, and the mixture then added continuously until the crucible is full. After cooling, the crucible is broken open and the regulus of metallic chromium removed. At the meeting of the German Electrochemical Society, at which the author communicated his paper, he showed a mass of chromium weighing 25 kilos. prepared in this way. By making two apertures in the crucible, one for the introduction of the mixture and the other for the outflow of the chromium, the process may be worked continuously, as in the case of the electric furnace.

It is claimed that the temperature attained is higher than in the ordinary electric furnace, and that a given quantity of material can be put through in a shorter time. Moreover, the chromium is also free from carbon or carbides. The slag (alumina) produced in the furnace may be reconverted into aluminium and used over and over again, or it may be used for polishing, since it possesses certain advantages over emery.

Almost all metals may be reduced from their oxides in this way, and the yield is nearly theoretical. The different alloys, *e.g.*, "chrom-manganin," copper-chromium, &c., can also be prepared. The slag from the furnace contains minute rubies, which owe their colour to chromium.

For the preparation of pure metals it is necessary to use pure aluminium, but for heating purposes only, crude (50 per cent.) aluminium may be employed. Besides oxides the oxy-salts of certain metals may be reduced in the same way.

The author looks upon aluminium as a "*heat accumulator*," since it is possible to transport it, and by its union with oxygen develop, wherever necessary, an amount of energy corresponding to that which was originally required for its preparation.—J. S.

*Copper, Technical Separation of, from the Group of Positive Metals (Nickel, Cobalt, Iron, Zinc).* B. Neumann. Zeits. f. Elektrochem. (1898), **4**, 316—322 and 333—338.

During the electrolytic refining of copper, the following foreign metals contained in the anode (crude copper) pass directly into the anode mud: *viz.*, silver, gold, platinum, bismuth, tin, arsenic, antimony, and lead; whilst others are only precipitated from the solution on long standing or after saturation. The more positive metals, nickel, cobalt, iron, and zinc, on the other hand, are dissolved out of the anode along with the copper, but are not deposited on the cathode, owing to the low E.M.F. employed. The bath, which was originally an acidified copper sulphate solution, thus becomes richer in these positive metals and poorer in copper. If the anode contains much larger quantities of the positive metals than are usually found in crude copper—*e.g.*, German silver, brass (old cartridge cases), &c.—then pure copper will be deposited on the cathode as before, provided the E.M.F. does not exceed that generally employed in the electrolytic refining of copper. The problem which the author set himself was to determine the effect of the large quantities of positive metals dissolved in the bath on the nature of the copper deposited on the cathode. The conclusions at which he has arrived may be summarised as follows:—

Alloys of copper with the more positive metals, nickel, cobalt, iron, zinc, and aluminium, may be refined in the ordinary way. The copper only is deposited on the cathode, whilst the other metals remain in solution. The E.M.F. per bath should not much exceed 0·5 volt. As the electrolysis proceeds, the resistance of the electrolyte increases, owing to its becoming poorer in copper and richer in the other metals, and for this reason the efficiency of the current falls. Not only is this so, but the quality of the copper deposited gets worse and worse, so that practically the electrolysis must be stopped at a certain point. The experiments of Forster and Seidel have shown that the precipitation of brownish-red powdery copper is connected with a certain low concentration of the copper ions in solution, and hence, during the refining of the above alloys, a point must ultimately be reached when spongy copper is deposited. Addition of more free sulphuric acid can in no way prevent this. According to the law of mass action it would rather tend to facilitate the formation of spongy copper. This being so, the suggestion of certain authors to employ nothing but sulphuric acid in the bath is misleading. Starting with a bath containing sulphuric acid only, the copper obtained by the author still remained spongy in texture, even after the electrolysis had been allowed to proceed for 20 hours. It has also been stated that during the refining of the above alloys the bath ultimately becomes entirely free from copper. This is not borne out by actual experiment, and the author considers it cheaper to remove the remainder of the dissolved copper by means of sulphuretted hydrogen than by the introduction of insoluble anodes, or anodes composed of the more positive metals.

Since an Austrian firm has succeeded in electrolytically refining crude copper containing 20 per cent. of impurities, the author considers that Canadian nickel ore will probably, in the near future, become of greater importance, if it can be treated in a similar manner.—J. S.

### *Chromium, Electromotive Behaviour of.* [*Active and Passive States of.*] W. Hittorf. Zeits. für Elektrochem. 1898, **4**, [21], 481—492.

In an address to the German Electrochemical Society, Hittorf described the results of experiments made with two specimens of metallic chromium supplied by Moissan and Goldschmidt (see accompanying [Goldschmidt] abstract) respectively.

Zinc, according to Wöhler, precipitates metallic chromium from its fused salts, and it was therefore to be expected that chromium would occupy a position immediately following zinc at the positive end in the electromotive series of metals. It was found, however, that when chromium was dipped into solutions (say dilute HCl, &c., which do not cause the liberation of hydrogen at the ordinary temperature), it was not only electro-negative towards zinc, but also with respect to cadmium, iron, nickel, copper, mercury, and silver. It had no action on the neutral salts of these metals, even at the boiling point; the chlorides of gold and platinum and palladious chloride were also unattacked.

Mercuric and cupric chlorides and cupric bromide only were reduced to the lower salts at the boiling point. In this respect chromium behaves like silver, but at low temperatures generally it acts like a noble metal.

Attention is called to the fact that chromium forms three classes of compounds, corresponding to the three stages of oxidation, CrO, $Cr_2O_3$, and $CrO_3$, and that when it is employed as the anode during the electrolysis of one and the same electrolyte ($ZnCl_2$), it dissolves with the formation of different compounds, according to the temperature and the solvent used.

Chromium exists in three different states, *viz.*, *passive*, *active*, and *indifferent*, and in each of these states it occupies a different position in the electromotive series and possesses a different chemical behaviour.

In the *passive* state it is a noble metal, it possesses no reducing action on the salts of other metals, and follows platinum at the electro-negative end of the electromotive series of metals. This is its normal condition at the ordinary temperature; and when it serves as anode during the electrolysis of $ZnCl_2$, NaCl, HCl, &c., it dissolves with formation of chromic acid, which colours the solution in the neighbourhood of the anode yellow.

In the *active* state (produced generally at a higher temperature) it immediately follows zinc in the Volta-series, and replaces all the more electro-negative metals from their salts.

Employed as anode during electrolysis of (boiling) chlorides, it dissolves as $CrCl_2$, imparting a bluish-green colour to the solution. In certain cases the solution requires to be heated above 100°, as, for example, when the chlorides of magnesium and zinc are employed. A chromium anode still remains passive in a boiling dilute solution of zinc chloride (to which an excess of zinc oxide has been added to neutralise all the free hydrochloric acid), and the yellow colour of chromic acid is observed. If, however, the solution is gradually evaporated, the temperature rises as it becomes more concentrated, and finally, at about 130° C., the green coloration due to the formation of $CrCl_2$ appears.

The passage of chromium from the inactive or passive condition to the active is also exemplified by the following experiment. Passive chromium may be boiled for any length of time in solutions of the chlorides of copper, gold, palladium, or platinum, without reducing them. If, however, it be introduced into a boiling solution of potassium or sodium chloride, it immediately acquires its new active character. Consequently, a fragment of chromium which, on being employed as an anode, has yielded chromic acid during electrolysis may be boiled in a test tube, for any length of time, with the chlorides of the heavy metals without causing any reduction; but if any of the chlorides of the alkali metals be now added, reduction immediately takes place. This action becomes slower and slower the less the quantity of KCl, &c. added, and finally becomes inappreciable. Moreover, the reducing action only lasts as long as the high temperature is maintained, and ceases as soon as the solution cools down again.

In the *intermediate* state it takes up an intermediate position in the electromotive series, and forms an intermediate compound. An alcoholic solution of zinc chloride, on electrolysis, yields a beautiful green solution of $CrCl_3$ at the chromium anode.

Passive chromium does not combine with free iodine, even in the nascent state, whilst the active modification removes iodine from hydriodic acid.

The behaviour of chromium towards melted salts is also described.

Hittorf considers that the passive condition of iron may well be ascribed to the formation of an invisible film of oxide, which protects it; but a similar explanation of the passive condition of chromium does not hold good, as is shown by numerous experiments. Up to the present no satisfactory explanation has been given of the remarkable behaviour of chromium. In its three different states it possesses such widely divergent properties as have hitherto only been found in different metals.—J. S.

### *Gold, The Electrolytic Deposition of.* E. Wohlwill. Zeits. für Electrochem. **4**, [16], [17], and [18], 379—385; 402—409; 421—423.

A very lengthy article, giving the results of experiments made at the Nord-deutsche Affinerie, Hamburg. Pettenkofer showed 50 years ago that gold purified by the usual sulphuric acid parting process always contained small amounts of platinum and silver. The utility of nitre as a purifying agent he proved to be due to its oxidising action upon the platinum, and he was also able to show the presence of platinum in the nitre slag from such fusions. The practical results of these discoveries was the Aqua-regia parting process for gold and platinum.

The Hamburg Nord-deutsche Affinerie chemists commenced to investigate the subject of the electrolytic separation of gold from platinum and silver 20 years ago, and at the Paris Exhibition of 1881 a bar of electrolytic gold, testing $\frac{1000}{1000}$ fine, was shown.

The method of electrolytic parting has been improved since that date, and is now patented in the author's name in Germany, England, the Transvaal, and the Australian colonies. Gold chloride solution was the only possible form of electrolyte; the cyanide was not practicable, since both silver and copper—impurities of raw gold—form soluble cyanide salts; and the electrolytic gold produced by the Siemens and Halske process always contains the silver and copper present in the electrolyte. The author found, however, that neither a neutral solution of auric chloride, nor a solution of the hydrogen auric chloride salt, would serve his purpose, since the chlorine liberated at the anode did not attack the raw gold, but either escaped as gas or reacted with the water and liberated oxygen. This is all the more remarkable, since if hydrochloric acid be electrolysed with a gold anode, solution occurs. The author explains the different behaviour of the gold by the theory that solution only occurs when the salts—HCl, NaCl, KCl, &c.—are present in the electrolyte in sufficient quantity to permit the formation of complex salts of the hydrogen auric chloride type; that is, when the conditions are favourable to the production of the ion—$AuCl_4$. Hittorf showed long ago that when solutions of complex salts of this type are electrolysed a complex anion separates at the anode. In the case of the anion—$AuCl_4$, the deposit of gold at the cathode is due to a secondary reaction between this —$AuCl_4$ and the hydrogen or potassium liberated at the cathode. The presence of the ion —$AuCl_4$—in such an electrolyte can be proved by adding silver nitrate. A complex precipitate containing gold is first formed, and the silver chloride which eventually separates on standing or exposure to light, is due to a secondary reaction. In the conduct of the electrolytic process at Hamburg, the conditions favourable to the solution of the gold anodes are obtained by continual additions of hydrochloric acid to the anode chambers of the cells. The higher the current density employed, the more frequent must these additions be. Increase of temperature of the electrolyte has the same effect, and if chlorine has commenced to escape as a gas at the anodes, it can be stopped by heating the electrolyte. If sufficient hydrochloric acid were present, the author found that a current density of 300 ampères per sq. metre (273 ampères per sq. ft.) might be safely used; and under these conditions a plate of gold 4 mm. thick and 5 kilos. in weight could be wholly dissolved by a current of 308 ampères in 5 hours.

In actual work a current density of one-third the above and an E.M.F. of only 1 volt is used; the time required for the solution of such a plate of gold is thus increased to 15 hours.

The gold is obtained at the cathode in coarse crystalline particles, which do not fall away from the surface, but which can be easily detached by mechanical means. The electrolyte should contain 30 grms. of gold per litre. The electrodes can be placed very close together, as there is no "treeing" of the deposit, and the volume of electrolyte required per cell is therefore not great. The depositing vats of the "Frankfurter Gold u. Silber Scheide Anstalt" in Hamburg suffice to yield 75 kilos. fine gold per 24 hours, yet they only cover a ground space of 6 sq. m.

All the constituents of the anode, with the exception of the silver, pass into solution, but, owing to the lower E.M.F. required to separate the gold, only this metal is deposited. The electrolyte is used for some months before the separation of the platinum and palladium is effected by means of ammonium chloride. The silver is found as anode slime, on the bottom of the cell. The average fineness of the electrolytic gold is over 999·8.

The author next considers the advantages of the electrolytic method of separation, as compared with that by means of ferrous chloride from the aqua-regia solution.

Under certain conditions named in the patent specification, this electrolytic method of parting the noble metals can be used for many of the alloys used in exchange, or in the arts.

The weight of gold deposited at the cathode per ampère hour, varied greatly in all the author's experiments, but it was always in excess of the theoretical amount calculated from the electro-chemical equivalent for trivalent gold. The author was led to suspect the presence of monovalent gold ions in the electrolyte, by noticing that the gold found with the silver chloride slime beneath the anodes had not the appearance of mechanically separated particles, but appeared to be the result of secondary chemical reactions. He assumed therefore, that both aurous and auric chloride are formed at the anode, and that the former then decomposes to form auric chloride, according to the following equation, $3AuCl = AuCl_3 + Au_2$. A similar reaction is held to occur in copper refining. The presence of these monovalent gold ions in the electrolyte leads to the transfer of a certain proportion of them to the cathode by the action of the current, and to the variation in the amounts of gold deposited per ampère hour. This theory also explains the facts relating to the loss of weight by the anode, which is greater for a given amount of current than would be the case if only the trivalent gold salt, $AuCl_4H$, were formed. The author gives some tables of results showing the connection between the loss of weight at the anodes, and the current density and temperature of the depositing cell. A high-current density was found to promote the formation of trivalent gold ions, but the experiments require to be conducted under exactly similar conditions as regards temperature and acidity of the electrolyte, in order to observe correctly the effects of varying current densities. The practical advantage of high-current density is the diminution of the gold found in the silver chloride slime, since the supply of the monovalent gold ions is reduced to a minimum, and the secondary reaction noted above cannot occur.

The experiments described above were undertaken under conditions which permitted free circulation of the electrolyte from anode to cathode. Under other conditions the results obtained would be different.

The author finally carried out some experiments in order to see whether continued diminution of the current density would lead to the production of a greater number of monovalent ions of gold at the anode.

The solvent action of the electrolyte upon the gold anode at the temperature used (65°—70° C.), even when no current was passing, had to be allowed for in these experiments. A fall of temperature in such a solution can also cause increase of weight of the anode, due to the gold set free by the reaction $3AuCl = AuCl_3 + Au_2$.

Allowing for all these disturbing influences, it was found possible to obtain direct proof that a diminishing current density caused increase in the ratio of monovalent to trivalent ions of gold. In other words, the electro-chemical equivalent of gold increased, as the current density diminished. With a current density of 136 ampères per sq. ft. or over, it is at a minimum, and nearly equal to that calculated for $Au^{III}$; with a current density of 0·09 ampère per sq. ft. it is at a maximum, and but little below that calculated for $Au^{I}$.

This peculiarity of a metal forming simultaneously at the anode, ions of differing valencies, may possibly find examples in the case of other metals.—J. B. C. K.

*Electrolytic Iron, The Properties of.* F. Haber. Zeits. f. Elektrochem. 4, 110—113.

In the spring of 1897 the author had opportunities for inspecting the electrotype methods used by the Government printers of bank notes in Germany and Austria. Both in Russia and in the latter country the notes are printed from steel-faced electrotypes, made by electrolytic deposition of iron from a bath prepared according to Klein's formula (ferrous and magnesium sulphates), under conditions of temperature and current density which have not been divulged. The latter is certainly very small, as a plate only 2 mm. in thickness requires 1½ months for deposition. The plates prepared in this way have the appearance of steel-faced copper plates; the under side is slightly rough, but the author did not observe any of the oval pittings spoken of by Lenz in his earlier paper on this subject. The thin iron electrotypes prepared as above are backed by copper in the usual manner. Their advantage lies in the fineness and hardness of the metal which is first deposited, and in the delicacy of the copy of the original which is thus obtained. The other plan of preparing these plates is, first to obtain a copper electrotype of the original, and then to face this with iron by suspension as cathode in the above bath of ferrous and magnesium sulphates. The printing results are, however, not nearly so good as when the former method is employed. These plates coated with iron can be used for a much larger number of impressions than those of copper alone. The author notes that the first deposit of electrolytic iron is harder than the succeeding ones, and that the variations in hardness are measured by the proportion of hydrogen present in the deposit. The disadvantage of these copper-backed iron plates is that they can only be renewed by the repetition of the whole process, whereas the iron-faced copper electrotypes can have their faces renewed in a few minutes.

The author had an opportunity of examining the chemical constitution of one of the plates deposited according to the methods used by the Austrian bank-note printers. The plate was 3 mm. thick, and could be bent double without fracture. Left for three-quarters of a year in a damp laboratory atmosphere, it showed no apparent signs of rusting.

The original paper by the author contains details of the gravimetric method employed for determining the hydrogen contained in this iron. He obtained a mean percentage of 0·015 per cent., which worked out to a volume ratio of hydrogen to iron of 17·3, or to a volume ratio of 12·0 when corrected for the errors of analysis. Lenz obtained volume ratios of hydrogen to iron varying from 13·4 to 16·3 in his experiments upon electrolytic iron.

The author next undertook the investigation of the conditions necessary in order to obtain a maximum amount of hydrogen in the deposited iron. Using a current density of 2,140 ampères per square foot, he obtained a deposit containing from 0·085 per cent. to 0·110 per cent. of hydrogen. This equals a mean volume ratio (hydrogen to iron) of 112. Lenz obtained ratios between 105 and 129 for this type of iron deposit. The latter found, in addition to hydrogen, carbonic acid, carbonic oxide and nitrogen gas present in electrolytic iron, but the author has not had an opportunity for verifying these results.

The deposit obtained by continued use of the high-current density just named was perfect in every respect, with the exception of its tendency to cause curvature of the cathode. The author overcame this by using a revolving cylinder as cathode, within a larger iron tube which acted as anode.

If the electrolytic iron be heated in a Bunsen flame, the hydrogen and other gases escape with small explosions, and the remaining metal is then found to have lost its hardness, but is still less soft and pliant than wrought iron of a similar thickness.

In conclusion, the author states his opinion that iron containing hydrogen undergoes change in physical structure with time. He commenced experiments bearing upon this point, with intermittent currents, but these are not yet completed.—J. B. C. K.

*Copper from Mercury, and Copper from Arsenic, Electrolytic Separation of.* N. Revay. Zeits. f. Elektrochem. 1898, **4**, [14], 329.

*See under* XXIII., *page* 603.

*Cobalt from Nickel, Method for the Separation of.* Coehn. Zeits. f. Elektrochem. 1898, **4**, [22], 501.

*See under* XXIII., *page* 605.

### PATENT.

*Anodes, Impts. in Electro-depositing.* H. L. Haas, New York, U.S.A. Eng. Pat. 2967, Feb. 5, 1898.

The anode, which is an improvement on Eng. Pat. 7358, 1896 (this Journal, 601, 1896), consists of a cast-metal plate or body, and a hooked hanger cast integrally with it, the bearing surface of the hanger having two or more flat faces.—G. H. R.

## XII.—FATS, OILS, AND SOAP.

*The Cholesterols of Plants of Low Development.* E. Gérard. J. Pharm. Chim. 1898, **7**, [8], 373—375.

Continuing his researches on the cholesterols of cryptogamous plants, which have been shown to differ from animal cholesterol and from the phytosterol of phanerogams, and to be related to the "ergosterin" of Tanret, the author has isolated similar bodies from a micro-organism, *Staphylococcus albus*, and from an alga, *Fucus crispus*. In each case the cholesterol was obtained by washing out with ether the saponified fatty matter derived from the plants, and gave identical reactions, dissolving completely in sulphuric acid, with the formation of a red colour; this solution shaken with chloroform, did not colour that solvent. The acid solution diluted with water gave a greenish precipitate; ordinary cholesterols so precipitated are white. A solution of these new cholesterols in carbon tetrachloride colours sulphuric acid blood-red, whilst the layer of the tetrachloride acquires a bright green tint. On exposure to the air, these cholesterols, like ergosterol, are changed and become coloured. —J. O. B.

*Wool-Fat, Examination of.* J. Lifschütz. Pharm. Zeit. **43**, 230. Chem. Zeit. Rep. **22**, [14], 119. (This Journal, 1898, 253.)

From results of the examination of purified wool-fat, the author concludes that this product should conform to the following tests:—(1.) A well-purified wool-fat must be fatty, soft, and pliable, and must not lose these characters on being exposed for some time to the air. It must not possess the smell which accompanies crude wool-fat. (2.) It must not darken on heating. (3.) 0·5 grm. boiled with 5 c.c. of glacial acetic acid, after cooling and filtering, should have at most a brownish-yellow tinge, and no green colour, when four or five drops of concentrated sulphuric acid are added. (4.) 2 grms. dissolved in ether with two drops of phenolphthalein solution must give a red coloration with one or at most two drops of a decinormal caustic potash solution. (5.) On ignition, it must leave no ash. (6.) It must be free from chlorine, and this is ascertained by boiling some of the wool-fat with absolute alcohol to which a drop of nitric acid has been added. The liquid is then filtered, and tested with an alcoholic solution of silver nitrate.—J. McC.

*Cotton Seed Oil in Olive and other Oils, Methods of Detection of.* M. Tortelli and R. Ruggeri. Chem. Zeit. Rep. **22**, 101.

*See under* XXIII., *page* 607.

*Fats, Testing, for Rancidity.* A. Schmid. Zeits. anal. Chem. **37**, [5], 301.

*See under* XXIII., *page* 610.

*Fatty Substances, Estimation of Ash in.* A. Delecoeuillerie. Bull. Assoc. Belge des Chim. **12**, [1], 5.

*See under* XXIII., *page* 610.

*Fatty Oils, Uniform Methods in Testing.* D. Holde. Chem. Rev. Fett- u. Harz-Ind. **5**, [3], 41.

*See under* XXIII., *page* 610.

*Cod Liver Oil, Testing.* A. Gawalowski. Chem. Rev. Fett- u. Harz-Ind. **5**, [47], 67.

*See under* XXIII., *page* 607.

### PATENTS.

*Oils, Oxidising of, Impts. in the, and in Apparatus for the Purpose of.* S. Rosenblum, S. Rideal, both of Westminster, and the Commercial Ozone Syndicate, Ltd., London. Eng. Pat. 9529, April 14, 1897.

The improvement claimed is for the oxidation of oils, by themselves or mixed with other oils, by means of ozone, and particularly when used in conjunction with soluble siccatives, such as the resinates or linoleates of lead and manganese. Claim is also made for a special apparatus for the purpose, in which the oils, heated when required, are brought into contact with ozone under pressure and then discharged as a fine spray into a covered vessel, thus becoming intimately mixed with the gas. Here they meet a current of ozone, preferably passing upwards, and are further ozonised. The oil is collected at the bottom of this vessel, and, if desired, can be conducted back to the first vessel, and the process of oxidation repeated. It is stated that a mixture in equal proportions of linseed oil with cotton-seed oil, or with rape oil, is much more quickly solidified by this process than linseed oil alone, and that the reaction is thus suitable for the cheap production of semi-solid products for linoleums and india-rubber substitutes. —C. A. M.

*Soaps and all Toilet Preparations, Impts. in.* R. T. Cardell, Bodmin. Eng. Pat. 12,456, May 20, 1897.

An addition of extract of laurel, with the object of whitening the skin and destroying pimples, is claimed.—C. A. M.

*Fats and Oils, Impts. in Apparatus for Treating.* C. G. Hepburn, Sydney, New South Wales. Eng. Pat. 30,684, Dec. 28, 1897.

This invention, primarily intended for the removal of residual water from hot liquid fats, such as tallow, consists of a chamber through which a current of air, previously warmed, or warmed and dried in annular casings of the chamber, is made to ascend by means of a propeller. The oil or fat, heated until liquid at the top of the chamber, is distributed in a fine spray by means of centrifugal action, and falls on to baffles encircling the sprayer, whence, after coming into contact in a fine state of division with the current of hot air, it falls into a collecting tray at the bottom of the chamber.—C. A. M.

*Fats suitable for Lubricating Purposes, Impts. in the Production of.* G. Pommerhanz and C. Ristic, both of Vienna. Eng. Pat. 3585, Feb. 12, 1898.

A mixture of neutral wool-fat, ordinary wool-fat, and of animal, vegetable, or mineral oil, mixed with a decoction of Iceland moss, and the whole stirred up with alkaline lye (preferably of 38° B.) or with a solution of common salt (1 : 3).—C. A. M.

*Fats or Oils, Separation of, into Glycerin and Fatty Acids; Impts. relating to the, and to Compounds for Use therein.* H. H. Lake, London. From E. Twitchell, Cincinnati, Ohio, U.S.A. Eng. Pat. 4741, Feb. 25, 1898.

In this process the neutral fats, with the addition of a small quantity of free fatty acid, if not already present, are decomposed into fatty acids and glycerin by mixing them with a small quantity of a sulph derivative of a fatty acid or acids (sulpho-stearic, sulpholeic acid, &c.), preferably in the proportion of about 1 per cent., adding 50 per cent. of water, and boiling for several hours. At the end of the operation the glycerin and water are drawn off from the bottom of the tank, and the reagent recovered by precipitation with sulphuric acid or a metallic salt, such as lime. When separated, it is capable of being used again. It is stated that the fatty acids and glycerin are thus obtained without material discoloration, that a larger yield is obtained, and that the cost is less than by other methods, since no further refining is required. Various modifications of this process are described in the patent. The sulpho reagent may be prepared by treating commercial oleic acid with sulphuric acid in excess, preferably at a temperature of about 100° C., skimming off the compound formed, and washing it free from the excess of acid. Special claim is also made for a class of compounds which the inventor terms "sulpho-fatty aromatic compounds," which are prepared with much less waste, and which are not so liable to discoloration as the ordinary sulpho-derivatives of fatty acids. A fatty acid, such as oleic acid, is mixed, preferably in molecular proportions, with an aromatic compound, such as benzene, phenol, naphthalene, &c., the mixture treated with sulphuric acid, and after the reaction has taken place, the excess of sulphuric acid is removed by washing with water. The resulting double compound is a clear oil containing an aromatic radical and a fatty acid radical in combination with the sulphonic radical. It is stated that, besides replacing the ordinary sulpho-derivatives in the process described above, these compounds are capable of extended use in the arts, on account of their great stability.
—C. A. M.

# XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

## (*A.*)—*PIGMENTS, PAINTS.*

*Oil Paints, Testing.* H. Loester. Chem. Rev. Fett- u. Harz-Ind. 5, [4], 61.

*See under* XXIII., *page* 608.

### PATENT.

*Paint or Composition to be used as a Coating, more especially in the Manufacture of Artificial Slates, and for Coating Tiles, Plates, and other Articles.* C. A. Hakanson and J. H. Hakanson, both of Kalmar, Sweden, and A. R. Hakanson, Karlshamn, Sweden. Eng. Pat. 8510, April 2, 1897.

*See under* IX., *page* 508.

## (*B.*)—RESINS, VARNISHES.

*Oxygen, Absorption of, by Linseed Varnish.* R. Kissling. Zeits. angew. Chem. 1898, 361.

Some years ago (this Journal 1891, 778) the author outlined a method for the examination of drying oils, which depended on a determination of the increase in weight they exhibit during exposure to air under certain conditions for a definite period of time. This method has lately (this Journal 1897, 924) been approved by Amsel; but Weger has criticised it, objecting that the phenomena which accompany the desiccation of a thick layer of oil (such as the author would adopt), are not fairly comparable with those which occur during the drying of an ordinary film of varnish or paint, so that Kissling's results are of no practical significance.

Kissling does not claim that his process gives the total amount of oxygen each variety of drying oil will ultimately take up per unit of area or weight, but simply that it enables several samples to be compared one with another for drying power, provided they are exposed to the air under strictly identical conditions.—F. H. L.

*Oxygen, Absorption of, by Drying Oils and Boiled Oils.* W. Lippert. Zeits. angew. Chem. 1898, 412.

This article consists of a contribution to the discussion now taking place between Kissling, Weger, and Amsel as to whether, in attempting to value drying oils by a determination of the oxygen they absorb on exposure to air, it is necessary to pay particular attention to the thickness of the film under treatment. After remarking the somewhat contradictory statements in Mulder's book, "Die Chemie der austrocknenden Oele," Lippert proceeds to refer to Kissling's first experiments (this Journal, 1891, 778), where 10 grms. of oil were exposed in vessels having a superficies of 35 sq. cm.; and he agrees with Weger that the tests were vitiated by the excessive depth of the liquids, so that the oxygen absorptions were far too small.

Lippert prefers to use plates of sheet iron (perhaps aluminium or celluloid would be better) about 18 × 10 cm. in size, weighing 56 grms. A strip 1 cm. wide is left clean at one end, while the rest is covered with the sample to be examined in such a way that not more than 0·1 grm. is applied per 100 sq. cm. In order to obtain as uniform a coating as possible, the plate is inclined at an angle of 30°, and allowed to drain for 10 minutes; the excess of oil which collects at the bottom edge is then wiped off with blotting paper, and the plate is put on the pan of the balance tilted in the opposite direction and weighed rapidly, whilst the liquid is running back towards the thinner portions of the film. Exposure to air is carried out in a ventilated glass box placed in a north light where the temperature is fairly constant throughout the night and day. Four tests with a good specimen of boiled oil, the thickness of the film differing in each case, yielded (considering the probabilities of error owing to the excessive weight of the metal supports) almost identical results; and showed that within certain limits, as Mulder has stated, variations in the said thickness do not affect the test.

The author finds that under the conditions obtaining in his experiments, raw linseed oil increases in weight, slowly for three days, on the fourth day more quickly, and becomes dry on the seventh; the total gain being much the same as that given by Mulder, *viz.*, 12·4 per cent. It is not advisable to heat the plate to 80° C. for the removal of condensed moisture, as suggested by Mulder, seeing that, as Bauer and Hazura have pointed out, this may give rise to the formation of anhydrides.

Linseed oil strongly boiled without driers (stout lithographic varnish) dries more slowly than raw oil, and is more difficult to manipulate, owing to its great viscosity, but eventually it absorbs within 2 per cent. of the oxygen taken up by the untreated product in spite of the partial oxidation and the assumed polymerisation which occur during the boiling process. Tung oil behaves in a very similar manner, but gains in weight more slowly, although the amount of oxygen ultimately absorbed is very nearly the same. Ordinary boiled oils cannot properly be compared with raw linseed without taking note of the drier they

contain, and the way in which they have been prepared; Lippert intends to return to this branch of the subject in a future communication. Altogether he suggests that the speed of drying of an oil or boiled oil shall be defined as the time required for it to attain its maximum weight.
—F. H. L.

### (C.)—INDIA-RUBBER, &c.

*India-rubber, Vulcanised, The Analysis of.* C. O. Weber. Zeits. f. angew. Chem. 1898, 313.

*See under* XXIII., *page* 610.

### PATENTS.

*Pneumatic Tyres [India-rubber Cement], A Method and Liquid for Closing Punctures in.* G. B. Ellis, London. From La Société Chimique des Usines du Rhône, formerly Gilliard, P. Monnet et Cartier, Lyons, France. Eng. Pat. 12,076, May 15, 1897.

A solution is employed containing 30 parts of water glass, 20 parts of sugar, 5 parts of dextrin, and 45 parts of water, in which the sugar and dextrin may be replaced by other soluble carbohydrates, "and generally by any agglutinate soluble in water which is indifferent to water glass." The method of application consists in pouring about 100 grms. of the solution through the valve of the tyre, "which is then pumped up in the usual way, and the liquid is allowed to remain therein during use."—E. S.

*Gutta-Percha and like Substances, Impts. in the Treatment of.* W. Ramsay, London. Eng. Pat. 6344, March 15, 1898.

The object of this patent is to effect the polymerisation of purified gutta-percha, by which process the inventor states that the material becomes harder, tougher, and improved generally in its various properties. The refined gutta-percha is prepared by the usual process of solution in toluene followed by precipitation with acetone (or otherwise); it is then heated under pressure at 180° or 200° C. for 12 hours with 5 per cent. of hydrochloric acid, or with common salt, calcium chloride, or plain water. The product is macerated under hot water or in the dry state at a temperature exceeding 100° C., and is then ready for employment. By varying the temperature, pressure, and time of heating, and also the acid or salt dissolved in the water, different qualities of gutta-percha can be obtained: some more, some less, tough and resistant.—F. H. L.

*Reclaiming ["Devulcanising"] Rubber, Impts. in.* P. L. Clark, Chicago. Eng. Pat. 6968, March 22, 1898.

This process consists in soaking the article in a liquid solvent till it is thoroughly swollen, then heating it for one to three hours under a pressure not exceeding 100 lb. per sq. in. at a temperature ranging between 95° and 150° C., until the material is sufficiently softened to be fit for working up again. The solvent, preferably petroleum spirit, must be selected with a boiling point well below the melting or "disorganising" point of the rubber, and it must be capable of dissolving both the rubber and the sulphur. Although the substance is thus apparently devulcanised, no considerable loss of sulphur occurs; and re-vulcanisation may usually be effected without the addition of fresh sulphur.
—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Glutin-peptone Salts.* C. Paal. Ber. **31**, [7], 956—966.

By the action of dilute hydrochloric acid in excess on collagene (glue, gelatin), products are obtained containing more or less combined hydrochloric acid (Ber. **25**, 1202), to which the author has given the name of "glutin peptone salts." The molecular weight of these decreases as the percentage of combined hydrochloric acid rises, and the fact that those containing smaller amounts can be salted out from aqueous solution with ammonium sulphate shows that they belong to the class of propeptones (albumoses), whereas the non-precipitated derivatives, with lower molecular weight and containing more hydrochloric acid, may be classed among the true peptones as defined by Kühne. On the average, these glutin-peptone hydrochlorides in anhydrous condition contained:—Ash, 0·18 to 0·58; HCl, 10·38 to 13·14; C, 43·28 to 45·95, and H, 6·43 to 7·13 per cent.

It was found possible to effect a fractionation of these derivatives by means of dialysis. One specimen experimented with was hardly soluble in ethylic alcohol, but readily dissolved in cold methylic alcohol. The diffused salt, of which over 60 per cent. of the original substance was recovered, contained:—Ash, 0·89; HCl, 13·44; C, 42·27, and H, 6·69 per cent.; corresponding to C., 48·86, and H, 7·31 per cent. in the free peptone. On dialysing this latter salt into water, a further separation was effected, the portion diffusing within the first 24 hours being an extremely hygroscopic yellow mass very soluble in alcohol, and containing 1·21 per cent. of ash, and 16·06 per cent. of hydrochloric acid; whilst the portion diffusing in the second 24 hours was much less soluble in alcohol, less hygroscopic, and containing less hydrochloric acid (9·38 per cent.). The dialysis of another peptone hydrochloride yielded analogous results.

These glutin-peptone salts are partially precipitated by phospho-tungstic acid with liberation of the peptone. The author finds that the non-precipitated portion gives the biuret reaction plainly, and suggests that this may be accounted for by the theory that by the action of dilute hydrochloric acid on collagene, the main bulk is peptonised, whilst a small amount undergoes further hydrolysis, with the formation of products intermediate between peptones and amido acids. By treating the phosphotungstic precipitate with barium hydroxide in excess, a barium glutin-peptone is obtained, and this reacts with ferrous sulphate, yielding an aqueous solution of glutin ferropeptone. The latter changes on contact with the air, rapidly on warming, absorbing oxygen and forming free glutin-peptone, which remains in solution, and ferric oxide which is quantitatively precipitated.

Glutin-peptone salts dissolved in absolute alcohol and the solution saturated with gaseous hydrochloric acid, whilst warmed on the water bath under a reflux condenser, show only a trifling increase in the amount of combined hydrochloric acid, and further peptonisation only occurs in the presence of water.—C. A. M.

*Raw Tanning Materials, Analysis of.* W. K. Alsop and J. H. Yocum. J. Amer. Chem. Soc. **20**, [5], 338.

*See under* XXIII., *page* 611.

### PATENTS.

*Leather, The Manufacture of New Iron Compounds and their Application to the Preparation of.* J. E. Chevallot, Nanterre, France. [Under International Convention.] Eng. Pat. 14,052, June 9, 1897.

Ferrous sulphate in aqueous solution is subjected to the oxidising action of an alkali permanganate or bichromate in presence of such a proportion of sulphuric acid as to give a product in which the oxygen of the base is to that of the acid as one to three. The three products specified are $Fe_3O_4.4SO_3$; $Fe_4O_5.5SO_3$; and $Fe_5O_6.6SO_3$, and the proportions of the reagents required to produce any one of these are given. The following is the specification (given as an example) of the preparation of a tanning bath:—

Water, 100 litres; ferrous sulphate, 30 kilos.; sulphuric acid, 6·750 kilos.; potassium bichromate, 2·640 kilos.: and potassium sulphocyanide, 0·045 kilo. The sulphocyanide is added to give a drab-coloured leather. "The process of tanning consists in saturating the freshly unhaired hides or skins in a solution of one of the ferric salts described, agitating them in the moving apparatuses used in tanning and tawing. The operation lasts from 12 to 96 hours, according to the thickness of the hides or skins. The suppleness and the faculty of absorbing possessed by the leathers produced are greater accordingly as the salt employed has been more or less oxidised." Leather thus tanned is stated not to be liable to the growth of moulds.—E. S.

*Leather, Impts. in or appertaining to Waterproofing, and Treating Tan-Coloured and other Boots and the like Leather Goods.* D. Mart, Wigan. Eng. Pat. 2482, Jan. 31, 1898.

The leather is impregnated with a solid hydrocarbon, which is applied either by heating it until absorbed or when mixed to a paste or solution with a volatile hydrocarbon. Claim is also made for such pastes or solutions containing either solid hydrocarbons of the series $C_nH_{2n+2}$ or a mixture of bees-wax and hydrocarbons of this series and of the $C_nH_{2n}$ series in "mineral naphtha," oil of turpentine, or the like.—C. A. M.

## XV.—MANURES, Etc.

*Wiborgh Phosphate: Manure prepared from Gellivara Apatite.* L. F. Nilson. Kongl. landtbruksakad. handl. o. tidskr. 1898, 1—17.

The magnetite found at Grängesberg and Gellivara is often mixed with considerable amounts of apatite. To separate the two substances the mineral is crushed until the grains are about 1 mm., and the iron compounds are then separated magnetically. The iron ore thus obtained contains about 71 per cent. of iron, and very little phosphate, and is very suitable for the Martin process. The refuse contains about 80 per cent. of apatite, the rest being mainly felspar, with some quartz, mica, and non-magnetic ferric oxides.

As, at the present time, there is little prospect of this substance being utilised as basic slag, the following process has been proposed by Prof. Wiborgh, for converting it into a phosphatic manure. The process, which is suitable for all crude phosphates, consists in igniting the substance with sodium carbonate at about 900—1,000°. The following reactions take place between the carbonate and the apatite and felspar respectively:—

$$2Ca_5(PO_4)_3F + 3Na_2CO_3 = (Na_2O)_2(CaO)_{10}(P_2O_5)_3 + 2NaF + 3CO_2$$

and—

$$K_2Al_2Si_6O_{16} + 2Na_2CO_3 = K_2Na_4Al_2Si_6O_{18} + 2CO_2.$$

A sample of Wiborgh phosphate thus prepared contained: $P_2O_5$, 27·01; $SiO_2$, 9·99; $SO_3$, 0·27; $K_2O$, 1·54; $Na_2O$, 14·69; CaO, 38·12; MgO, 2·88; $Fe_2O_3$ and $Al_2O_3$, 4·50; and $F_2$ and loss on ignition, 1·00 per cent. It dissolves completely in hydrochloric acid, but only very slightly in water. The citrate solubility of the phosphoric acid is readily brought up to 95 per cent. In preparing the phosphate the best results are obtained with 30 parts of sodium carbonate to 100 parts of apatite, when the latter contains 17 per cent. of felspar. Apatite free from felspar is much less readily attacked by sodium carbonate, and the product contains much less citrate-soluble phosphoric acid.

Experiments in which oats, peas, and sugar-beet were manured with equal amounts of citrate-soluble phosphoric acid in the form of Wiborgh phosphate, basic slag, and superphosphate respectively, showed that Wiborgh phosphate and basic slag are about equal in value when large amounts are applied; in the case of beetroot, basic slag produced the most organic matter, possibly owing to a part of the phosphoric acid insoluble in citrate, being utilised.

—N. H. J. M.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Ranson Process [Sugar]. Results obtained by the.* Gouthière. Bull de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [10], 1011—1014.

The results obtained in working the Ranson process under very varied conditions during last season in France, Belgium, Germany, and Russia show an improvement in colour of at least 75 per cent., a great diminution of viscosity, facilitating working generally, particularly of low products, so that the second runnings could be boiled to grain, and molasses could be produced during the manufacture. The increase of rendement was 0·75 grm. per 100 kilos. of beetroot at Crépy; 0·6 to 0·7 at Lobejun; 0·5 at Voitovetz. The sugar obtained was whiter and of higher titre. In no factory was any formation of glucose observed, or any alteration or fermentation of low products. The process was everywhere found to be easily worked, and not complicated or delicate.—L. J. de W.

*Sugar, Baker Process for the Production of Extra White Centrifugal Sugar.* Puwrez de Groulard. Bull de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [10], 1007—1009.

Steam at 108° C. (0·4 atmosphere) is superheated to from 180 to 200° before its admission into the centrifugal machine. This steam may give up the whole of its heat in cooling to 100° C. without producing a single drop of water. The object of the process is to condense in the machine only the quantity of vapour necessary to wash the crystals and at the same time to heat the massecuite from the centre to the outside without any superfluous dissolving of sugar. Blue is introduced along with the steam by compressed air. An extremely small quantity (25 to 30 c.c. for a machine holding 250 kilos. of massecuite) is required.—L. J. de W.

*Invertive Power of Sulphurous Acid. [Sugar.]* L. Battut. Bull de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [10], 991—998.

By heating or evaporating syrup or molasses under conditions obtaining in practice, sulphurous acid is found to produce considerable inversion of sugar. The great factor in inversion appears to be, not the amount of free sulphurous acid present, nor its transformation into sulphuric acid, but the temperature. When the temperature of the liquor does not exceed 55° C., and the fall of temperature between the heating surface and the liquor is not too great, no inversion occurs. The great danger of working acid lies in the fact that any inversion cannot be detected after boiling a few hours, for the greater part of the acid having passed off, if the massecuite has become alkaline, the glucose produced is transformed and is no longer indicated by reagents. This is an unknown loss of sugar. With this reserve, provided all care is used, the author sees no disadvantage in boiling acid syrups if necessary.

—L. J. de W.

*Starch, The Saccharification of, by Malt Amylase.* H. Pottevin. Comptes Rendus, 1898, **126**, 1218—1221.

If malt extract be kept at 79°—80° C. for 15—20 minutes, it loses the power of converting starch into maltose, although it retains the power of liquefying starch paste. A 10 per cent. solution of starch was acted on for 12 hours at 60° C. by some malt extract thus heated. The optical activity of the solution was unchanged although the starch was completely liquefied. The solution was fractioned by alcohol into three portions. The most insoluble portion gave, with a trace of iodine, a blue coloration, changing to a violet brown with excess; the middle fraction gave a reddish tint, and the most soluble fraction no coloration. None of these fractions contained maltose, and when subjected to the action of fresh malt extract at 63° C., every 100 parts of each dextrin yielded respectively 71, 82, and 95 parts of maltose.

Wheat starch was acted on by malt extract at 63° C.; it gradually dissolved until only about 10 per cent remained. Two transformations were made under similar conditions, the one from the entire starch and the other from this final 10 per cent. They yielded maltose in the proportion of 75 and 41.

The author considers he has proved that the transformation of starch is the result of two distinct reactions—the conversion of starch into dextrin and the conversion of dextrin into maltose. Dextrins only differ physically. The tougher portions of a starch granule yield a dextrin more difficult to transform than the other portions.—A. L. S

*Carbohydrates, The Precipitation of, by Neutral Salts.* R. A. Young. J. of Physiology, 1898, **22**, 401—422.

It has long been known that many colloid substances, as well as certain carbohydrates, can be precipitated from their solutions by saturation with neutral salts. The reason of the present investigation is to determine whether this method can be employed to separate the various carbohydrates and their digestion cleavage products from one another. Neutral solutions containing from 1 to 2 per cent. of the carbohydrate were most frequently used. Saturation was effected by adding successive quantities of the finely powdered salt to solutions of the carbohydrate and shaking. An excess of salt was always present, and the solution allowed to stand for some time. The principal salts employed were ammonium sulphate, magnesium sulphate, and sodium sulphate. In some cases, especially with starch and its products of hydrolysis, a fractional precipitation was carried out either by half saturating with ammonium sulphate, filtering off any precipitate and then completely saturating with the same salt, or first saturating with magnesium sulphate, and then with sodium sulphate either at the ordinary temperature or at 33° C. The author's results may briefly be summarised as follows :—

The salt precipitation method can be as easily applied to colloid carbohydrates as to proteïns and can be used as a method for separating them. Ammonium and magnesium sulphate are most generally useful; sodium sulphate at 33° C. is sometimes very convenient. None of the crystalline sugars are precipitated by neutral salts, but some sugars form compounds with certain salts. Starch and soluble starch are both readily precipitable by these salts. Contrary to the views of Musculus Meyer and of Ost, erythrodextrin consists of a series of substances distinct, from starch or the achrodextrins. One erythrodextrin is not precipitated by salts; the precipitate obtained by saturation of mixed erythrodextrins can be fractionated into two. These three products the author distinguishes as erythrodextrins I., II., and III.; they give reactions very similar to the products obtained by Lintner and Düll. Most of the achrodextrins are not precipitated by salts.

Glycogen is readily and completely precipitated by neutral salts, especially by ammonium sulphate or by sodium sulphate, at 33° C. Inulin is partly precipitated from its solutions by magnesium and ammonium sulphate, the former being more active in this respect. All the soluble iodine compounds of starch, soluble starch and the erythrodextrins are precipitated by salts as a rule more easily than the original carbohydrate. The colour of the iodine compounds varies with the concentration of the iodine, and with the state of the compound itself, whether solid or in solution. Soluble starch gives a blue colour with iodine under all conditions, and has a greater attraction for it than the erythrodextrins, so that a trace of the former can be detected in the presence of a large excess of erythrodextrin, provided the iodine solution is weak and added carefully drop by drop.

There is no evidence of the existence of a combination of the colloid precipitated and the salt precipitating; the colloid precipitating power of a salt does not appear to be a function of its molecular weight or solubility.—J. L. B.

*Dextrins, Contributions to our Knowledge of the.* J. Henderson. Inaug. Diss., Munich, 1897.

The author has obtained the following results:—"Erythrodextrin II.*a*," having $[\alpha]_D = 194°$ and $R = 8\cdot5$, is obtained both by the action of diastase or acids on starch; generally speaking, the same dextrins are formed by the hydrolysis of starch with diastase or acids. The action of diastase practically ceases when the conversion products have a reducing power of 80 per cent. of maltose. By the hydrolysis of starch with diastase, dextrins are formed which resist the further action of the enzyme. These dextrins appear to have the composition of the "achrodextrin I. and II." of Lintner and Düll, but the possibility is not excluded that an "isomaltose" which is not attacked by diastase and yeast may occur with these substances.

The substances present in malt extract which cause the optical rotatory and cupric reducing power may be removed by fermentation, without impairing the hydrolytic action of the extract; fermentation is therefore recommended for the preparation of diastase. The torrefaction dextrins of commerce are distinguished from those obtained by diastatic and acid hydrolysis, by containing a more or less large proportion of substances of low rotatory and reducing power (probably reversion products), and the former dextrins cannot at present be identified with the latter.—J. L. B.

*Gums from German South-West Africa.* K. Dieterich. Ber. Deut. Pharm. Ges. **7**, [3], 1898.

Three gums derived from the Hinterland of Angra Pequeña —"Tlach," "Amrad," and "Auraur"—are thus described. Gum "Tlach" is soluble in 10 parts of water; the solution is without action on Fehling's reagent, and is entirely precipitated by alcohol. The mucilage is brown in colour, cloudy, and gives a gelatinous precipitate with ferric chloride solution. Gum "Auraur" is not entirely precipitated from aqueous solution by alcohol, but is rendered cloudy; it gives a precipitate with basic lead acetate, but not with the neutral lead salt. It reduces Fehling's solution. Gum "Amrad" does not reduce Fehling's solution, and is not precipitated by lead salts. All three gums are strongly adhesive, but, as they differ in chemical composition from the official gums, are not suited for use in medicine or in pharmacy.—J. O. B.

*Massecuite, Determination of Crystallised Sugar in.* E. Lallemant. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [10], 1038.

*See under* XXIII., *page* 611.

*Sugar, Estimation of, Electrolytically.* J. Formánek. Zeits. Unters. Nahr. und Genussmittel, 1898, [5], 350.

*See under* XXIII., *page* 611.

*Carbohydrates in the Presence of Iron, The Action of Hydrogen Peroxide on.* C. F. Cross, E. J. Bevan, and C. Smith. J. Chem. Soc. Trans. 1898, **73**, 463—472.

The results obtained by H. J. H. Fenton in oxidising tartaric acid by hydrogen peroxide in presence of ferrous salts suggested the application of the method to the sugars. The typical hexoses and cane sugar, when treated with hydrogen peroxide, are, as stated by Wurster, comparatively unacted on. If, however, a trace of ferrous salt be added, even at ordinary temperatures, and with an amount of hydrogen peroxide corresponding to 1 or 2 atoms of active oxygen, a marked rise of temperature occurs, and the solutions become acid to the taste, the quantity of acid formed from levulose being less than from dextrose. The volatile acids are formic and acetic acids, and represent 15—20 per cent. and 4—7 per cent. respectively of the weight of dextrose. A non-volatile acid forming an insoluble lead salt was isolated; it gave values on analysis agreeing with those required for tartronic acid.

After removal of the acids, the furfural in the distillate from hydrochloric acid (sp. gr. 1·08) in the case of dextrose or cane sugar corresponds with 7—9 per cent. of furfuroids. The products yielding furfural are not acid in character, but there is no evidence that they are pentoses. From levulose, only traces of these products are formed. The solutions give a well-marked iodoform reaction, indicating that the hexose molecules undergo internal re-arrangement, and that the phenomena are not those of a simple oxidation.

The products of the reaction, when dried in a vacuum, form a gummy solid, destitute of any appearance of crystallisation; they react with phenylhydrazine acetate in the cold, forming compounds resembling the osazones, and they also reduce Fehling's solution in the cold. The yield of osazones is considerable, amounting to 30—60 per cent. of the weight of the carbohydrate in the case of levulose and of cane sugar; and to 12—20 per cent. in that of dextrose. Two groups of osazones have been obtained: (1) compounds melting at 185°—195° and resembling the glucosazones in properties, but differing from them in composition, the nitrogen ($N = 17—20$ per cent.) being 2—3 per cent. higher; (2) compounds with a low melting point (130°), and freely soluble in hot water.

The authors conclude (1) that hydrogen peroxide acts primarily by determining a constitutional change in the hexose molecule, *i.e.*, by internal rearrangement, such effects bearing no direct proportion to the quantity added; and (2) that the oxidising actions observed, *e.g.*, the formation of dicarboxylic acids, are subordinate or secondary effects.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Fermentation without Yeast Cells.* E. Buchner. Ber. 1898, **31**, 568—574.

Pasteur regarded fermentation as a physiological act intimately connected with the life processes of the yeast cell. Other investigators such as Moritz Traube, Berthelot, Liebig, and Hoppe Seyler adopted the view that, since a chemical substance, the unorganised ferment or enzyme, invertin, which converted cane sugar into dextrose and levulose, was produced in yeast, that it might similarly engender a substance which would give rise to alcoholic fermentation. This theory could not, however, be supported by experimental evidence, so that the vitalistic hypothesis remained in undisputed possession of the field.

The author then proceeds to give an account of the preparation of a liquid obtained from yeast cells which could ferment (this Journal, 1897, 156). Amongst the enzymes present in pressed yeast are invertase, maltase, and an enzyme which hydrolyses glycogen. An oxydase appears to be present, as the pressed yeast becomes brown after standing some hours in contact with the air, probably accompanied with the absorption of oxygen (compare Bertrand, *ibid.*, 1894, 1071). Hahn has also found a proteohydrolytic enzyme. It has been shown that the gas produced by fermenting sugar with pressed yeast-extract is completely absorbed by alkalis, thus proving that it is carbon dioxide.

From the various experiments which have been carried out (*ibid.*, 1897, 549; 1898, 59), the author concludes that fermentation is not dependent on the living yeast cell, and is not, therefore, a physiological act, but is due to an enzyme-like substance zymase, which exists in the yeast cell. The isolation of zymase is not possible at present, on account of its instability, and the presence of other enzymes.—J. L. B.

*Bacterium of Sorbose, Action on the Polyatomic Alcohols.* G. Bertrand. Bull. Soc. Chim. 1898, **19**, [8], 347—349.

The author has shown that sorbite can be easily transformed into sorbose under the influence of a particular bacterium, hence termed the "Bacterium of sorbose." Among the polyatomic alcohols, glycol, xylite, and dulcite, are found to be unfavourable to the growth of the sorbose bacterium, and they resist its oxidising action, but glycerin, sorbite, and mannite, the coefficient of carbon of which is three or a multiple of three, support the growth of the microbe most favourably, and, at the same time, part with two atoms of hydrogen, forming the sugars "erythrite, arabite, volemite and perseite" respectively. There is, therefore, a direct relation between the stereochemical structure of these alcohols and their behaviour with this ferment, since it is only those which contain a link CH.OH so placed that there is not an atom of hydrogen beside an attackable hydroxyl group, which are oxidised by the sorbose bacterium.—J. O. B.

*Gentianose, The Physiology of; and Inversion by Soluble Ferments.* E. Bourquelot. Comptes Rend. 1898, **126**, 1045—1047.

The author points out the physiological similarity existing between gentianose (this Journal, 1898, 257) and cane-sugar, in respect of these sugars undergoing inversion and being utilised by the plant during the growing period. Experimental proof is brought forwards to show that the fresh roots of *Gentiana lutea* contain no enzyme capable of inverting gentianose, whilst the reverse is the case with a growing plant of *G. acaulis*. Diastase and emulsin are without action upon gentianose, but *Aspergillus* water inverted it completely. Experiments showed that invertin acted more slowly on gentianose than cane sugar, and further, that whilst the inversion of saccharose was complete, that of the gentianose was not. In explanation of this result, the author states that *Aspergillus* water contains besides invertin, other enzymes capable of hydrolysing polyglucoses.—J. L. B.

*Malt, The Dangers of Judging by the "Sinker" Test.* N. van Laer. J. Fed. Inst. Brewing, 1898, **4**, 300—311.

This method of judging the quality of a malt consists in taking 100 corns and placing them in a glass of water. After stirring well, the "sinkers" are allowed to settle, the malt being judged by the quantity of these; a large number being considered indicative of a bad malt. The author condemns this method, since the number of corns which sink, varies from day to day in the same sample of malt, and also with the water, whether distilled or natural. Experiments with a series of malts taken at different times from the kiln illustrate these variations. The following analysis of malt in its original form and that of the sinkers separated therefrom is given:—

| — | Malt. | | Sinkers. | |
|---|---|---|---|---|
| | I. | II. | I. | II. |
| Diastatic capacity | 40·0 | 20·0 | 33·3 | 14·0 |
| Opticity of wort $[\alpha]_D$ | 121·9 | 112·5 | 121·5 | 114·2 |
| Colour of wort | 2·65 | 2·8 | 3·2 | 3·2 |
| Saccharification period | 30 min. | 39 min. | 58 min. | 55 min. |
| Condition of wort | Bright | Fairly bright. | Fairly bright. | Fairly bright. |
| Extract | 93·81 | 88·09 | 92·66 | 84·0 |
| Moisture | 1·1 | 1·4 | 2·5 | 2·0 |
| Acidity | .. | 0·12 | .. | 0·159 |

It will be seen from the above analyses that the chemical composition is different to what might have been expected. They indicate poverty in diastase, the corns not having grown or only partially so, whereas the diastatic capacity is but little less than in the non-sinking portion.—J. L. B.

*Beer Yeast, Investigations into the Degeneration of.* A. Jorgensen. Zeits. für d. ges. Brauwesen, **21**, [10], 113—117.

As a rule selected races of yeast retain their chief properties, in practical use under ordinary conditions, except when bacteria or wild yeasts make their appearance. In some instances, however, the yeast acquires fresh characteristics, which are persistent so long as the prevailing conditions are maintained. The ordinary microscopical examination and test methods of culture do not suffice to determine whether these changes are due to an actual modification of the yeast itself or to an invasion by other races of culture yeast. In the author's laboratory the following method is employed to solve this problem:—A large number of cells are taken, as an average sample, of the suspected yeast, and if the microscopical and biological examination fails to reveal the presence of strange species, it is assumed that actual degeneration has occurred. To determine whether this is general or confined to a portion of the cells, a large number of pure cultures are prepared from the sample, each culture being allowed to develop normally, without forcing, and then employed for fermentation experiments in large sterilised glass vessels, a uniform wort being used throughout.

In this way it has been shown, in the case of degenerated low-fermentation yeast, that the cultures possessed different powers of attenuation and could be classified in groups; the degeneration, when manifested by insufficient attenuation, being remediable by selecting the most active cultures for reproduction, a method which yielded results similar to those obtained from the original stock of the culture yeast in question.

A case is also cited where portions of a pure race (low-fermentation) yeast acquired, as a fixed habit, the property of developing a bitter flavour in beer wort, though no morphological difference could be detected, whereas other cells in the same batch retained their original characteristics, and it was found possible to obtain a good yeast by giving them facilities for reproduction. On the other hand, the superficial film vegetation developed in a pure top-fermentation yeast stored for some time in beer wort exhibited a morphological variation from the main stock, the cells being thinner and more elongated, and also giving rise to bitterness of flavour. The cause of these alterations is considered by the author to arise from the altered conditions of environment to which these film vegetations are exposed, from the presence of air and accumulations of albuminoid matters, hop resins, &c., in consequence of which varieties may develop by natural selection.—C. S.

*Yeast, The Utilisation of the Expressed Sap of.* O. Reinke. Zeits. für Spiritusind. 1898, 125.

The liquid expressed from yeast by Buchner's process is very rich in albuminoids and ferments, and might be employed for a yeast nutriment, for a similar purpose in vinegar manufacture, for fermenting concentrated sugar solutions, for medicinal purposes, and for the after-conditioning of beers.—A. L. S.

*Furfural in Beer.* W. Windisch. Wochenschr. für Brau. 1898, 189—191.

A number of beers were tested for furfural by distilling, collecting the first 5, 10, and 20 c.c. separately, and testing with aniline hydrochloride. The distillate is collected in a small white basin, three drops of pure aniline are added and a drop of pure hydrochloric acid. The whole is then stirred with a glass rod, the drops of aniline at length collecting in the centre, at the bottom of the basin. Finally, a drop of hydrochloric acid is added. The presence of only traces of furfural is indicated by a distinct reddening of the liquid, and especially of the undissolved drops of aniline. The first distillates were found to give no reaction, whilst the last fractions invariably gave a well marked furfural reaction. As an aqueous solution of furfural gives when distilled the furfural reaction with the first few drops of distillate, the author concludes that furfural is not present as such in the beer, but is formed during the boiling. It was also found that the manner in which the beer was heated influenced the production of furfural, a beer which gave furfural when boiled over the naked flame gave none when the flask was heated on wire gauze.

Experiments were made on the presence of furfural in worts, and it was found that infusion worts did not contain furfural, but decoction worts invariably did, and that furfural was produced by the boiling of the mash, its amount being dependent on the acidity of the worts.

As the author on no occasion found furfural in the beer, but always in the decoction wort from which the German beer is prepared, he made experiments to determine how the furfural is destroyed, and finds that yeast very quickly destroys that body, even if present in comparatively large quantity. It is evident, therefore, that the furfural found in spirit is not a product of the fermentation, but is produced by the distillation. In the same way the author found that furfural was produced by pasteurising beer. The bread-like smell and taste sometimes noted in beers the author states to be characteristic of the destruction of furfural by yeast.—A. L. S.

*Mashing Temperatures, Appropriate.* Krieger. Amerik. Bierbrauer; Allgem. Anzeig. für Brauereien, **14**, [13], 401—404.

The main point in mashing is the regulation of the conversion of starch, too little being at present accurately known respecting the influence of the mashing temperature on the nitrogenous constituents of malt for these to be borne in mind, notwithstanding their undoubted connection with the formation of head.

The following general rules for worts of ordinary gravity may be deduced from scientific research and practical experience:—

Temperatures of 25°—50° C. facilitate the modifying action of peptase and cytase on the albuminoids and cell-texture; those up to 66¼° C. are the most favourable for saccharification; the malto-dextrins are produced most extensively between 67½° and 70° C.; flavorless dextrins are formed up to 75° C.; and higher temperatures are fatal to diastase. It is also established that when the mash is heated to any given temperature, the corresponding changes alone occur, even if the temperature be subsequently reduced.

As regards the practical application of these rules, malt imperfectly modified on the floor, steely, through defective curing, or too highly kilned, can be in part rectified by keeping the mash at 25°—50° C. for some time—maximum two hours.

To ensure high attenuation and keeping quality in the beer, the mash should be slowly raised to 65° C. to facilitate the formation of maltose; and palate fulness may be secured by taking care to reserve sufficient starch for conversion into intermediate products at 67½°—70° C., an object attained by quick mashing, a high initial temperature, or by the English or American systems of mashing. Should an insufficiency of prepared starch for the purpose be present, the deficiency must be supplied by the addition of suitable preparations or of cooked unmalted grain; and, if the mash has been overheated, more malt or diastase, must be added.

The foregoing temperatures apply to worts of average strength, and should be raised in the case of more highly concentrated worts, small-grained malt, or high percentages of diastase, albumin, or acids—the latter arising from the presence of an abundance of ungerminated corns and favouring the formation of sugar, whilst diastase will stand greater heat in concentrated solutions, and is more active when present in a high ratio to starch. On the other hand, low-gravity worts and malts poor in diastase, require mashing at temperatures below the average.—C. S.

*Wine, Micro-organisms of Ropy.* F. Bardas, Joulin, and de Raczkowski. Comptes Rend. 1898, **126**, 1050—1053.

The authors have isolated several micro-organisms from the wines of Algeria and the south of France. Among these are two filiform bacilli, which behave differently under similar conditions of cultivation.

One of them, which is called *B. roseus vini*, is never met with alone, it occurs with other micro-organisms, more particularly, however, with the second but unnamed filiform bacillus. *B. roseus vini* possesses properties different from those usually attributed to the so-called ropy filaments. In yeast water containing 10 per cent. of glucose, this organism grows as a thick folded film, becoming pink some days after being sown, whilst the medium turns brown and develops ammonia. The bacillus is ovoid in shape, possesses flagella, is mobile, forms spores, and changes nitrates into nitrites. When grown in yeast water containing glycerin, a substance is produced which is apparent on the second organism, but disappears subsequently. It is a reducing body, and forms a compound with phenylhydrazine; the authors believe that it may be dihydroxyacetone (compare Bertrand, Comptes Rend. 1898, **126**, 842; this Journal, 1898, 502). The bacillus is without action on alcohol, and does not ferment cane sugar. When sown in wine, *B. roseus vini* produces an abundant deposit. The glucose and glycerin had diminished, whilst the tartrate and acidity were unaltered.
—J. L. B.

*Champagne, Chemical Composition of.* L. Grünhut. Zeits. Anal. Chem. **37**, 231—242.

This is a reply to Kulisch's objection (Mitth. über Weinbau und Kellerwirthschaft, **9**, 11, 1897) to the author's statement that champagnes are often prepared from diluted wines and wines too attenuated, by gallising ("Gallisiren"). In support of this contention, the author quotes a number of old analyses of sweet champagnes from various sources, and 14 unselected analyses of dry champagnes, recently analysed by W. Fresenius and himself. Out of a total of 38 analyses, 13 show a content of mineral substances of less than 0·14 grm. in 100 c.c., six contain 0.14 grm., and nine contain 0·15 grm. According to the author, these data indicate extraordinary dilution, for although natural wines are known which do contain very small amounts of mineral substances, still they are very exceptional. Moreover, this poverty cannot be ascribed to assimilation by the ferment during fermentation in the bottle, for this would only suffice to reduce the amount by about 0·01 grm. per 100 c.c. In the case of champagnes, the addition of the large amount of sugar which is required to give the necessary percentage of alcohol (9—11 grms. in 100 c.c.) tends, by the consequent increase of glycerin, so to raise the amount of extract that occasionally this figure gives no indication of an over-gallised wine. According to the author, however, the "total extractive residue," which is calculated by subtracting the amounts of non-volatile acids, of glycerin, and of mineral substances from the extract, should not be less than 0·35 grm. in 100 c.c. for natural wines, and moderately gallised wines. Of the new analyses quoted, however, in five cases this extreme limit was nearly approached, and there is no single case in which the value reaches 0·80 to 1·00, which is a common figure for ordinary wines.

Moreover, the author points out that, whilst a legal limit is placed on the attenuation of ordinary wines by gallising, there is no such limit for sparkling wines; this he regards as a safe indication that such a practice is common.

Finally, in support of the statement, objected to by Kulisch, that the amount of sugar in champagne reaches 18 grm. per 100 c.c., the author cites three analyses of sweet champagnes, showing a content of 17·31, 18·28, and 17·61 respectively.—E. W. W.

*Moulds in the Distillery, Function of.* L. Levy. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [10], 998—1007.

It has been the custom to regard moulds as harmful and destructive, but the useful services they may render are worthy of notice. Their injurious effects are of only secondary importance, and may be avoided by scrupulous cleanliness. Their useful applications depend on the formation of diastases capable of acting on starch and certain sugars, and, under special conditions, on the production of true fermentation.

These applications are illustrated by an account of *Aspergillus oryzæ*, which not only saccharifies starch, acts on dextrin and maltose, and inverts cane-sugar to the extent of 70 per cent., but peptonises albuminous matters. *Amylomyces Rouxii* has similar properties, but does not seem to act on saccharose. The moulds, however, are not capable of supporting an entirely anærobic existence, and they leave a considerable amount of carbohydrate unacted upon. The addition of yeast to amylomyces doubled the yield of alcohol by acting on the residual carbohydrate, but the percentage of alcohol the moulds can support is small, hence the disadvantage of a large consumption of fuel and the enormous vats required.—L. J. de W.

*Wine and Acetic Acid, Detection and Approximate Estimation of Sulphuric Acid in.* F. Bimbi. Bull. Chim. Farm. **37**, 133.

*See under* XXIII., *page* 611.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Food-Preservatives, Organic.* R. Hefelmann. Zeits. fur öffentl. Chem. **4**, 128—131; Chem. Centr. 1898, **1**, [14], 854.

At the present time the only organic preservatives of any importance are benzoic acid, salicylic acid, saccharine, and formaldehyde. Benzoic acid and saccharine have no particular antiseptic action, but according to physiological experiments, they are completely harmless in the ordinary dilution. Salicylic acid, which is the most important organic antiseptic for foods, is also stated to be harmless in the proportions usually employed. On the other hand, the author is opposed to the preservation of foods by means of formaldehyde, since the latter, according to physiological observations, acts on the albuminoid substances present, and considerably reduces the digestibility of the foods. The author considers that organic preservatives should be judged according to these principles, and that only these should be tolerated, under a "declaration of control," that are appreciably antiseptic; in the ordinary dilution, neither act directly toxic nor sensibly retard digestion; rapidly pass undecomposed through the body; are employed in a soluble form; and, finally, may be accurately detected analytically. —A. S.

*Food-Preservatives, Inorganic.* F. Filsinger. Zeits. für öffentl. Chem. **4**, 121—128; Chem. Centr. 1898, **1**, [14], 853—854.

It is stated that the following substances are useful as preservatives:—Sodium chloride, aluminium compounds, especially alum and aluminium sulphate, metaphosphoric acid and its salts, hydrofluoric acid and its salts, potassium nitrate, sulphur dioxide and bisulphites, and boric acid and borates; but in practice, besides the substances—nitre and common salt—usually employed in former times, only compounds of sulphurous and boric acids are now of any importance. There is a great diversity of opinion as to the amount of sulphur dioxide which is injurious, and an official regulation is considered necessary. With regard to compounds of boric acid, the author considers that in small doses they are quite harmless. In conclusion, the author suggests that there should be a thorough physiological examination of organic preservatives, a fixing of the highest permissible quantities, and a "declaration of control" for the preserved foods.—A. S.

*Yeast, New Process for the Preparation of Preserved.* E. Buchner. Zeits. f. Spiritusind. 1898, **21**, 152. (Ger. Pat. 97,240.)

The pressed yeast which has hitherto been used in bakeries possesses the disadvantage of undergoing bacterial and other changes during the warm weather. The author has found (this Journal, 1897, 156) that alcoholic fermentation is not a direct consequence of the living yeast cell, but is due to an enzyme "zymase" present in the contents of the cell, and further that (*ibid.*, 1897, 548) "zymase" can withstand a gradual drying at low temperatures, and in a nearly dry condition, a heating at 100° C. A yeast dried at high temperatures, and therefore dead, can thus be employed for purposes of fermentation.

The washed yeast is carefully drained, spread out in thin layers, and air-dried. After one or two days the temperature is raised from 50° to 100° C., and the heating continued until the yeast is dead. The product may or may not be ground. Experiments have shown that an addition of 5 to 10 per cent. to the flour suffices for raising the dough. —J. L. B.

*Annatto in Milk, Detection of.* A. Leys. J. Pharm. Chim. 1898, **7**, [6], 286.

*See under* XXIII., *page* 608.

*Farinaceous Materials, Detection of Wood Sawdust in.* G. A. le Roy. Comptes Rend. 1898, **126**, [14], 1047.

*See under* XXIII., *page* 608.

*Sesame Oil in Margarine.* M. Siegfeld. Chem. Zeit. 1898, **22**, [32], 319.

*See under* XXIII., *page* 611.

*Glutin-peptone Salts.* C. Paal. Ber. **31**, [7], 956.

*See under* XIV., *page* 589.

*Unboiled Milk, Distinguishing Boiled from.* H. Leffmann. Analyst, 1898, **23**, [265], 85.

*See under* XXIII., *page* 608.

### (*B.*)—SANITATION; WATER PURIFICATION.

### PATENTS.

*Sewer Air or other Obnoxious Gases, and also Sewage Matter during Transit in Drains, Impts. in or connected with the Treatment of.* R. H. Reeves, Putney. Eng. Pat. 9566, April 14, 1897.

The process consists in forcing a spray of water over chemicals, which are thus taken up and diffused throughout the air shaft or man-hole of a sewer. The chemicals are contained in two vessels fitting in a recess of the man-hole, and consist of dilute sulphuric acid and a solution of potassium permanganate or a mixture of lime and tar respectively.

These are run from the two vessels into a mixing cup and from thence on to porous plates, where they meet the water spray. The water-supply is so arranged that it seals off the sewer gas from contact with the outside air.—W. P. S.

*Sewage, Impts. in or relating to the Treatment of.* R. H. Reeves, Putney. Eng. Pat. 9558, April 14, 1897.

The use is claimed of a mixture of quicklime and gas-tar, this mixture being added to the sewage at the outfall. The gases produced by mixing the tar and lime are also used as deodorants.—W. P. S.

*Smoke or Foul Air Mask, for enabling Persons to Breathe in Poisonous Atmospheres.* R. Dowling, Reading. Eng. Pat. 12,452, May 20, 1897.

The mask consists of a casing, which may be of metal or other suitable substance, and is shaped and padded to fit the face of the wearer. A glass window is in the front and the whole is attached to the head by tapes or strings. Air is pumped into the mask by the wearer by means of a suitable pump, the said air being passed through an absorbent or antiseptic material on entering the mask. An exit valve is of course also provided.—W. P. S.

*Sewage and other Polluted Waters and Liquids, Impts. in or relating to the Treatment and Purification of.* W. J. Lomax, Bolton; and T. Aspinall, Farnworth, near Manchester. Eng. Pat. 14,058, June 9, 1897.

A process is claimed in which sewage is treated with chlorine gas (or bromine or iodine) with or without the previous addition of lime or other precipitants.—W. P. S.

*Nightsoil, Garbage, Waste Products, and other Noxious Matters, Impts. in Furnace Processes and Apparatus for the Destruction of.* H. G. Downton, Croydon, N.S.W., and W. H. Nicholls, Croydon, N.S.W. Eng. Pat. 26,625, Nov. 15, 1897.

The matter to be destroyed is completely incinerated in a closed oven or retort, which is so constructed that the gases or products of combustion are led from the oven over a water-tank under the heating furnace. A portion of the fumes is condensed and the remainder, after being mixed with hot air, is passed into the furnace. The night-soil, &c., is filled into the oven by an opening at the top, and as the ashes collect they are drawn off through suitable doors in the sides of the oven.

The construction of the plant is claimed and the various parts of it, consisting of the oven itself, a specially constructed furnace, an automatically closing water-tank, and openings with covers for filling the oven.—W. P. S.

*Mask or Inhaling Apparatus for Use in Noxious Atmospheres, An Improved.* Fredk. Wanz and Franz Wanz, Vienna. Eng. Pat. 30,032, Dec. 18, 1897.

The apparatus consists of a hood fitting the head of the wearer closely. In front is a glass window, which is provided with a "wiper" inside, and under this window, in the lower part of the mask, is a bag or pouch for containing solid caustic potash for absorbing respired carbonic acid and moisture. Oxygen is supplied from a cylinder containing this gas in a compressed state, the cylinder being preferably separate from the mask, and connected to it by a flexible tube.—W. P. S.

*Apparatus for Use in Sterilising or Disinfecting, applicable also for use in Bacteriological Culture; Impts. in or relating to.* E. Fournier, Paris. Eng. Pat. 1725, Jan. 21, 1898.

The apparatus claimed, comprises a chamber closed with an air-tight cover, and a heating lamp. The chamber rests upon an outer casing, through which are ventilating holes. This outer casing surrounds the lamp.

The lamp consists of a main heating burner, connected by means of a tubular passage to an annular reservoir containing the combustible liquid, the reservoir being placed outside the above-mentioned casing. The flame may be increased or extinguished by a lever extending through the outer casing. A small burner is also provided and is connected to the same annular reservoir. A special feature of this burner is a metallic gauze hood over the wick. This small burner serves to keep the temperature constant after the chamber has been once heated up by the main burner.

When the apparatus is to be used for cultures, a vessel is used which fits inside the above chamber, and is provided with open tubes for aëration.

For wet or dry sterilising, another vessel is used, being placed inside the main chamber, and is supplied with an air-tight lid having a screw-plug opening. This plug has a small orifice through it, which may be closed by turning the plug, and allows air to enter or steam to escape from the vessel. Steam pipes also enter this vessel.—W. P. S.

### (C.)—DISINFECTANTS.

*Formaldehyde, Action of, on Germ Power.* W. Kinzel. Landw. Vers.-Stat. **49**, 461—466; Chem. Centr. 1898, **1**, [13], 785.

The author carried out a series of experiments to determine the utility of formaldehyde as a means for the destruction of spores of mildewed fungus in seed corn. The trials were carried out with rye, wheat, oats, barley, clover, lupin, and pea, and also with *Ustilago segetum Bull.* of green oats, half-ripe oats, and ripe oats. The results upon the whole show that there should be no objection to the practical employment of a 0·1 per cent. formaldehyde solution for the destruction of spores of mildewed fungus by one hour's action. Such a 0·1 per cent. solution still showed complete efficacy after 10 days.—A. S.

*Carbon Monoxide, Action of Reagents on, and Determination of, in the Air.* A. Gautier. Comptes Rend. **126**, [12], 871.

*See under* XXIII., *page* 603.

### PATENTS.

*Barrels, Casks, and the like, Impts. in or relating to the Purification or Sweetening of.* W. J. Engledue, Byfleet, and C. J. Yarnold, Brixton. Eng. Pat. 9300, April 12, 1897.

The process consists in subjecting the previously washed or steamed barrels to a current of ozonised air.

The plant claimed is composed of a filtering and drying chamber for the air used, a fan, which draws the air from this chamber into the ozoniser, a receptacle for the barrels, and a furnace for consuming the exhausted ozone and other vapours.—W. P. S.

*Insecticides and Fungicides, Impts. in.* A. Blackie, Tunbridge Wells. Eng. Pat. 14,715, June 17, 1897.

The claims are for a number of solutions of insecticides in various combinations, more particularly—(1) soap, alcohol, and extract of quassia; (2) soap, alcohol, and aloes; (3) soap, alcohol, and potassium or sodium sulphide, or both; (4) a mixture of (1) and (3); (5) a mixture of (2) and (3).—W. P. S.

## XIX.—PAPER, PASTEBOARD, Etc.

*Paper, Deterioration of; Report of the Committee on the.* Sir J. Evans (chairman); Sir O. Tudor Burne; Sir W. Anderson; M. Carteighe; G. Clulow; C. F. Cross; R. Garnett; H. Müller; W. J. Russell; H. R. Tedder; W. L. Thomas; J. M. Thomson; Q. Wirtz; Sir H. Trueman Wood (secretary). J. Soc. Arts, 1898, **46**, [2374], 597—601.

The main purpose of the work of the committee has been to ascertain the validity of the supposition that the fibrous raw materials—esparto, "mechanical wood" or ground wood pulp, wood celluloses, &c.—more recently introduced are inferior, with regard to the relative permanence of the paper made from them, to the older celluloses obtained from cotton, flax, and hemp. The physical qualities of paper may be expressed under the following three heads:—

*Strength*, measured in terms of the weight or strain which it will support.

*Elasticity and Texture*, measured by elongation under strain and resistance to rubbing.

*Bulk*, expressed in the precise terms of specific gravity or weight per unit of volume.

There must also be taken into account the properties belonging to the paper as a chemical substance (or mixture), *i.e.*, its actual composition, and the liability to change under whatever conditions of storage or use to which it may be subjected. The committee feel convinced that these physical and chemical constants must come more and more into practical consideration, and ultimately regulate the consumption and, therefore, production of papers.

With regard to the deterioration of papers, the Committee have come to the following conclusions:—The tendencies to deterioration of papers are marked (1) by disintegration, (2) by discoloration. These are independent effects, but may be concurrent, and indeed are notably so in paper containing mechanical wood pulp.

1. *Disintegration*.—Actual disintegration has been found to occur in all grades of paper; it is the result of chemical change of the fibres themselves, and in some cases could be traced to the effect of illuminating gas upon the atmosphere of the rooms in which the books had been stored. In the case of the rag papers examined, the effects appeared to be due to acid bodies, which may have been present in the original paper as made, may have resulted from reactions going on in the paper itself after making, or may have been due to products of gas consumption. With papers made from mechanical wood pulp, disintegration is due to oxidation, and is accompanied by an alkaline reaction of the paper.

*Discoloration* is also stated to affect all papers to a certain degree, in some cases a uniform discoloration taking place, whilst in others the more marked effect of a darkening of colour spreading inwards from the margins, occurs. The former is the result of changes in the paper itself, whilst the latter is caused by external means, such as the action of an atmosphere charged with the products of combustion of coal gas. As a general conclusion, it is stated that discoloration of ordinary cellulose papers (as distinguished from those containing mechanical wood pulp), under usual conditions of storage, is proportional to the amount of rosin which they contain, or more generally to the rosin and the conditions employed for fixing it in the ordinary process of engine-sizing. With regard to paper likely to be used in chemical laboratories, &c., it should be noted that mechanical wood pulp is stained a deep yellow, and esparto and straw celluloses, pink, by aniline and other coal-tar bases.

The following hints are given in connection with the preparation of printing papers:—

*Sizing* (engine-sizing).—Whenever possible, book paper should be sized with a minimum quantity of rosin. Two per cent. (calculated dry) is suggested as a maximum, and it is stated that the quality of "hard-sizing," usually produced with large proportions of rosin, is superfluous for nearly all the ordinary uses of printing papers. The presence of starch is objectionable, though its employment may be necessary in the case of heavily-loaded papers.

*Acidity*.—A slightly acid reaction (acid to litmus, neutral to Methyl Orange) should be given to the paper by finishing with the normal slight excess of alum.

*Chlorides*.—The paper should contain a minimum quantity of chlorides.

*Colour*.—It is stated that in ordinary practice the question of the permanence of the colour of the paper when made is not much considered. The committee suggest that when very white printing papers are required, the sizing should be reduced to the practical minimum; and also that for ordinary printing papers a lower standard of colour might be adopted.

*Loading*.—It is concluded that 10 per cent. of total mineral constituents is the extreme limit for papers for publications of permanent value.

*Texture*.—This quality is largely controlled by the mechanical process of preparation (beating) of the fibres, but is imitated by the addition of sizing and loading agents. It is essential, for publications of permanent value, that paper be used, the close texture of which has been obtained by the method of mechanical preparation.

Paper-making fibres are classified in the following manner with regard to the permanence of papers prepared from them:—A. Cotton, flax, and hemp; B. Wood celluloses, (*a*) sulphite process, and (*b*) soda and sulphite process; C. Esparto and straw celluloses; D. Mechanical wood pulp (see J. Soc. Arts, 1897, **45**, 690—696).

The committee suggest that the normal standard of quality for book papers required for publications of permanent value should be specified as follows:—

*Fibres*.—Not less than 70 per cent. of fibres of Class A.

*Sizing*.—Not more than 2 per cent. of rosin, and finished with the normal acidity of pure alum.

*Loading*.—Not more than 10 per cent. of total mineral matter (ash).

For written documents, the paper should be prepared from fibres of Class A., and should be sized with gelatin, and not with rosin.—A. S.

*Waste Liquor from Sulphite Wood Pulp Manufacture.* H. Seidel and L. Hanak. Mitt. d. Tech. Gewerbe-Museums in Wien, 7, 1897, [12], 283.

In previous articles the authors have shown that an organic substance containing sulphur can be obtained from waste sulphite liquor in different ways, and the products were proved to be similar by the corresponding amount of sulphur contained therein.

Since then, different boilings from the same Silesian cellulose mills have been analysed, and the former statements corroborated.

The products obtained reduce Fehling's solution, are soluble in water, show an acid reaction, are insoluble in absolute alcohol and in a concentrated solution of common salt, give a precipitate with phenylhydrazine, give the reaction with α-naphthol and sulphuric acid, give the same shades of colour with ferric chloride, and almost the same percentages of mineral ash and sulphur. They also give precipitates with barium chloride and lead acetate, soluble with difficulty, but cannot be completely precipitated by the latter reagents.

The percentage of sulphur was slightly lower than in the products first analysed, being about 8 per cent., but it always corresponded within very narrow limits in the products of the same liquor, again showing the identity of the organic product, and also proving that the amount of organically combined sulphur depends upon the manner of conducting the boiling process.

To obtain the principal constituent of the organic matter as pure as possible, a dilute solution of waste liquor was acidified with hydrochloric acid and boiled, and to this barium chloride was added in the quantity calculated to completely precipitate the sulphuric acid present. The filtrate showed no turbidity with either barium chloride or sulphuric acid, and as the barium salt of the organic substance is soluble in water, this filtrate therefore contained it in solution. After evaporating part of the latter, alcohol was added, and a precipitate obtained, which the authors termed "Gum free from sulphuric acid." This precipitate yields 13·32 per cent. of ash, which, calculated as calcium sulphate, gives the percentage of CaO as 5·64. With oxalic acid, the CaO was calculated as 5·66 per cent. The amount of sulphur contained was 8·44 per cent. The substance shows all the before-mentioned reactions.

The authors then endeavoured to substitute the calcium in the organic salt by other metals, but whilst they succeeded in this substitution in the case of barium, they only attained partial success with zinc, the experiments being carried out as follows:—

15 grms. of gum free from sulphuric acid were dissolved in 150 c.c. of water, and digested with 9 grms. of crystallised barium chloride. Alcohol was then added, giving a brown precipitate, which was decanted with alcohol

and carefully dried. This was then incinerated, and the ash treated with sulphuric acid. The barium was then estimated by precipitation, and also in the melting tube, according to Carius's method, to eliminate the error caused by small quantities of soluble sulphates, the mean of the analyses showing a percentage of 14·49 of BaO. The sulphur contained was 7·77 per cent. The substance contained no chlorine. The zinc salt was obtained by dissolving 5 grms. of the gum in 100 c.c. of water and adding 2 grms. of zinc chloride. The ash from the precipitate with alcohol gave 6·95 per cent. of ZnO and 7·86 per cent. of sulphur.

On comparing the three results the metals were found to be combined in almost molecular proportions. This leads to the conclusion that the precipitates obtained are salts of a definite organic acid, which is the principal constituent of the organic matter in the waste liquor, and is produced by the action of the sulphite lye upon the non-cellulose constituents of the wood. This supposition is further supported by the percentage of sulphur in the different precipitates, which, when calculated in proportion to the molecular weight of the salt, gives, for the barium salt, 9·04; calcium salt, 8·94; and the zinc salt, 8·44 per cent. of sulphur, the average being 8·80 per cent.

Ten grms. of the organic barium salt were mixed with the calculated amount of sulphuric acid, and the mixture was dissolved in excess of water. A small amount of barium sulphate which was formed was filtered off. The mixture was allowed to stand for some days at a temperature of about 40° C., a white precipitate of barium sulphate being gradually formed, which was filtered off. This treatment was continued for about three weeks, until no further precipitate was produced. No trace of either barium or sulphuric acid could then be detected in the liquid by the ordinary analytical methods. The solution was evaporated to a syrup and alcohol added. No precipitate was obtained, showing that the pure organic acid is soluble in alcohol. A brown flaky precipitate was obtained from this aqueous alcoholic solution by ether, and this was carefully dried. The amount obtained was about 3 grms.

The substance thus purified is a brown, glossy, hygroscopic powder, soluble in water. It has an acid reaction, reduces Fehling's solution, yields with phenylhydrazine a yellowish-white precipitate, and gives precipitates with barium chloride and acetate of lead, which are soluble with difficulty. The ash, estimated as BaO, amounts to 1·26 per cent.

The percentage of sulphur was 8·80, which corresponds with the amount obtained from the calcium, barium, and zinc salts.

The average elementary composition of the ash-free organic substance containing sulphur is therefore :—From Silesian waste liquors: C, 53·69; H, 5·22; S, 8·80; O, 32·29 per cent. From Austrian waste liquors: C, 56·27; H, 5·87; S, 5·52; O, 32·24 per cent. Consequently much more sulphur, and less hydrogen and carbon, are contained in the Silesian than in the Austrian waste liquor.

When allowed to stand for some months in the open air, the sulphite liquor deposits $CaSO_4$, and turns lighter in colour. The dried substance obtained contains only 6·72 per cent. of sulphur, and the liquor itself contains 0·067 per cent. of sulphur as sulphate. Oxidation of the organic matter has therefore taken place.

In the reduced solution left exposed in the open air, a light yellow gum-like precipitate was thrown down with alcohol, containing 7·01 per cent. of sulphur. On distilling with steam, a milky liquid was obtained showing the same properties as the gas; it contains furfural. The quantities obtained, however, show no definite proportion to the amount of waste liquor used.

Nitric acid acts energetically on the organic substance obtained from waste sulphite liquor. A considerable number of acids could be obtained, one of which was identified as oxalic acid.

The halogens have also an energetic action on waste liquor. With chlorine the liquor becomes blood-red, and a yellow precipitate is deposited, which decomposes on heating, giving off hydrochloric acid. It can be dried in a vacuum, is soluble in water and alcohol, reduces Fehling's solution, and gives a brown precipitate with phenylhydrazine. On oxidising with fuming nitric acid, chlorine and sulphur can be recognised.

A remarkable occurrence in the sulphite cellulose manufacture is a deposit of sulphur in the wooden pipes through which the waste liquor is discharged from the digesters. In these pipes a greyish-white crystalline substance settles down, which has the following composition: — Water, 6·28; calcium sulphate, 87·41; FeO and $Al_2O_3$, 0·13; silica, 0·28; sulphur, 5·75; total, 99·85.

The occurrence of this sulphur shows that the $SO_2$ in the digester is not only oxidised, but some of it is reduced as well.

A waste liquor from Württemberg was analysed. The composition proved to be similar to that of the Silesian liquor.—S. P. E.

### PATENTS.

*Paper, Impts. in the Manufacture or Treatment of.* W. A. Hall, Vermont, U.S.A. Eng. Pat. 12,208, May 17, 1897.

The inventor finds that although formaldehyde precipitates glue from its solution instantly, if the latter be incorporated with the clay, satin-white, or other material used in "facing" paper before the formaldehyde is added, the action is considerably delayed, the mixture adheres well to the support, and will not wash off when dry. Suitable proportions are: clay, &c., 85 per cent.; albumin, glue, or casein, 13 per cent.; 40 per cent. solution of formalin, 2 per cent.—F. H. L.

*Peat, Manufacture of Fibrous Material suitable for Making Paper from.* K. A. Zschorner, Vienna, Austria. Eng. Pat. 15,797, July 2, 1897.

Washed peat is treated at the ordinary temperature, and under pressure, with a dilute (2 per cent.) solution of caustic alkali, removed, and washed. Then follows a treatment with a dilute solution (1°—2° B.) of calcium or sodium hypochlorite, likewise under pressure. If necessary, the operations can be repeated several times, but then more dilute solutions are preferably employed. It is claimed that the use of the dilute solutions and the low temperature prevent the fibres from being injured during the process.—H. I.

*Peat Fibres, Improved Process for the Manufacture of Chemically Prepared.* C. Geige, Düsseldorf-Grafenberg, Germany. Eng. Pat. 881, Jan. 12, 1898. (See also Eng. Pat. 20,501, Oct. 30, 1895; this Journal, 1896, 353.)

The peat, after leaching with alkali, is dried and disintegrated. It is then placed in a bath containing ½—¾ per cent. of sulphuric acid, or 1 kilo. of diastase per cubic metre, and maintained at 50°—60° C. for several hours, whereby the starchy matters are converted into glucose. A treatment in a bath containing 1—1·5 litres of yeast per cubic metre at 30°—40° C. for 4—5 days follows. The fermentation converts the glucose into alcohol and carbonic acid. The fibres are then removed, washed, and the resins and fats dissolved out by treatment with hot ether or petroleum spirit. Washing with water, boiling with dilute acid or alkali, and a final washing, complete the process.—H. I.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Cerite Group, Commercial Salts of the.* N. Orlow. Farmaz. J. 1898, **20**, 3; Chem. Zeit. Rep. 1898, **8**, 66.

The cerium salt which is most used therapeutically is the oxalate, which occurs in commerce in various colours—white, yellow, rose, and violet—indicating the presence of impurities. Purchased *cerium oxalicum* showed on analysis about 60 per cent. of didymium compounds—even more than was found in a purchased specimem of didymium sulphate.—H. B.

*Neodidymium.* O. Boudouard. Comptes Rend. **126**, [12], 900—901.

By treating oxides of the yttria group, as sulphates, with potassium sulphate solution, a double sulphate separated from the solution after some hours, the oxide from which, when carefully separated from any thoria also present, gave the reactions and the spectrum of neodidymium compounds. The author is investigating the solubilities in alkali sulphate solutions of the double sulphates of these metals and potassium, as promising a better method than that of fractional crystallisation, for separating neodidymium from praseodidymium.—J. T. D.

*Acid Phosphoglycerates.* Adrian and Trillat. Comptes Rend. 1898, **126**, 1215—1218.

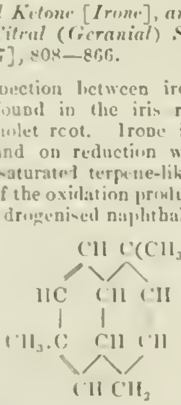
The acid phosphoglycerates may be prepared by decomposing the neutral salts by the theoretical quantity of sulphuric acid, using Helianthin as an indicator; or by double decomposition between the acid barium salt and a soluble sulphate.

The acid phosphoglycerates are distinguished from the neutral salts by their greater solubility in water, and by being precipitated by alcohol with much greater difficulty. They do not crystallise, and their solutions are decomposed by boiling with the formation of the neutral salt, glycerin and phosphoric acid, and by alkalis; the neutral salts being formed. This reaction may serve for their estimation, the titration being performed with a standard solution of potash, using phenolphthalein or Helianthin as an indicator.

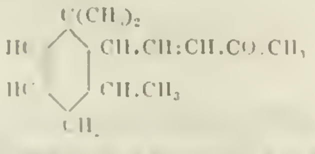
The acid phosphoglycerates are also decomposed by solutions of the organic bases, and thus the neutral phosphoglycerate of the base may be prepared.—A. L. S.

*The Violet-scented Ketone [Irone], and the related Compounds of the Citral (Geranial) Series.* F. Tiemann. Ber. 1898, **31**, [7], 808—866.

There is no connection between irone, the ketone of violet-like odour found in the iris root, and iridin, the glucoside of the violet root. Irone is a methyl ketone, $C_{11}H_{17}.CO.CH_3$, and on reduction with hydriodic acid, it passes into the unsaturated terpene-like hydrocarbon irene, $C_{13}H_{20}$. A study of the oxidation products of this body have shown it to be a hydrogenised naphthalene derivative—



From the transformation of irone into irene by reduction and the formation of chloroform and the unsaturated acid $C_{11}H_{17}.CO_2H$ by the action of a solution of alkali hypochlorite, it follows that the constitution of irone is represented by the formula—



Ionone is an isomer of irone, obtained by the action of weak alkalis on citral and acetone, pseudo-ionone, an unsaturated aliphatic ketone, being thus first produced, and by the action of dilute acids, is converted into the isomeric ionone. (Eng. Pat. 8736, 1893; this Journal, 1897, 1038.) Ionone, on reduction, forms a hydrocarbon, $C_{13}H_{18}$, ionene.

*Citral.*—Methylheptenone is obtained by the oxidation of citral, and also by its decomposition with weak alkalis (Verley, this Journal, 1897, 351). The ketone so obtained, which is identical with that occurring in nature, has been synthetised by Barbier and Bouveault and by Verley, and thus its constitution and that of citral proved (*loc. cit.*). Citral is separated from essential oils by means of its sodium bisulphite compound; this must be decomposed by alkalis at as low a temperature as possible. If methylheptenone be also present, it must first be removed by steam distillation and distillation *in vacuo*. Citral is recognised by its oxime, boiling at 143°—145° C. under 12 mm. pressure, which may be converted into geranionitrile boiling at 110° under 10 mm. Doebner's method consists in heating 20 parts of citral with 20 parts of β-naphthylamine and 12 parts of pyrotartaric acid, when citryl-β-naphthocinchoninic acid, readily crystallisable from alcohol and melting at 197° C., is formed.

*Geranic Acid.*—

$$C(CH_3)_2:CH.CH_2.CH_2.C(CH_3):CH.CO_2H$$

(see Ber. **26**, 2717, for physical properties). This acid is best prepared as follows:—55·7 parts of bromethylacetic ester, 42 parts of methylheptenone and 1 part of zinc filings are warmed on the water-bath until the reaction begins, zinc is then gradually added until the calculated quantity (21·8 parts) has been used. The mixture is warmed on the water-bath for three hours, poured into dilute sulphuric acid (1 : 10), shaken for 12 hours, extracted with ether, the ethereal solution washed with dilute acid until it no longer contains zinc, the solvent distilled off and the residue fractionated. The oxydihydrogeranic ester so obtained is a colourless liquid, boiling at 150° under 25 mm.; it is saponified by heating for 5—6 hours with 15 per cent. aqueous potash. The acid forms a colourless oil, boiling at 168° C. under 8 mm., slightly soluble in water. On shaking with 70 per cent. sulphuric acid, it is readily converted into the cyclic isogeranic acid, melting at 106° C. Geranic acid is obtained from oxydihydrogeranic acid by boiling with an equal weight of acetic anhydride and one-third of its weight of anhydrous sodium acetate. The oily product which separates on the addition of water is shaken with dilute soda to remove the larger part of the acetic acid it contains, and then steam-distilled, the first runnings being rejected. The impure acid is then dissolved in soda, neutral impurities extracted by ether, and the acid separated by sulphuric acid. Under 18 mm. pressure, geranic acid boils at 157·5°—159·5° C. A mixture of calcium geraniate and formate diluted with sand gives, on dry distillation *in vacuo*, citral in considerable quantity. Geranic acid is readily recognised by its conversion into isogeranic acid by shaking with 60—70 per cent. sulphuric acid, or by warming with dilute mineral acids. The isogeranic acid, after repeated crystallisations from petroleum ether, melts at 106°; 103°—105° was formerly given.

*Geraniol.*—

$$C(CH_3)_2:CH.CH_2.CH_2.C(CH_3):CH.CH_2.OH.$$

Citral is readily obtained by the oxidation of geraniol with chromic acid, and can be reduced to geraniol by sodium amalgam in feebly acid (acetic) alcoholic solution (Ber. **29**, 906; this Journal, 1896, 564). To obtain a pure product, the solution should be heated with alkali, to decompose unaltered citral, and steam-distilled, rejecting the last portions of the distillate. For the preparation of pure geraniol see Tiemann and Krüger, this Journal, 1896, 469; and Bertram and Gildemeister, 1898, 267. The geranyl ester of diphenylcarbamic acid serves for the identification of geraniol (this Journal, 1896, 292; 1897, 823). This compound is, however, more simply obtained by the action of diphenylurea chloride upon the sodium alcoholate suspended in ether.

*Linalool.*—

$$C(CH_3)_2:CH.CH_2.CH_2.C(CH_3)(OH).CH:CH_2.$$

This alcohol differs from the compounds of the same series already described in its optical activity. In essential oils, mixtures of *l*- and *d*-linalool are generally found. Thus, linalool differs in activity according to its source. Mixtures of the two modifications are known under the names of aurantiol, lavendol, nerolol, &c. The purest form of *d*-linalool is obtained from coriander oil. The most pure form of *l*-linalool is known as licareol. The physical properties of these two alcohols are given in the following table:—

| —— | Licareol. | Coriandrol. |
|---|---|---|
| Boiling point ........ | 86°—87° under 14 mm. | 85°—86° under 12 mm. |
| Density.............. | 0·8622 at 20° | 0·8726 at 17·5° |
| Refractive index $n_D$ .. | 1·46108 | 1·46455 |
| Molecular refraction . | 49·13 | 48·78 |
| Rotation in 1 dm. tube | 16° 55′ | 11° 36′ |
| $[\alpha]_D$ ................. | 19° 37′ | 13° 19′ |

*Pseudoionone.*—

$C(CH_3)_2:CH.CH_2.CH_2.C(CH_3):CH.CH:CH.CO.CH_3$.

Acids and alkalis gradually convert pseudoionone into ionone; if the reaction become violent, resins are produced.

Pseudoionone may be recognised by converting it into ionone by boiling with 10 per cent. mineral acid for 1—2 hours, extracting with ether and distilling *in vacuo*. The ionone so obtained is converted into the *p*-bromphenylhydrazone, as described in this Journal, 1895, 825. Another method is to allow 1 part of the oil to be tested, to drop slowly into 3—4 parts of well-cooled concentrated sulphuric acid, allowing the temperature to rise to 30° C. at the conclusion. Then pour over ice, extract with ether, steam-distil the ethereal extract, dissolve in alcohol, and add rather more than the calculated quantity of an aqueous solution of semicarbazide hydrochloride containing excess of sodium acetate. The semicarbazone of β-ionone crystallises out in several hours; after recrystallisation from alcohol this is obtained in characteristic prisms, melting at 148° C.

*Ionone* is most readily purified by means of the semicarbazone (this Journal, 1895, 827); if the ionone contain pseudoionone, its semi-carbazone is decomposed by the prolonged action of acids into ionone, similarly also the oxime and phenylhydrazone. Ionone unites with sodium bisulphite under the same conditions as pseudoionone (*i.e.*, on boiling with an excess of sulphite solution). Ionone is recognised by means of the bromphenylhydrazine test (*loc. cit.*), or by preparation of β-ionone semicarbazone) see following abstract).—A. C. W.

*Ionone, The Resolution of, into Two Isomers: α- and β-Ionone.* F. Tiemann. Ber. 1898, **31**, [7], 867—881.

By the conversion of pseudoionone into ionone by means of concentrated sulphuric acid, E. de Laire (American Pat. 600,429, 1898) obtains an ionone of practically the same boiling point as the ketone described in the preceding abstract, but formed a different semicarbazone, and from which the characteristic β-bromophenylhydrazone is only obtained with difficulty. Ionone is converted by concentrated sulphuric acid into the same substance, which the author calls iso-ionone or β-ionone, α-ionone being ordinary ionone.

*β-Ionone* is obtained from ionone or ψ-ionone by the process described for the recognition of ψ-ionone in the preceding abstract. The steam-distillate is fractionated *in vacuo*, the fraction boiling between 135° and 145° under 18 mm. is dissolved in alcohol, and the semicarbazone prepared as described. After repeated recrystallisation from alcohol the compound of β-ionone is separated from the more readily soluble α-ionone carbazone. By decomposition with dilute sulphuric acid in alcoholic solution, addition of water, extraction with ether, and fractionation of the extract, β-ionone is obtained boiling at 127°—128·5° C. under 10 mm. pressure, of density, 0·946 at 17° C., and refractive index, 1·521. The odour of β-ionone is not essentially different from that of α-ionone. The *p*-bromophenylhydrazone of β-ionone is more soluble in the ordinary solvents than the α-ionone compound.

*α-Ionone.*—In all preparations of ionone the β-modification is formed; similarly, all ionones prepared with the strongest acids contain some α-ionone. If the oily oxime, prepared from ordinary ionone, be kept for a long time at a low temperature, it crystallises; the crystals, after recrystallisation from petroleum ether in a freezing mixture, consist of pure α-ionone-oxime, melting at 89°—90°. This may also be prepared from the ionone separated from the mother liquors of β-ionone semicarbazone (see this Journal, 1895, 827). The physical properties of α- and β-ionone and of ordinary ionone, and some of their derivatives, are compared in the following table:—

| —— | α-Ionone. | β-Ionone. | Ordinary Ionone. |
|---|---|---|---|
| Boiling point, 18 mm. pressure. | .. | 140° C. | .. |
| Boiling point, 17 mm. pressure. | 134°—136° | .. | .. |
| Boiling point, 14·5 mm. pressure. | .. | 134·5°—135·5° | .. |
| Boiling point, 12 mm. pressure. | .. | .. | 126°—128° |
| Boiling point, 11 mm. pressure. | 123°—124° | .. | .. |
| Boiling point, 10 mm. pressure. | .. | 127°—128·5° | .. |
| Density at 20° C. ....... | 0·932 | 0·946 | 0·9351 |
| Refractive index $n_D$ ..... | 1·4980 | 1·521 | 1·507 |
| Oxime. melting point ... | 89°—90° | Oil | 89°—90° |
| Oximeacetate, melting point. | 98°—99° | 103° | 103° |
| Semicarbazone, melting point. | 107°—108° | 148° | 109°—110° |
| *p*-bromphenylhydrazone, melting point. | 142°—143° | 115°—116° | 142°—143° |
| Hydrazone— $C_{13}H_{20}:N_2:C_{13}H_{20}$ melting point. | 99° | 104°—105° | .. |

—A. C. W.

*Geraniol, Decomposition Products of.* Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 27.

Wallach and Naschold have stated that geraniol is completely resolved into hydrocarbons, when heated in an autoclave at 250° C., the principal product being dipentene. It has been found, however, that when geraniol is thus treated at 200° C. partial decomposition results, and an alcohol, differing from geraniol, but having the same odour and physical properties as linalool, is obtained, which differs from linalool, however, in its optical properties, since it is inactive. This was converted into the sodium salt similar to the sodium-linalyl-acid-phthalate, and the alcohol liberated from this combination had the odour and physical characters of linalool. The matter is being further investigated.—J. O. B.

*Rose, Oil of.* F. Dietze. Süd-deutsche Apoth. Zeit. 1898, 174; Apoth. Zeit. **13**, 212; through Chem. Centr. 1898, **1**, [17], 1001.

The author replies to the criticisms of Raikow (this Journal, 1898, 386), attributing the figures obtained by the latter for the free acid and ester number of rose oil to the use of an oil filtered at normal temperatures, by which, the stearoptene being removed, higher values were necessarily obtained. Dietze obtained the following results with filtered and unfiltered oils:—

*Unfiltered Oils.*—Free acid number, 2·1 and 1·7; ester number, 4·2 and 12·8; ratio number, 6·3 and 14·5. *The same oils filtered:* Acid number, 2·3 and 1·7; ester number, 5·0 and 14·0; ratio number, 7·3 and 15·7. The ratio number is only to be taken into consideration when the saponification numbers are not abnormally high. The congealing point should, in the author's opinion, be taken as the point at which crystals of stearoptene begin to form.
—J. O. B.

*Rosemary Oil, Adulteration of.* Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 41—42.

An instance of adulteration with, or possibly substitution of, light camphor oil for rosemary oil is recorded. This sample had a specific gravity of 0·885 at 15° C., and its rotation was +22° 28′ at 10° C. It was soluble with slight turbidity in 12 volumes of 80 per cent. alcohol. When distilled, however, nothing was obtained below 160° C. The German customs regulations require rosemary oil used for denaturing alcohol to have a sp. gr. 0·880 to 0·900, and to give a clear solution with 12 volumes of 80 per cent. alcohol; on distillation, 5 per cent. must pass over below 160°, and at least 90 per cent. between 160° and 230° C. These requirements are misleading, as pure Italian rosemary oil has a specific gravity of 0·900 to 0·909, a rotation of

+ 0° 45′ to 4° 30′, and gives a clear solution with 10 parts of 80 per cent. alcohol.—J. O. B.

*Sandal-Wood Oil, East Indian.* Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 43—45.

COMMENTING on the recently published paper on the purity of sandal oil by W. Dulière (Ann. de Pharm. **3**, 553; and **4**, 4, 8, 52, and 98), Messrs. Schimmel in the main agree with that author's standards, but extend them thus:—Sp. gr. 0·970 to 0·980; solubility in 70 per cent. alcohol, 1 in 5; saponification number at most 15, equivalent to 7 per cent. of santalyl acetate; percentage of santalol at least 90.—J. O. B.

*Mace Oil* [*Volatile*]. Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 35.

MACE oil is stated to be generally distilled from rejected nutmegs, and the true volatile oil of the axillus of the fruit is not met with in commerce. The genuine volatile oil of mace has the following characters:—Sp. gr. 0·905 at 15° C., opt. rot. + 11° 7′ at 17° C., and is soluble in two volumes of 90 per cent. alcohol; whilst nutmeg oil has a sp. gr. of 0·865 to 0·920; opt. rot., + 14° to + 30°, and is soluble in three volumes of 90 per cent. alcohol.—J. O. B.

*Cedar-Leaf Oil.* Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 14.

IN America, where this oil is chiefly distilled, considerable confusion arises from the application of the name "cedar" to several distinct species of conifers. Thus, in addition to the leaves of the true American red cedar, *Juniperus virginiana*, those of *Thuja occidentalis* and of *Chamæcyparis sphæroidea* are indiscriminately used by distillers, and even the oils obtained from these erroneous sources are not always free from admixture. Thuja oil is frequently sold as "cedar oil," and true oil of red cedar appears to be unobtainable in commerce. The authentic oil distilled from the leaves of *Juniperus virginiana* has a sp. gr. of 0·887 and an optical rotation of + 59° 25′ in 100 mm. It is insoluble in 16 parts of 80 per cent. alcohol. The odour is pleasant and sweet. It consists mainly of limonene, cadinene, some borneol, and small quantities of bornyl esters, the saponification number being 10·9 and the acetylisation number 39·1. Valerianic acid is probably contained in combination in one of the esters present.—J. O. B.

*Essence of Portugal* [*Sweet Orange*]. J. Flatau and H. Labbé. Bull. Soc. Chim. 1898, **19**, [8], 361—364.

A PRELIMINARY investigation on the bodies which give to sweet orange its peculiar and distinctive aroma has led the authors to attribute this, in great part, to a hitherto undescribed ester; this is found to be insoluble in alcohol. After distilling off 95 per cent. of the limonene, this is precipitated from the residue by treatment with alcohol, the form of a yellow powder, having the characteristic odour of the oil and melting at 64°—65° C. It is readily soluble in ether and in chloroform, and absorbs large quantities of bromine. Neither the component alcohol nor the acid have yet been isolated in sufficient purity to permit of their identification, but the analytical data so far obtained point to the probability of the latter containing 21 atoms of carbon. A further investigation is being proceeded with.
—J. O. B.

*Mandarin-Orange Oil.* J. Flatau and H. Labbé. Bull. Soc. Chim. 1898, **19**, [8], 361.

THE ester isolated by the authors from sweet orange oil, as described above, is also present in mandarin-orange oil, to the extent of 1 per cent., together with a trace of citral and 98 per cent. of limonene.—J. O. B.

*Methyl Salicylate, Plants in which it is found.* E. Kremers and M. M. James. Pharm. Review, **16**, 1898, 100—105; through Semi-annual Report of Schimmel and Co., Leipsic, April 1898, 51—52.

METHYL salicylate has been detected in the following plants:—*Betula lenta, Spiræa ulmaria, Erythroxylon coca, Polygala senega, P. Baldwinii, P. variabilis, P. javana, P. oleifera, P. serpyllacea, P. calcarea, P. vulgaris, Hypopitys multiflora, Gaultheria procumbens, G. fragrantissima*, and *G. leucocarpa*. To this list *Thea sinensis* must be added.—J. O. B.

*Methyl Salicylate.* Adrian. Journ. Pharm. Chim. 1898, **7**, [9], 122—424.

THE following table contains the result of distillations of 1 kilo. of wintergreen oil and methyl salicylate:—

| — | Methyl Salicylate. | | Oil of Wintergreen. | |
|---|---|---|---|---|
| | Weight. | Density at 15°. | Weight. | Density at 15°. |
| | Grms. | | Grms. | |
| Water | 5 | .. | 5 | .. |
| Before 220° (nearly all at 219°). | 110 | 1·1785 | .. | .. |
| 220°—222° | 105 | 1·1780 | 615 | 1·185 |
| 222°—223° | 155 | 1·1790 | 190 | 1·185 |
| 223°—225° | 350 | 1·1800 | 85 | 1·185 |
| 225°—230° | 185 | .. | 50 | 1·185 |
| Residue | 90 | .. | 55 | .. |

The author concludes that pure methyl salicylate distils between 220° and 223°. It is almost insoluble in water, and readily soluble in alcohol and ether. The aqueous solution gives a violet coloration with ferric chloride. Methyl salicylate forms a potash salt crystallising in pearly leaflets; excess of potash saponifies it. If the ester be mixed with five or six volumes of concentrated ammonia, it dissolves after some time, and on evaporation of the solution and distillation of the residue, a yellow mass of salicylamide is obtained. Allowed to drop on to anhydrous lime or baryta, methyl salicylate is converted into anisol, and the carbonate of the base is produced.

Adulteration with alcohol or fixed oils would lower the density: both would also be recognised on distillation. Methyl salicylate, or one of the distilled fractions mixed with an equal volume of sulphuric acid, gives no rise of temperature and only a slight yellow coloration. Oil of wintergreen, and especially the fraction 220°—222° (containing the terpene gaultherylene), give under the same treatment a considerable rise in temperature and a rose coloration changing to a wine-red, and finally to brownish-red.—A. C. W.

*Guaiacum Resin. Investigation of the Constituents of.* J. Herzig and F. Schiff. Monatsh. für Chem. 1898, **19**, [2 and 3], 95—105.

IN former researches (this Journal, 1897, 352; 1898, 267) the authors have shown that the acid of this resin contains two methoxyl and two hydroxyl groups. The products of its decomposition by heat are guaiacol and pyroguaiacin; for the latter, the results of ultimate analysis and the methoxyl group determination point to the formula $C_{12}H_{11}O(OCH_3)$. Acetyl pyroguaiacin is obtained by means of acetic anhydride and sodium acetate; it crystallises from hot alcohol in flat shining needles, melting at 122°—124°. The formula of this compound was shown by analysis and molecular weight determination to agree with the formula $C_{12}H_{10}(OH)(OCH_3)$ for pyroguaiacin. The acid of guaiacum resin by ordinary processes yields a diethyl derivative, showing its hydroxyl groups to be alcoholic rather than phenolic in character. The compound obtained by the reduction of this diethyl derivative by hydriodic acid, had the melting point of norguaiacum resin acid (185° C.), and its acetyl derivative that of acetylnorguaiacum resin acid (100°—102° C.).—A. C. W.

*Terpenes, A Compound of some, with Alkali Salicylates.* Duyk. Bull. Acad. roy. Belgique, **35**, [3], 15. Chem. Centr. 1898, **1**, [15], 892.

ON shaking a solution of sodium salicylate with a terpene there is formed a crystalline compound, which, by dissolving in water, or by heating for some time at 100° C., is decomposed into its components again.—A. S.

### *Kola Nut, Content of Caffeïne in Fresh and Dried.* G. François. Répert. Pharm. 1898, [3], **10**, 60.

Knebel has stated that the substance *kolanine*, found by him in the nut, breaks up under the influence of a ferment present in the nut into caffeïne, glucose, and kola-red, and that, as this process must have gone farther in the dried nut, the latter must be richer in alkaloid than the fresh nut. The author has shown that this is not so. He also finds that the red and white nuts, which are found often on the same branch, show no differences in the amounts of alkaloid that they contain.—J. T. D.

### *Scopolamine.* E. Schmidt. Arch. Pharm. 1898, **236**, 47.

The scopolamine hydrobromide of commerce often has a smaller optical rotation than that specified in the German Pharmacopœia. This is due to admixture with an optically inactive variety, which is formed during the extraction of the bases from the root, by the action of the alkalis used. The author has isolated this inactive scopolamine, and finds it to be identical with Hesse's *atroscine*.—J. T. D.

### *Scopolamine.* E. Schmidt. Arch. Pharm., 1898, **236**, 9.

The German Pharmacopœia has substituted for *Hyoscinum hydrobromicum* ($C_{17}H_{23}NO_3.HBr + 3\frac{1}{2}H_2O$), *Scopolaminum hydrobromicum* ($C_{17}H_{21}NO_4.HBr + 3H_2O$). The name Scopolamine should not, as Hesse proposes, be expunged and substituted by Hyoscine for the base $C_{17}H_{21}NO_4$; the latter name ought to represent an isomeride of Atropine and Hyoscyamine.—J. T. D.

### *Some Scopoleïnes.* W. Luboldt. Arch. Pharm., 1898, **236**, 33.

Scopoline readily forms esters. Scopolamine is a tropylscopoleïne. Acetyl-, benzoyl-, and cinnamyl-scopoleïnes are now to be had commercially. The author has prepared *salicylscopoleïne*, consisting of white needles, readily soluble in alcohol, slightly in water, and *phenylglycolylscopoleïne*, as well as several of the salts of both these bodies. Artificial tropylscopoleïne is an isomeride of, but not identical with, the natural scopolamine, which becomes identical with the artificial product on long heating at 230° C. The artificial substance has a less powerful action on the eye than natural scopolamine.—J. T. D.

### *Ouabaïn, Products of Duplication of, by Hydrolysis.* — Arnaud. Comptes Rend. 1898, **126**, [17], 1208.

The author, induced by the criticism of Feist (Berichte, **30**, 537) as to the identity of the reducing sugar formed by the hydrolysis of ouabaïn, first stated by him (Comptes Rend. **126**, 346; this Journal, 1898, 268) to be rhamnose, has repeated his experiments, and has obtained physical and chemical data which prove conclusively that this is correct. To the resin which forms simultaneously with the sugar in the process of hydrolysis, the formula $C_{24}H_{36}O_8$ is assigned; on drying, this parts with four molecules of water, giving a body having the composition $C_{24}H_{28}O_4$. The hydrolysis of ouabaïn is represented by the equation—

$$C_{30}H_{46}O_{12} + H_2O = C_6H_{12}O_5 + C_{24}H_{36}O_8.$$

—J. O. B.

### *Codeine, Synthesis of.* [*Methylation of Morphine by means of Diazomethane.*] Bayer and Co. Ger. Pat. 92,789; through Chem. and Drug. 1898, **52**, (941), 694.

The process consists in adding, to a cold ethyl alcoholic solution of diazomethane, the strength of which has been previously determined by titration with iodine, an equivalent molecular weight of morphine in very fine powder, or in solution in methylic or ethylic alcohol. As soon as the nitro compound begins to change colour, the solvent may be removed by evaporation, and the codeine, which, if it does not solidify spontaneously, will do so on rubbing; it may be purified in the usual manner.—J. O. B.

### *Pancreatins, The Activity of.* E. Choay. J. Pharm. Chim., 6 ser., **7**, [9], 418.

It is found that if the aqueous extract of pancreas, instead of being evaporated in the air at a temperature not exceeding 45° C., as directed by the Codex, be dried *in vacuo* not above 38° C., the saponifying action on fats is markedly increased, in the ratio of 60:104. The proteohydrolytic and amylohydrolytic action is practically the same as that of a carefully prepared specimen of official pancreatin. A commercial pancreatin examined, was found, however, to possess but little action upon fibrin, and less than half the normal amylohydrolytic action of the official sample. It was almost inert towards fats.—J. O. B.

### *Alkaloids in Tinctures and Extracts, Determination of.* J. Katz. Pharm. Zeit. 1898, **43**, 31, 273.

*See under* XXIII., *page* 612.

### *Hyperborates and Hypertitanates.* P. Melikoff and L. Pissarjewsky. Ber. **31**, [7], 953.

*See under* VII., *page* 579.

### *Cerium Salts, Application in Dyeing and Calico Printing.* R. Gnehm. Rev Gén. des Mat. Col. 1898, **2**, [16], 135.

*See under* VI., *page* 577.

### *Iodoform, Determination of.* G. Meillière. Rep. Pharm. 1898, [3], **10**, 102.

*See under* XXIII., *page* 611.

### *Atropine, Estimation of.* H. M. Gordin and A. B. Prescott. J. Amer. Chem. Soc. **20**, [5], 329.

*See under* XXIII., *page* 612.

### *Partial Racemism.* A. Ladenburg and W. Herz. Ber. 1898, **31**, [7], 637.

*See under* XXIV., *page* 614.

## PATENTS.

### *Ichthyol, Manufacture of Compounds of.* W. L. Wise, London. From Knoll and Co., Ludwigshafen a Rhine, Germany. Eng. Pat. 11,344, May 6, 1897.

The claims are for the manufacture of compounds of ichthyol and albumin, obtained by precipitating a solution of ichthyol and albumin by means of a stronger acid, or by the addition of free ichthyol sulphonic acid to an albumin solution, and for the conversion of the compound so obtained or other non-fluid ichthyol precipitates, into nearly odourless and tasteless substances by prolonged heating (at 125° C.), or by extraction with a solvent such as alcohol.—A. C. W.

### *Halogen-albumin, Process for the Production of Divisional Products of, by means of Alkalis.* F. Blum, Frankfort, Germany. Eng. Pat. 12,817, May 24, 1897.

The process is claimed for separating chlor-, brom-, and iodalbumin into different products, which consists in heating the halogen compound with alkali and filtering. The precipitate is purified by washing with water and alcohol. From the filtrate, acids separate a substance which is partially soluble in alcohol. The products of these processes are also claimed.—A. C. W.

### *Aldehydes, Impts. in the Method of Preparing, from Alcohols.* J. B. Cohen, Leeds. Eng. Pat. 13,214, May 28, 1897.

The process for the manufacture of aromatic aldehydes from alcohols is claimed, which consists in subjecting the alcohol to the action of nitrogen tetroxide in the gaseous or liquid form, and in solution in chloroform or other solvent by which it is not decomposed.—A. C. W.

### *Essential Matters contained in Aromatic Plants, Impts. in Apparatus for Extracting, and Analogous Purposes.* M. Otto, Neuilly, France. Eng. Pat. 14,305, June 12, 1897.

The apparatus claimed, consists of three vessels, connected together and to a pump or compressed air supply, and each provided with a worm condenser. The first vessel contains the solvent, the second the substance to be exhausted, and has means for circulating the solvent, the third receives the solvent charged with extracted matters. At the commencement of the operation, connection is made between the first and second vessels; when exhaustion is complete, between the second and third, from the latter of which the solvent is evaporated and received in the first. At the end of the last exhaustion, the exhausted materials are heated; the solvent distils and collects in the first vessel. The modification is claimed which consists in replacing the worms by suction and delivery pumps, so that the apparatus may be adapted for the use of solvents of very low boiling point.—A. C. W.

*Xanthines and their Derivatives [Synthesis of Theobromine and Caffeine], Impts. in the Manufacture or Production of.* George W. Johnson, London. From C. F. Bohringer and Söhne, Waldhof, Mannheim, Germany. Eng. Pat. 5949, March 10, 1898.

The claims are for the preparation of alkylised chloroxanthines by the action of phosphorus oxychloride upon alkylised uric acids, having no alkyl group in the position 9; and for the preparation of xanthines by the reduction of the chloroxanthines thus obtained, whereby a preceding or subsequent further alkylisation is allowed. Also for the new compound 3 methyl chloroxanthine,

$$C_6H_5N_4O_2Cl + 2H_2O,$$

a yellow crystalline body, insoluble in most solvents, but dissolved by dilute alkalis.—J. O. B.

*Vanillin, Impts. relating to the Employment and Administration of.* John Clayton Mewburn, London. From the Chemische Fabrik von Heyden, Dresden, Saxony. Eng. Pat. 4280, April 9, 1898.

The claim is for the preparation of solid preparations of various forms and shapes composed of an indifferent powder, flavoured with any proportion of vanillin, so that any determined number may be taken, as equivalent to the amount of vanillin contained in a vanilla pod. Also for the production of a tablet, pill or the like containing a definite amount of vanillin mixed with an indifferent powder such as sugar or flour.—J. O. B.

*New Pharmaceutical Compounds [Hexamethylene Tetramine, and Tannin], The Manufacture or Production of.* Henry E. Newton, London. From the Farbenfabr. vormals Friedr. Bayer and Co., Elberfield, Germany. Eng. Pat. No. 10,790, April 9, 1898.

The new condensation products described, contain respectively three and six molecules of tannin, combined with one of hexamethylene-tetramine. They may be obtained by precipitating concentrated aqueous solutions of the constituents in molecular proportions. The patent claims the preparation of these bodies and of mixtures of them; also the resulting products of the action of tannin, or substances containing tannin, on mixtures of ammonia and formaldehyde. They form tasteless powders which are powerful astringents.—J. O. B.

*Nicotine and Kindred Alkaloids, Salts of: an Improved Method of Obtaining, in a Concentrated State from Vegetable Substances which Contain them.* John Uri Lloyd, Cincinnati, U.S.A. Eng. Pat. 1989, April 23, 1898.

The process consists in the distillation of the alkaloids by means of a specially constructed apparatus in which they are by igniting at the bottom, a column of the dried herb, such as tobacco, in a closed chamber, so that the alkaloids are set free in advance of combustion and are aspirated through the mass of unburned material into acid solutions in suitable reservoirs. The alkaloidal salts, such as sulphates, are then purified from the ammonium sulphate which accompanies them, by percolating the crystalline mass with alcohol. A figure and description of the apparatus employed is given.
—J. O. B.

*Acetophenonphenetidide, Impts. in the Production of.* Friedrich Valentiner, Leipsic Plagwitz, Germany. Eng. Pat. 5156, April 23, 1898.

The process consists of heating together molecular proportions of acetophenone and of p-phenetidine *in vacuo*, to the temperature of combination, and then distilling off *in vacuo* at 210°—212° C. the acetophenone-phenetidide formed. By this means, combination is effected in 4—5 hours, and the product, amounting to from 60 to 90 per cent. of the theoretical yield, is obtained in a state of great purity.
—J. O. B.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Roburite as a Blasting Agent.* Mäckler. Thonind. Zeit. **22**, [35], 470—472.

Possessing the thrusting power of powder and nearly equalling dynamite in explosive force, it is stated that roburite forms a particularly useful blasting agent wherever the material to be dislodged is required in large blocks, such as is the case in limestone or chalk quarries and clay pits.

A detonating capsule, containing $1\frac{1}{2}$—2 grms. of mercury fulminate, is needed to explode the roburite cartridge. The latter is stemmed with clay (noburite being lighter than water), except in deep vertical holes, where a considerable pressure of water can be obtained, in which event a metal cartridge is used. Roth's safety igniter, in which concentrated sulphuric acid acts on sugar and potassium chlorate, is preferably employed for igniting the fuze; or the modern electric detonators may be used to explode the fulminate capsule.—C. S.

### PATENTS.

*Firework, An Improved; and Process of Making the Same.* W. Weiffenbach, Stuttgart, Germany. Eng. Pat. 7579, March 29, 1898.

A firework giving a non-dangerous spray of blinding white light is produced by mixing together 12 parts of aluminium filings, 12 parts of barium nitrate, 12 parts of saltpetre, 2 parts of yellow dextrin, 2 parts of sulphur, 5 parts of gum arabic, and filling the mass, dry, into tubes, or the mass may be made into a paste with water and spread on any suitable articles.—W. M.

*Explosives, An Improved Process for Increasing the Resistance of; to Atmospheric Influences and for Increasing their Explosive Power.* L. Muller, Berlin, Eng Pat. 8044, April 4, 1898.

The improvement consists in subjecting ammonium nitrate explosives, in which the melting point of the carbon compound is at least 40 C., to hydraulic pressure, and then disintegrating and granulating the explosive.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### *APPARATUS, ETC.*

*Analytical Balance with Rock Crystal Pans, Disturbances in.* K. Farnsteiner. Zeits. Unters. Nahr. und Genussmittel, 1898, [5], 315—316.

Erratic movements of the pointer were observed in working a very accurate balance with pans of rock crystal, so that it was often difficult, or impossible, to obtain an exact weighing. These disturbances were traced to the electrification of the crystal by friction when cleaning it. A counterpoised flask, after being wiped and replaced on the balance, showed an increased weight. This phenomenon was never observed with metal pans.—L. J. de W.

### *INORGANIC CHEMISTRY—QUALITATIVE.*

*Chili Nitre, Perchlorates in; Microchemical Detection of.* M. van Breukeleveen. Rec. trav. chim. Pays-Bas., **17**, 94–95. Chem. Centr. 1898, **1**, [16], 960.

Behrens' test for the detection of perchlorates in Chili saltpetre consists in adding to the solution of the latter a little rubidium chloride, which in presence of perchlorates, forms rhombic crystals of rubidium perchlorate. These crystals may be detected under the microscope after the addition of permanganate solution, since by the action of this reagent, the crystals become strongly coloured, whilst the solution remains colourless. In this way, perchlorate present to the extent of 0·6 per cent. or more may be detected. For the detection of smaller quantities, the author recommends that 10 grms. of the sample be dissolved in 10 c.c. of warm water, 50 c.c. of 95 per cent. alcohol added, the mixture heated to boiling, and then allowed to cool for 1—2 hours. The greater part of the saltpetre is thus separated, and after filtering, the filtrate consists of a concentrated solution of the perchlorate, which may be tested for in the above-described manner.—A. S.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Potassium Pyrogallate, Absorption of Oxygen by.* Berthelot. Comptes Rend. 1898, **126**, [15], 1066—1072.

The relations which subsist between the proportion of oxygen absorbed, and the proportions of pyrogallic acid and potash used, were studied under varying conditions. The observations proved that the reaction whereon the fixation of the oxygen depends is uninfluenced by great variations in the degree of dilution of the absorbent, and that the absorption is the same throughout a wide range of temperature, *viz.*, from 10° to 62° C. When the proportion of potash per equivalent of pyrogallic acid ranges from one to three equivalents, the absorption is constant, but when it falls below one equivalent, the absorption is proportional to the weight of potash. The limit of the absorption is reached when three atoms of oxygen are absorbed, thus $C_6H_5KO_3 + O_3 = C_6H_5KO_6$, in which the last formula represents either the actual compound produced, or the products of its decomposition, among which an oxyquinone, $C_6H_4O_5$, has been isolated. The amount of carbonic oxide liberated during the absorption of the oxygen is negligeable when a distinct excess of potash is present, and the pyrogallic acid used would absorb, at the limit of its power of absorption, four to five times as much oxygen as the mixture under examination contains. The carbonic oxide produced, does not under these conditions exceed a thousandth part of the volume of oxygen absorbed. In practice, solution of pyrogallic acid concentrated to such a degree that it will absorb 90 times its volume of oxygen should be employed. A volume of this solution exceeding a twentieth of the gaseous mixture should be introduced to the latter, and followed by excess of potash either in saturated solution or in small pellets. Absorption of oxygen is complete within a few minutes.—J. A. B.

*Carbon Monoxide, Action of Reagents on; and Determination of, in the Air.* A. Gautier. Comptes Rend. **126**, [12], 871—875.

In a town like Paris, the amount of carbon monoxide formed by imperfect combustion of fuel amounts to about "8 litres per square metre of area of the town daily." The author has tried to determine the amount of carbon monoxide in gaseous mixtures of a dilution corresponding with that in the atmosphere of towns, by absorption with cuprous solution, or with ammoniacal silver nitrate, and by oxidation with permanganate or other reagents; none of these methods, however, have given satisfactory results. Of the various oxidising agents tried (permanganate, chromic acid, iodic acid and anhydride), iodic anhydride at 60° C. is the only one which promises to furnish a satisfactory method. As qualitative reagents for carbon monoxide in minute quantity, the author suggests chloride of gold, which is immediately reduced by it, and moist silver oxide, which absorbs it to form a compound regarded by the author as argentous carbonate, since sulphuric acid breaks it up into silver sulphate, metallic silver, and carbon dioxide.

—J. T. D.

*Carbon Monoxide, Estimation of small Quantities of; in Air and Normal Blood.* L. de Saint-Martin. Comptes Rend. 1898, **126**, [14], 1036—1039.

Gautier has stated that when the gas to be analysed only contains some $\frac{1}{1000}$ to $\frac{1}{10000}$ of carbon monoxide, the estimation of this gas by cuprous chloride, even with the modification of the author (Comptes Rend. **117**, 1006), is incorrect, since acetylene and other hydrocarbons are calculated as carbon monoxide when the gases removed by vacuum from the reagent are submitted to eudiometric analysis. The author points out that the analysis of the gas includes the measurement of the mixture before and after explosion, and after absorption of carbon dioxide. The difference of the first two readings corresponds to the contraction, which should be exactly one-half the volume of the carbon dioxide produced, as determined by the third reading.

In determining small quantities of carbon monoxide in normal blood, the author insists on the necessity of exhausting the liquid twice, the first time to eliminate all gases other than carbon monoxide, consisting of the greater portion of carbon dioxide, oxygen, nitrogen, traces of hydrogen, &c., then, after an interval, a second time in presence of tartaric acid to remove the carbon monoxide mixed with carbon dioxide and traces of nitrogen.—J. L. B.

*Copper, "Pure, for Analysis."* J. W. Westmoreland. Analyst, 1898, **23**, [265], 86.

The author gives some results obtained by him on testing samples of so-called pure copper foil obtained from practically every dealer in the United Kingdom. It was found that in extremely few instances was the copper really pure. One sample sold by a provincial dealer as "pure copper foil" gave the following extraordinary results on analysis: Copper, 94·42 per cent.; lead, 0·39 per cent.; zinc, 4·91 per cent.; iron, 0·26 per cent.; arsenic, traces. The electrolytic and iodide methods were used for determining the percentage of copper, and the results were confirmed by standardising thiosulphate solutions by means of the various samples, in conjunction with copper obtained by the electrolysis of a solution of pure copper sulphate, the results thus obtained agreeing within the limits of experimental error.—A. S.

*Lead Salts, Action of Alkaline Stannous Chloride on.* L. Vanino and F. Treubert. Ber. **31**, [8], 1118—1119.

Metallic lead can be completely precipitated from solutions of lead salts by stannous chloride and alkali. To a solution of lead nitrate there is added a sufficiency of stannous chloride and a large excess of 10 per cent. caustic soda solution, and the mixture is heated to boiling. The precipitate is then clotted by shaking or stirring, and allowed to settle. The liquid is decanted off, and the precipitate washed twice with 5 per cent. sulphuric acid, pressed with a rod into cohering masses, brought on to a filter, washed with water and alcohol, dried at 105° C., and weighed. Five experiments with lead nitrate gave figures varying from 62·26 to 62·70 per cent., the average being 62·45 per cent., against 62·51 per cent. calculated.

—J. T. D.

*Copper from Mercury and Copper from Arsenic, Electrolytic Separation of.* N. Revay. Zeits. f. Elektrochem, 1898, **4**, 329-333. (Compare, this Journal, 1898, 184—185.)

### I. *Separation of Mercury from Copper.*

A. *Deposition in Acid Solution.*—Although Freudenburg is stated to have separated mercury from copper in acid solution, the author was unable to obtain a satisfactory deposition. The electrolysis was conducted with all due precautions and the conditions were varied within wide limits but without success.

B. *Deposition from the Double Oxalate Solution.*—From what is already known regarding the electrolysis of oxalate solutions, it may be taken that this method is trustworthy.

C. *Deposition from Potassium Cyanide Solutions.*—This method has given good results in the hands of Smith and Freudenburg, but Smith and Fränkel could not effect a complete separation when the quantity of copper exceeded 26 per cent. This is due to the fact that the potential difference between mercury and a solution of its cyanide varies greatly with the concentration. According to Werner and Bolton the smaller the number of mercury ions present the greater is the opposing polarisation.

When the amount of copper present in the solution is increased, its point of decomposition gradually becomes lower and approaches that of mercury. Employing about 0·5—0·6 grm. of crystallised copper sulphate and quantities of mercuric chloride varying from 0·25—1·00 grm., together with 3 grms. of potassium cyanide, the author found the following percentage of mercury in the mercuric chloride:—(1), 73·60; (2), 73·75; (3), 73·73; and (4), 73·76; the theoretical percentage being 73·80. These results were obtained with an E.M.F. of 1·8—1·95 volt, and the mercury was free from all traces of copper.

### II. *Separation of Copper from Arsenic.*

In order to effect this separation it is necessary to arrange the conditions so that the copper will be deposited on the cathode and the arsenic on the anode. This is easily accomplished if the arsenic exist in solution as an anion (*e.g.*, in potassium arseniate, $K_3AsO_4$) and is not easily transformed into a compound in which it plays the part of a cathion. Acid, ammoniacal, or potassium cyanide solutions may be employed with an E.M.F. of about 1·6—1·8 volt. When the E.M.F. is too high, a portion of the arsenic is precipitated as a grey deposit on the cathode, probably due to the reduction of pentavalent to trivalent arsenic. This occurs most readily when the trivalent arsenic can form a salt with the acid ion, *e.g.*, in hydrochloric acid solution. It is less likely to take place in a nitric acid solution, and still less so in a sulphuric acid solution. For this reason it is preferable to work with a sulphuric acid solution. Cyanogen has only a slight tendency to form salts with arsenic, and consequently the separation may be equally well effected in a potassium cyanide solution. Fairly good results were also obtained with a blue ammoniacal solution.—J. S.

### *Cadmium, The Electrolytic Estimation of.* E. Rimbach. Zeits. Anal. Chem. **37**, [5], 284—290.

The estimation is best carried out by diluting the solution containing the cadmium to 150—200 c.c., adding an excess of potassium cyanide ("pure," 98 per cent.), and electrolysing by passing through the solution a current of electricity of 0·02—0·01 ampère and 3·0—3·3 volt. The operation should be allowed to proceed for 12 hours, when the cadmium is completely precipitated, and, after washing and drying, is ready to be weighed.

This method was found to answer well for the analysis of alloys consisting of aluminium, cadmium, and tin, with small quantities of copper. After dissolving the alloy in nitric acid, it is advisable to evaporate down the solution of the nitrates with the addition of hydrochloric acid, before proceeding with the electrolysis.

The separation of cadmium from magnesium is possible by the above process, provided that the latter metal is present in not too great a proportion, and that a quantity of ammonium chloride is added to the solution, to prevent the precipitation of magnesium.—W. P. S.

### *Cadmium and Iron, The Electrolytic Separation of.* W. Stortenbecker. Zeits. f. Electrochem. **4**, [17], 409—410.

Smith has described a method for separating these two metals in slightly acid solution. The cyanide solution, however, yields much better deposits of cadmium, and the author has made experiments in order to see if the solution could not be used to effect the separation of cadmium and iron.

When excess of potassium cyanide is added to solutions of the ferro salts of iron, soluble potassium ferro-cyanide is produced. With ferric salt solutions, ferric hydrate is precipitated, and only a small proportion of the iron goes into solution in the potassium cyanide. The author bases his method upon the non-electrolytic decomposition of the soluble potassium ferrocyanide salt.

The mixed cadmium and iron salts are dissolved in about 100 c.c. water, which have been slightly acidified beforehand with a few drops of dilute sulphuric acid; 2—3 grms. of pure potassium cyanide are added, and the mixture is heated until clear. If this demands unusual time, a few drops of potassium hydrate solution are added. The solution is then diluted to between 200 and 250 c.c., and when cold is electrolysed with a current of from 0·05—0·10 ampères $N.\text{-}D._{100}$ (0·47—3·8 ampères per square foot). The cadmium deposit obtained is very satisfactory. When a small amount of ferric salts is present, the ferric hydrate floating in the electrolyte, may be neglected; when larger amounts are present, it is necessary, before adding the potassium cyanide, to first reduce the ferric iron to the ferrous state by means of sulphurous acid. The addition of sodium sulphite to the slightly acid solution, and the application of heat, is the best method by which to effect this.

The author employed this method in making some tests of pure cadmium sulphate to which varying amounts of ferrous and ferric salts had been added. The results were in every case satisfactory.—J. B. C. K.

### *Uranium, Cadmium, &c., Electrolytic Determinations of.* E. F. Smith and D. L. Wallace. J. Amer. Chem. Soc. 1898, **20**, [4] 279-281.

Heidenreich having disputed the accuracy of the authors' electrolytic process for determining uranium, the experiments were repeated, and were found to give perfect results. 10 c.c. of an uranium acetate solution, containing 0·1185 grm. of $U_3O_8$, was mixed with ½ c.c. of strong acetic acid and diluted to 40 c.c. with water; it was then electrolysed with a current of $N.D._{40}$ = 0·18 ampères and 3 volts, at a temperature of 70 C. The uranium was completely precipitated as the hydrated protosesquioxide in 6 hrs., and was ignited and weighed as $U_3O_8$.

Heidenreich failed also with the authors' method for cadmium. But perfectly accurate results were obtained when 10 c.c. of cadmium sulphate solution, containing 0·1656 grm. Cd., were added to an excess of $Na_2HPO_4$ (1·0358 sp. gr.), and 1·5 c.c. of phosphoric acid (1·347 sp. gr.), and the liquid, diluted to 100 c.c., was electrolysed at 50° C. with a current of $N.D._{100}$ = 0·06 ampères and 3 volts, which was increased after 4 hrs. to $N.D._{100}$ = 0·35 ampère and 7 volts. The distance between the electrodes was 1·25 ins. throughout. After 7 hours the acid liquid was siphoned out without interrupting the current, and the bright adherent metal, free from all trace of sponginess, was washed with hot water, dried and weighed.—W. G. M.

### *Antimony and Tin in Antifriction Metal and Similar Alloys, Separation of.* Prost. Bull. Assoc. Belge des Chim. **11**, [11], 337-338.

One gram of the alloy is treated with nitric acid (sp. gr. 1·3) and the solution evaporated to dryness, the mixture of tin and antimony hydroxides, with traces of copper and lead, then collected, being fused with "liver of sulphur." The mass is taken up with water, whilst the residue (lead sulphide and copper sulphide) is dissolved in nitric acid; the solution thus obtained is united with the primary liquid and used for the estimation of these two metals in the ordinary manner.

The sulpho-alkaline solution is acidified by hydrochloric acid, and the precipitated sulphides are redissolved in concentrated HCl, a little potassium chlorate being added to ensure the tin being in the stannic condition. On adding potassium hydroxide until a permanent precipitate is produced, then heating with 5—10 grms. of oxalic acid and treating with hydrogen sulphide, antimony (alone) is thrown down as sulphide.

The filtrate is neutralised with ammonia, and treated with sufficient ammonium sulphide to form sulpho-stannate. An excess of acetic acid being then added, the liquid is heated on the water bath until quite clear, and the stannic sulphide precipitate is estimated as usual, after washing with hot ammonium acetate.

When, as is generally the case, tin predominates in the alloy, it is advisable to make up the filtrate from the antimony precipitate to a definite volume and take an aliquot part for the estimation of tin.—C. S.

### *Steel, Carbon in, Determination of by the Moist Combustion Method.* G. Auchy. J. Amer. Chem. Soc. 1898, **20**, [4], 243—253.

Although in the usual dry combustion determination of steel carbons, one set of potash bulbs suffices to absorb all the carbon dioxide, it fails to effect complete absorption in the case of moist combustions with sulphuric acid and potassium bichromate, partly, perhaps, because in the latter process the gas is given off all at once, and therefore less dilute, at the beginning of the operation. This view is confirmed by the observation that a similar incompleteness of absorption is found when the combustion is effected by the lead chromate method.

Twenty-five experiments with two sets of bulbs showed a varying gain in the second set of from 0 to 0·7 (average 0·105) per cent. of carbon, which would have been lost if only one set had been employed. But this was with potash solution with a sp. gr. of 1·27. A stronger (sp. gr. 1·4) solution gave better results. 54 experiments showed a gain in the second bulbs of from 0 to 0·273 (average only 0·048) per cent. of carbon. It is also noteworthy that with the weaker solution only 4 out of 25 analyses gave complete absorption, while with the stronger, 27 out of 54 did so. The material operated upon contained in most cases between 0·5 and 2 per cent. of C. In the case of combustions with bichromate, about one-third of the carbon present passed through the first bulbs unabsorbed.

Another cause of error is that the drying apparatus may allow moisture to escape. It is probable that the slight gain in weight in the second bulbs when the stronger potash solution is used is, in most instances, due to moisture which has passed the drying train. After passing for half an hour through Liebig bulbs with strong sulphuric acid, then through a calcium chloride U-tube, and afterwards through more sulphuric acid bulbs, the gases in a blank experiment gave an absorption equal to 0·015—0·076 (average 0·05) per cent. of carbon in the first potash bulbs and guard-tubes, and of 0·005—0·06 (average 0·023) in the second set, in 10 experiments. So also the calcium chloride guard-tube attached to the potash bulb, allows small but varying quantities of moisture to escape, usually ranging from 0 to the equivalent of 0·08 per cent. of C., according to the freshness of the calcium chloride. The author, therefore, deems it best to use a sulphuric acid or other drying apparatus after the calcium chloride tube, seeing that a correction should be made in any case. The necessity for re-drying the calcium chloride is shown by the fact that a tube filled with the chloride taken from a half-emptied bottle may lose as much as 0·48 per cent. (in terms of carbon) when exposed to an aspirated current of dry air for two hours.

Further, with some samples of potash there will be a loss of weight (0·02—0·05 per cent. reckoned as carbon) during the analysis, unless air be drawn through them before use. And, lastly, there may be a gain in weight due probably to some chloro-chromic compound other than that absorbed by the "pyro" mixture, even after the gas has been passed through a purifying train, including cold water for HCl, ferrous sulphate for chlorine, Langley's pyro mixture for chlorine, oxides of chlorine, and chloro-chromic anhydride, and silver sulphate in strong sulphuric acid for HCl.

The moist combustion method must not, of course, be used for graphite determinations, as a portion of the carbon in this case escapes as monoxide.—W. G. M.

### *Manganese, Quantitative Estimation of; and Separation of Iron from Manganese, by Electrolysis.* Fr. Kaeppel. Zeits. für anorg. Chem. 16, 268. Chem. Zeit. Rep. 22, [14], 118.

The author has endeavoured to find a method of precipitating manganese as peroxide in a coagulated form, and to avoid the transformation of this to mangano-manganic oxide by igniting in a platinum vessel as the vessel is corroded in this operation.

The method employed was as follows:—The manganese salt was dissolved in about 150 c.c. of water and acetone added. The solution was kept at a constant temperature of 50°—55° C. and electrolysed by a current of 4—4·25 volts and 0·7—1·2 ampères. (Water was added to make up for that lost by evaporation.) 0·15—1·6 grms. of manganese peroxide was deposited in a black shining form, in 2—5½ hours according to the amount of acetone (1·5—10 grms.) added, and the amount of manganese present in the solution. The manganese peroxide was washed, without the current being stopped, dried at 150°—180° C. and weighed as $MnO_2$.

The electrolytic separation of iron and manganese devised by the author, is sufficiently accurate for technical purposes. In presence of a very large excess of sodium pyrophosphate and a few drops of phosphoric acid, a mixed solution of iron and manganese sulphates when electrolysed gives a deposit of metallic iron, and only very little manganese is precipitated in the form of $Mn_2O_3$ and $MnO_2$.

The method is as follows:—The mixed solution of the sulphates is stirred into a boiling solution of 12 grms. of sodium pyrophosphate, and after the solution has become quite clear, three or four drops of phosphoric acid are added. (Should a milkiness appear this is destroyed by addition of more sodium pyrophosphate.) The liquid, which should have a volume of 230—250 c.c. and a distinct alkaline reaction, is kept at a constant temperature of 35°—40° C. and electrolysed by a current of 1·8—2·5 ampères. The iron is completely precipitated in 8—9 hours. It is deposited on the platinum very firmly and after washing with absolute alcohol, whilst the current is still running, it is dried at not too high a temperature and weighed.—J. McC.

### *Cobalt from Nickel, Method for the Separation of.* Zeits. f. Elektrochem. 1898, 4, [22], 501—503.

If a glass tube be fused up at one end and dipped whilst still hot into a stream of water, it becomes permeated by an infinite number of narrow cracks. When such a tube is filled with acetone, and placed in an outer containing vessel also filled with the same liquid, and a difference of potential maintained by means of two electrodes, one of which is placed in the interior of the tube, whilst the other is disposed externally, then the acetone in the tube begins to rise or fall, according as to whether the internal electrode is negative or positive.

Some years ago it was noticed by Braun that, if solutions of certain metallic salts were substituted for the acetone, a deposition of metal, which he called *stenolysis*, occurred in the crack. By applying Helmholtz's theory of electrical double layers, it is shown that this deposition of metal must always take place, provided the difference of potential is sufficient to produce a displacement of the liquid as a whole. The trace of metal so deposited, however, only becomes visible when it continues to increase in weight. This is the case—

(*a.*) When the negative radicle does not attack (and re-dissolve) the precipitated metal, *e.g.*, in the case of all platinum salts.

(*b.*) When insoluble compounds are formed, and especially when peroxides are formed at the anode end of the minute metallic conductor.

(c.) When the solution contains subsalts, the negative ion of which can react on the remainder of the substance in solution to produce the persalts.

If a solution of silver nitrate is used, and the negative electrode is placed inside the tube, then the inner end of the fine metallic film in the crack becomes an anode, whilst the outer end becomes a cathode. Under these circumstances the deposition of silver continues to increase on the outside of the crack and, finally, thin leaves are formed, and fall to the bottom. Minute bubbles of oxygen are given off from the anode end, which is protected by a layer of insoluble peroxide.

When a solution of potassium silver cyanide is substituted for the silver nitrate, *stenolysis* does not take place, because of the continuous solution of the equivalent amount of silver. Similarly, a solution of copper sulphate does not show the phenomenon, whilst cuprous chloride does; the chlorine which is produced at the anode end, in this case, forms cupric chloride, with the excess of cuprous chloride, and does not dissolve the copper.

The salts of cobalt and nickel are particularly interesting; the former generally show *stenolysis*, whilst the latter do not. This is due to the fact that only the cobalt salts are capable of forming a protecting layer of electrolytic peroxide. The precipitate formed at the anode in a mixture of nickel and cobalt salts was found to be perfectly free from nickel.

If a solution of ordinary commercial nickel sulphate is placed in a platinum basin which acts as the anode, then, in a short time, the basin becomes covered with a dark film of peroxide. No precipitation occurs if solutions of nickel sulphate perfectly free from cobalt are employed. In order to separate all the cobalt from a solution, as peroxide, the precipitation of the metals nickel and cobalt on the cathode must be prevented by the addition of a more easily separated metal, *e.g.*, copper sulphate, and the solutions must be maintained neutral during the electrolysis.

The author considers that this forms the basis of a technical method for preparing pure cobalt, and promises further quantitative details.

The qualitative detection of the smallest traces of cobalt in nickel solutions may be accomplished by introducing two platinum wire electrodes into the warmed solution, and observing if a dark film forms on the anode.—J. S.

### *Magnesium-Ammonium Phosphate, The Ignition of.* M. Schmoeger. Zeits Anal. Chem. 37, [5], 308—310.

The following method for burning precipitates of magnesium-ammonium phosphate was found to be convenient. After the precipitate is completely dry, it is shaken out of the filter-paper into a covered platinum crucible, the filter-paper being then folded and also placed in the crucible. The latter may now be heated strongly in a Bunsen flame, until the paper is burnt. The crucible is finally heated over the blow-pipe. Figures are given in the original paper to show that the results obtained by this rapid burning agree very closely with those yielded by the older process of burning the filter and precipitate separately. —W. P. S.

### *Fuming Sulphuric Acid and Sulphuric Anhydride, Estimation of.* R. Rosenlecher. Zeits. Anal. Chem., 37, [4], 209—217

In order to weigh the acid or the anhydride, the author makes use of an apparatus which is made by blowing a small balb (8—10 mm. diameter) in the middle of the capillary portion of a drawn-out tube, and then whilst the glass is soft, inclining the tubes on either side of the bulb so as to make an angle of 135—140 with each other. The one limb is then cut to a length of 4—5 cm., the other to 5—6 cm., and the orifice of the latter is reduced to ¼—½ mm. The acid is sucked into the bulb by immersing the end of the longer limb in the sample and then cautiously withdrawing the air by sucking through the other limb, care being taken to avoid drawing any acid fumes into latter. The acid in the capillary tube is made to run down into the bulb by gently tapping. During the various operations the apparatus should never be touched directly with the warm hand but always with several thicknesses of paper interposed. After weighing, the acid is run into 20—30 c.c. of water containing some indicator, preferably a solution of paranitrophenol, contained in a flask of 250—300 c.c. capacity, furnished with a well fitting stopper. The flask is inclined so as to be nearly horizontal and the bulb then pushed into the neck as far as possible; it is then quickly released, the stopper inserted, and the flask at once further inclined, so that the water wets the inside of the stopper. If the operation have been carefully performed, the colour of the indicator will not yet have changed. The bulb is then broken by shaking sharply and the flask and contents gently shaken until all the white fumes are absorbed. The flask is then opened and the acid titrated with alkali.—E. W. W.

### *Sulphurous and Thiosulphuric Acids, The Detection and Estimation of.* W. Autenrieth and A. Windaus. Zeits. Anal. Chem., 37, [5], 290—300.

For the detection of sulphurous, thiosulphuric, and sulphuric acid, and also of sulphuretted hydrogen, when present together in solution as salts of the alkalis, the following scheme or method may be used.—

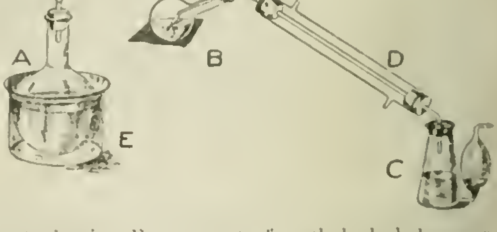
The solution is treated with an excess of zinc sulphate, and the precipitate of zinc sulphide is filtered off. The filtrate from this contains the sulphite, thiosulphate, and sulphate. Strontium nitrate is now added, when strontium sulphite and sulphate are precipitated and strontium thiosulphate remains in solution. After filtering off the two former, hydrochloric acid is added to the filtrate, and gives a white precipitate of sulphur by the decomposition of the thiosulphate. The sulphite and sulphate are washed and treated with dilute hydrochloric acid; the strontium sulphate remains insoluble, whilst strontium sulphite is decomposed, giving off sulphur dioxide, which may be tested for by oxidizing with iodine. As the addition of zinc sulphate of course introduces sulphuric acid, this acid must be looked for in a separate portion of the original solution. To this end a quantity of the solution is acidulated with dilute hydrochloric acid, the liberated sulphur is filtered off and the filtrate tested with barium chloride.

The quantitative estimation of sulphurous and thiosulphuric acids when occurring together is carried out as follows:—

A portion of the solution is titrated with a standard solution of iodine, the two acids being thus determined together. Another quantity of the original solution is placed in a 100 c.c. flask, treated with an excess of strontium nitrate, diluted to the mark, and well shaken. After standing over night, the contents of the flask are filtered through a dry filter, and the thiosulphate in the filtrate is titrated with standard solution of iodine. A small correction is necessary, as strontium sulphite is soluble in the proportion 1-30,000.

If sulphuric acid be present, a fresh portion of the solution is heated with hydrochloric acid in a current of carbon dioxide until the sulphurous acid is driven off, after which the sulphuric acid in the solution is precipitated with barium chloride. Should the solution contain soluble sulphides, it must be digested with moist cadmium carbonate, and filtered before proceeding with the analysis. The sulphur in the cadmium sulphide may be determined in the usual way by oxidising with bromine in hydrochloric acid solution, and precipitating the sulphuric acid formed with barium chloride. —W. P. S.

### *Boric Acid Determination.* T. S. Gladding. J. Amer. Chem. Soc. 1898, 20, [4], 288—289.



One grm. of the substance is washed into the 150-c.c. flask, B, with a little 95 per cent. methyl alcohol and 5 c.c. of syrupy (85 per cent.) phosphoric acid are added. The flask, A, is then two-thirds filled with 95 per cent. methyl alcohol, placed in the water-bath, E, and connected up to B, which is placed in communication through the condenser D, with the receiver C. On heating E, a current of methyl alcohol vapour is conducted through a long tube to the bottom of the liquid in B, and carries over the boric acid. Heat is applied under B and is so regulated that the volume of the liquid remains between 15 and 25 c.c. The distillation is thus continued for half-an-hour, by which time the volume of the distillate should be about 100 c.c. A mixture of 40 c.c. of glycerin and 100 c.c. of water is now carefully neutralised (with phenolphthalein as indicator) and is added to the distillate, which is then titrated with standard soda. The distillation must be continued until no more acid comes over, but is usually complete in 30 minutes. A blank experiment should be made with all the reagents, and correction should be applied if necessary. Results are accurate. Crystallised boric acid, distilled without addition of phosphoric or other acid, gave all the boric acid present, and a similar distillation of borax yielded 19·5 out of a total of 36·65 per cent. of boric acid. This behaviour of borax may be useful in some analyses. A gentle suction

may with advantage be kept up at C by means of an aspirator.
—W. G. M.

*Phosphoric Acid, Estimation of, by Titration.* A. Hebebrand. Zeits. Anal. Chem. **37**, 217–223.

To estimate the amount of phosphoric acid, the precipitate of ammonium magnesium phosphate after washing with ammonia, is washed with 96 per cent. alcohol to remove the free ammonia, and then dissolved in a standard solution of hydrochloric acid ($\frac{1}{5}$ normal). The excess of acid is then titrated with alkali, carminic acid (acidum carminicum, Merck) being used as an indicator. The change of colour from light yellowish-brown to violet is said to be sharp and readily distinguished after a little practice. Attempts to determine the phosphoric acid by heating the ammonium magnesium phosphate with dilute sodium hydrate solution and estimating the liberated ammonia, gave too high results, probably because the precipitate contained diammonium magnesium phosphate (compare Neubauer, this Journal, 1894, 1092–1093).—E. W. W.

## ORGANIC CHEMISTRY.—QUALITATIVE.

*β-Naphthol, Detection of α-Naphthol in.* A. Dubosc. Bull. de la Soc. Ind. de Rouen, 97, 434; through Färber Zeit. **9**, [4], 60.

Elbe (Oesterreichs. Wollen- und Leinen Ind. 1897, 1330) has recently made a communication with regard to the behaviour of β-naphthol contaminated by α-naphthol as a ground for the production of insoluble azo-colours on the fibre. He found that the production of the brown colour which appears on cotton prepared with β-naphthol after storing some time in presence of light and air, is favoured by the presence of α-naphthol. The presence of 1 per cent. of α-naphthol in the β-naphthol exerts but little influence, but the presence of 3 per cent. renders the use of the impure product impossible; if α-naphthol be present to the extent of 10 per cent., the goods acquire a greyish-brown colour during the process of impregnation. Cotton prepared with pure β-napthol, then dried at, or a little over, a temperature of 45° C., and cooled in a chamber protected from the light, remained almost unchanged for 48 hours; the first distinct appearance of the brown colour occurred after 60 hours; but the material was never rendered useless. An addition of tartar emetic, or of Lauber and Caberti's preparation partly prevents the rapid browning, but is not always applicable, and is without effect in the presence of a high percentage of α-naphthol.

The author, in view of the above article, gives the following method which permits of the detection of 1 part of α-naphthol in 100 parts of β-naphthol. The method depends on the behaviour of the naphthols towards a solution of sodium hypobromite (30 c.c. NaOH of 36° B., 100 c.c. of water, and 5 c.c. of bromine). 10 c.c. of the saturated naphthol solution, with 2 drops of the hypobromite, gives in the case of the α-compound, a dirty violet coloration that remains perceptible even on dilution with 9 vols. of water, whilst with the β-compound, a yellow coloration, changing gradually to green and again to yellow, is produced; if, however, an equal volume of water be added to the β-naphthol solution, the yellow colour first obtained, disappears immediately on shaking. In testing commercial β-naphthol, a saturated solution of the sample is diluted with an equal volume of water, and two drops of the above sodium hypobromite solution added. If the sample be pure, a yellow colour is formed which quickly disappears on shaking, but if α-naphthol be present, a violet to reddish-violet coloration is produced.—A. S.

*Codliver Oil, Testing.* A. Gawalowski. Chem. Rev. Fett- u. Harz-Ind. **5**, [4], 67—68.

For the detection of fish and seal oil in cod-liver oil, P. Tozelli (Deutsch-Amerikan. Apoth. Zeit.) recommends the treatment of separate quantities (each 20 parts) of the substance with, (A) 1 part of concentrated sulphuric acid; (B) 10 parts of the same reagent; (C) 1 part of concentrated nitric acid; and (D) 15 parts of litmus cake. The results are said to come out as follows:—

| I. Pure Cod-liver Oil. | II. Adulterated with much Fish Oil. | III. Adulterated with much Seal Oil. |
|---|---|---|
| A. Violet coloration changing to reddish-brown after a time. | Reddish-brown coloration. | Cochineal red coloration. |
| B. Consistency of soap after 2—3 hours. | Consistency of vaseline after 2 hours. | Consistency of soft extract after 1 hour. |
| C. Rose-red to orange red coloration. | Reddish coloration, changing to brown. | No coloration, but consistency changes as in B. |
| D. Remains blue for at least an hour. | Changes to red within the hour. | Consistency as in B. and C. |

The results A, B, and C do not, however, agree with those obtained by the author and other workers, and are not applicable for every kind of liver oil. He also finds that the litmus test is unreliable, and consequently maintains that Tozelli's proposals are unsuitable for the object in view.—C. S.

*Cotton-Seed Oil, New Method for the Detection of, in Olive Oil.* M. Tortelli and R. Ruggeri. Chem. Zeit. Rep. 1898, **22**, [12], 101.

The method is a modification of Becchi's test. 5 grms. of the oil are saponified with 30 c.c. of a solution of 60 grms. of potassium hydroxide in a litre of 90 per cent. alcohol, the soap solution exactly neutralised with 10 per cent. acetic acid, and poured in a thin stream into a hot solution of lead acetate (50 c.c. of a 10 per cent. solution with 250 c.c. of water). The resulting lead soap is well washed, dried on filter paper, and warmed for about 20 minutes with anhydrous ether under a reflux condenser. When cold the ethereal solution of the lead salts of the so-called liquid fatty acids, is filtered, and the fatty acids liberated by shaking the solution in a separating funnel with 60 c.c. of 10 per cent. hydrochloric acid. The aqueous layer is run off and the treatment with acid repeated. After being washed with water, the ethereal layer is decanted, the ether evaporated, and the residue mixed with 10 c.c. of 90 per cent. alcohol and 1 c.c. of a 5 per cent. aqueous solution of silver nitrate. The liquid is poured into a test tube and warmed in water to from 70° to 80° C. In the case of pure olive oil, the liquid remains unaltered after 30 minutes, but it is stated that when as little as 1 per cent. of cotton-seed oil is present, a reduction is plainly visible within two minutes.—C. A. M.

*Oil of Turpentine, The "Custom House" Technical Examination of; and a Simple Method for Distinguishing Pure Oil of Turpentine from "Patent" Oil of Turpentine, or Mixtures of both.* F. Evers. Zeits. für öffentl. Chem. **4**, 211—215; Chem. Centr. 1898, **1**, [14], 865.

The author regards as absolutely useless the official method for the examination of oil of turpentine, which depends on the increase of temperature brought about by shaking together equal volumes of the sample and of fuming hydrochloric acid (sp. gr. 1·19). It is stated that the increase of temperature with hydrochloric acid is greater, the older and more resinified is the oil, although the resinous substances (colophony and turpentine) closely related to oil of turpentine, have little influence on the reaction. A sample of oil of turpentine after treatment with sodium, showed only an increase of 2° to 5° C. as against 12° formerly. It is also stated that two samples of "patent oil of turpentine" showed an increase of 0° and 1·5° C. respectively, whilst mixtures of these with old resinified oil of turpentine corresponded perfectly to the official test (increase of 25° C.).

The author's new method depends on the fact that patent oil of turpentine is indifferent towards bromine water, whilst oil of turpentine combines with not inconsiderable quantities of bromine. 1 c.c. of the sample of oil of turpentine at

15° C. is treated in a stoppered 100 c.c. graduated glass cylinder with 10 c.c. of bromine water (saturated at 15° C.) and the mixture shaken for half a minute. If no decolorisation ensue, the sample consists of patent oil of turpentine. If the mixture become decolorised, 15 c.c. more of the bromine water are poured in and the mixture shaken for a minute. If a further decolorisation take place, the sample consists of pure oil of turpentine. If, however, the mixture is not completely decolorised, 10 c.c. of the original sample are distilled to one third, and 1 c.c. of the distillate tested with the bromine water. If no decolorisation take place, the sample consists of a mixture of oil of turpentine and "patent" oil of turpentine.—A. S.

### *Oil Paints, Testing.* H. Loesner. Chem. Rev. Fett- u. Harz-Ind. **5**, [4], 64—65.

Replying to criticisms of his steam test (Chem. Rev. 1897, 329; this Journal, 1898, 359) put forward by Treumann, the author asserts that some such test for the durability of paint is indispensable in the absence of any definite chemical knowledge on the physical properties of paint mixtures, and he recommends the adoption of mechanical or mechanico-chemical tests appropriate to the uses for which paints are destined, *e.g.*, a flexibility test for varnishes, &c., intended for use on sheet metal.

The steam test has shown that no paint composed of linseed varnish and an inorganic pigment is absolutely impermeable to gases; that pure linseed varnish blisters under the influence of heat; that the addition of fat increases the protective power of linseed varnish though retarding its drying; that minium is the most durable pigment; that various varnishes withstand steaming better when mixed with a pigment; that the addition of insoluble and chemically inert pigments is useless; and that heating to 120° C. reduces the penetrability of oil paints.

All these points agree with practical experience, and the steam test consequently possesses a definite technical value.
—C. S.

### *Farinaceous Materials, Detection of Wood Sawdust in.* G. A. Le Roy. Comptes Rend. 1898, **126**, [14], 1017—1018.

The adulteration of flour of inferior quality, meal, &c., by wood sawdust is difficult to detect since such substances generally contain the cellulosic débris derived from the grain. The author moistens the suspected material with an alcoholic solution of phloroglucinol, acidified with phosphoric acid. On gently warming, any particles of sawdust are coloured an intense reddish carmine. The coloration produced on the cellulosic matter is at first hardly noticeable. Phloroglucinol hydrochloride behaves in a similar manner, but the difference in colour between the particles of sawdust and cellulose is less decided.—J. L. B.

### *Unboiled Milk, Distinguishing Boiled from.* H. Leffmann. Analyst, 1898, **23**, [265], 85—86.

It has been previously stated (Bull. Soc. Pharm. Bordeaux, March, 1897) that a freshly prepared dilute (1·4) solution of diamidobenzene gives a deep blue colour with raw milk in the presence of a few drops of hydrogen peroxide solution. The author found that this colour is produced to a marked degree when the milk has been heated to 76·5° C., but not at all after heating to 82° C., and he considers it possible thus to distinguish between pasteurised and sterilised milk. Whole milk, ordinary skimmed, and separator-skimmed milk exhibit substantially the same colour. It was also noted that the photographic developer amidol gives a colour reaction with raw milk, but not eikonogen, and various amido-, hydroxy-, and carboxy-derivatives of benzene and naphthalene. Sour raw milk still shows the reaction, and even when more advanced decomposition has set in, but in the latter case it is less striking.

The author carried out some experiments with a view to determining which ingredient in the milk causes the reaction. Various commercial enzymes, malt-diastase, taka-diastase, peptenzyne, pancreatic extract, papaw-ferment, pine-apple ferment, and rennet ferment and solutions of blood-and-egg albumin did not give the reaction. A sample of raw milk treated with excess of magnesium sulphate and then filtered gave a deep blue colour with the 1.4 diamidobenzene, and a red tint with amidol, but no effect with other reagents.

The diamidobenzene solution must be freshly prepared, as, after standing for an hour or more, it gives a slight blue colour with both boiled and raw milk, without the addition of hydrogen peroxide.—A. S.

### *Annatto in Milk, Detection of.* A. Leys. Journ. Pharm. Chim. 1898, **7**, [6], 286—289.

Fifty c.c. of milk are shaken out in a separator with 100 c.c. of a mixture of alcohol (93 per cent.), 240 c.c.; ether, 320 c.c.; water, 20 c.c.; and solution of ammonia (sp.gr. 0·920) 8 c.c. After standing for 20 minutes, the lower watery layer is run off into another separator, and half its volume of 10 per cent. sodium sulphate solution is gradually added to it: the casein separates in white flocculent masses, which rise to the surface and allows the greater part of the liquid to be drawn off clear, and strained through a fine wire gauze. The liquid is then placed in a series of test tubes so as to two-thirds fill them, and each tube is strongly shaken with amylic alcohol. The tubes are then plunged in a beaker of cold water, which is gradually heated on the water bath to 80° C., when complete separation of the alcohol will generally have taken place. The alcoholic solution is collected and evaporated; the residue thus obtained is treated with warm water faintly alkaline with ammonia, and containing a trace of alcohol. A strip of bleached cotton is immersed in the liquid, which is heated until nearly dry. The yellow dyed cotton is now gently washed, and then plunged into a solution of citric acid. If annatto be present, a rose tint is instantly developed. A slight yellow tint is imparted to cotton by normal milk by this process, but no change of colour takes place with this in the acid bath, nor do the other yellow dyes, such as saffron or turmeric, give the rose coloration with the acid. The rose tint thus developed is characteristic of annatto.—J. O. B.

### *Furfural in Beer.* W. Windisch. Wochenschr. für Brau. 1898, 189—191.

*See under* XVII. *page* 593.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Coal Analysis, Preliminary Report of the Committee of the American Chemical Society on.* J. Amer. Chem. Soc. 1898, **20**, [4], 281—285.

The committee, consisting of W. F. Hillebrand, C. B. Dudley, and W. A. Noyes, publish outlines of the suggested standard method of analysis, and invite individual (private) criticism and discussion.

*Sampling.*—5 lb. of coal, carefully selected with a view to a fair average, are broken down, quartered, and fine-crushed at once, and as rapidly as possible, and stored in a tightly closed bottle.

*Moisture.*—1 grm. of coal is heated in an open porcelain or platinum crucible at 104°—107° C. for one hour, best in a double-walled bath containing pure toluene. It is then cooled in a desiccator and weighed covered. 20 hours over sulphuric acid *in vacuo*, however, gave slightly higher, and probably more correct, results. It is found that a small quantity of volatile matter other than water is lost at 105° C., but as it rarely exceeds 0·1 per cent. with bituminous coals, the loss may be neglected for most practical purposes.

*Volatile Combustible Matter.*—1 grm. of the undried coal is placed in a tightly closed platinum crucible weighing 20—30 grms., which is supported on a platinum triangle with its bottom 6—8 cm. above the top of a Bunsen flame, 20—25 cm. high when burning free. It is thus heated for 7 minutes in a place free from draughts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon. To estimate the volatile combustible matter, deduct the percentage of moisture from the total loss of weight. The determination is largely arbitrary, and to obtain uniformity of results among chemists it is desirable to make the method as simple as possible. The chief objection to this method is the danger of mechanical loss at the outset, but is is believed that the error is less than it would be were the application of the heat left to the judgment of the operator.

*Ash.*—The portion of coal used for the determination of moisture is burned, at first over a very low flame, with the crucible open and inclined, until free from carbon. The result is thus more rapidly attained than if the denser residue from the coke determination were used. Information is sought as to whether carbonates are ever present in such quantity that heating over a blast would be inadmissible. The sulphur in pyrites is replaced by only three-eighths of its weight of oxygen, but, although the error from this source may be very considerable, the committee does not recommend the application of a correction.

*Fixed Carbon.*—Subtract the percentage of ash from that of the coke.

*Sulphur.*—No method is given, but information is asked for from others.

*Heating Effect.*—The calorific power should be stated in centigrade calories per kilo., or in British thermal units per lb., and not as evaporative effect. The results of 21 analyses and determinations of calorific power (with the bomb calorimeter) of Indiana and Pittsburg bituminous coals have shown that the following rule gives numbers accurate to within 2 per cent.:—"Subtract from 100 the percentages of moisture and ash and one half the percentage of sulphur, and mutiply the remainder by 80·7. The product will be the heating effect of the coal burned to vapour of water, expressed in calories."

Communications on this subject should be addressed to Prof. W. A. Noyes, Rose Polytechnic Institute, Terre Haute, Ind., U.S.A.—W. G. M.

### *Ozokerite, Estimation of Cerasin in.* E. von Boyen. Zeits. angew. Chem. 1898, **17**, 383.

The following method is recommended for the estimation of cerasin in ozokerite:—5 grms. of ozokerite are placed in a porcelain basin and melted; 0·9 grm. of concentrated sulphuric acid is added, and the mixture is heated to 200° C. until no more sulphur dioxide is given off. The acid is then neutralised, and the mass is finally mixed with about 3 grms. of sawdust previously washed with petroleum spirit (benzine) and extracted with warm petroleum spirit. The extract is evaporated in a weighed porcelain basin, heated to 200° and weighed, the result being the amount of cerasin contained in the 5 grms. of ozokerite.—W. P. S.

### *Paraffin, Commercial; Estimation of the Solidifying Point of.* L. Singer. Chem. Rev. Fett- u. Harz-Ind. **5**, [4], 65—67.

The author agrees with Kissling (this Journal, 1898, April) that the English method is preferable to that employed in Germany, but characterises Kissling's proposed manner of performing the test as merely a modification of the method used in America, and as being attended with all the defects inherent in the latter. He considers that 50 grms. is too large a quantity to test, and recommends a smaller test glass (5 cm. wide and 7 cm. high). The *volume* of paraffin and the surface exposed to the air he regards as of more importance than the weight; differences in the mode of filling the vessel giving variations averaging 0·24° C. Consequently, the *depth* of paraffin in the glass should be defined in laying down rules for a uniform method. Stirring with a thermometer necessitates exposing the surface of the paraffin to the cooling influence of the air, and induces the formation of air-bubbles, which have a similar effect; hence, it is preferable to leave the paraffin quiescent.—C. S.

### *Indigo, Analysis of.* A. Brylinski. Bull. Soc. Ind. Mulhouse, 1898, **68**, 33–39.

The method of estimating indigotin, proposed by Brandt (Rev. Gén. des Mat. Col. I., 43), is untrustworthy, *first*, on account of the loss of indigotin caused by the destructive action upon it of the solvent (aniline) used, and *secondly*, owing to the fact that the indigotin extracted, even after washing with an acid and alcohol, contains aniline.

In consequence of these defects, the author has tried glacial acetic acid as a solvent, and he finds this to be entirely suitable for the purpose.

The acetic acid solution of indigotin obtained in the extraction (by means of a Soxhlet apparatus) of indigo or indigo-dyed fabrics, is cooled and diluted with four times its volume of cold water, any precipitate remaining on the sides of the flask being removed by washing with boiling water or, if necessary, being redissolved in acetic acid and precipitated with water. The precipitate is left at rest for a few minutes, in order that it may become flocculent, and is then collected on a filter, and washed successively with boiling water, alcohol, and ether. It is dried at 110° C. Washing with alcohol and ether is necessary to remove from the precipitate certain substances derived from the corks or filter-papers used, or the material extracted.

Owing to its comparatively slight solubility in acetic acid, extraction of indigotin with this menstruum takes much longer time than the process of extraction with aniline; about 0·15 grm of indigotin is dissolved per 5 hours.

The following results indicate the accuracy of the method:—

Taken 0·169 grm. of indigotin, crystallised from aniline and washed with dilute hydrochloric acid and alcohol, found 0·151 grm., or 89·4 per cent.

Taken 0·022 grm. of indigotin, crystallised from aniline, and then from acetic acid and washed; and found, 0·023 grm.

Taken 0·096 grm. of "indigo pure" (B.A.S.F.); found 0·095 grm.—E. B.

### *Methoxyl Estimation.* G. Gregor. Monatsh. für Chem. 1898, **19**, [2 and 3], 116—121.

The author modifies Zeisel's process for the estimation of the methoxyl group. In place of the alcoholic solution of silver nitrate prepared according to Zeisel's direction, a decinormal alcoholic solution is employed, made by dissolving 17 grms. in 30 c.c. of water, and diluting to one litre with commercial absolute alcohol. The solution is standardised against decinormal potassium thiocyanate. For an ordinary analysis, 50 c.c. of this solution are placed in the first flask, 25 c.c. in the second, both acidified by a few drops of nitric acid free from nitrous acid. The alkyl iodide is more quickly decomposed, and the separated silver iodide only contains traces of nitrate, which are readily removed by washing. After the completion of the operation, the clear liquid is poured off the silver iodide into a 250 c.c. flask, the iodide is repeatedly treated with cold distilled water, allowed to settle, and the water added to the solution in the flask. The solution in the second flask is diluted with water and also brought into the graduated flask, which is then filled up to the mark, well shaken, and the contents filtered through a dry filter into a dry vessel. For the titration, 50 or 100 c.c. are taken, acidified with nitric acid free from nitrous, and ferric sulphate added. The reduction of the silver nitrate caused by the use of phosphorus in the Geissler's apparatus is avoided by using instead, a solution of one part of potassium carbonate and arsenious acid in 10 parts of water. The arsenious acid retains free iodine; the excess of carbonate any hydriodic acid carried over. The whole operation by this process becomes complete in two hours. The test analyses quoted are satisfactory.—A. C. W.

### *Tartaric Acid in Crude Materials, Estimation of.* From The Chemische Fabrik, Winkel am Rh. Zeits. Anal. Chem. **37**, [5], 312—313.

For the estimation of the total tartaric acid in wine residues and crude tartar, the following method is recommended:—In a 100 c.c. flask 6 grms. of the finely powdered residue are digested with 9 c.c. of dilute hydrochloric acid (sp. gr. 1.1) for at least two hours at the ordinary temperature. This acid extract is then diluted to 100 c.c. with water, shaken and filtered through a dry filter. 50 c.c. of the filtrate are now placed in a covered beaker, 18 c.c. of a 20 per cent. solution of potassium carbonate are added, and the liquid is heated until the calcium carbonate formed, has settled down. This precipitate is filtered off and washed with hot water, and the filtrate evaporated in a porcelain basin to about 15 c.c. When cold, 3 c.c. of glacial acetic acid are added with stirring, and the whole allowed to stand overnight. 100 c.c. of alcohol (94—96 per cent.) are then stirred in, the precipitated tartaric acid is filtered off and washed with alcohol, and finally dissolved in hot water. It may now be titrated with ½ normal potash solution.

In the case of tartar or calcium tartrate, 3 grms. of the substance should be taken. After digesting with 9 c.c. of the hydrochloric acid, the solution is made up to 100·5 c.c. 50 c.c. of the filtered solution are used for the estimation.
—W. P. S.

### *Succinic Acid, Estimation of, in Presence of Tartaric and Lactic Acids.* F. Bordas, Joulin, and de Raczkowski.

Journ. Pharm. Chim. 1898, 7, [9], 417—418.

The solution of the three acids is neutralised by decinormal potash, excess of a concentrated solution of silver nitrate added, the precipitate filtered off and washed until the washings no longer precipitate potassium chromate. The silver succinate remaining on the filter is washed into a dish, two drops of neutral potassium chromate added, and then decinormal sodium chloride until the precipitate becomes white. Decinormal silver nitrate is now added until the silver chromate coloration appears. The difference between the volumes of sodium chloride and silver nitrate used is proportional to the amount of succinic acid, 1 c.c. = 0·0059 grm. The tartaric acid being determined by ordinary methods, the lactic acid is obtained by difference.
—A. C. W.

### *Hübl's Solution, the Formation of Acid in.* J. J. A. Wijs.

Zeits. Anal. Chem. 37, [5], 277—283.

The author does not agree with the statement of Schweitzer and Lungwitz (this Journal, 1895, 130—133 and 1030) that the acid formed by the action of Hübl's solution on fats, is hydriodic acid, but is, as already found by Waller, hydrochloric acid. As regards the formation of the acid, he suggests that it is produced by the addition of the iodine chloride and the splitting off of a halogen atom (HCl) from the addition product.—W. P. S.

### *Fatty Oils, Uniform Methods in Testing.* D. Holde.

Chem. Rev. Fett- u. Harz-Ind. 5, [3], 41—43.

*Iodine Number.*—The author agrees with Henriques that determinations made by the Waller method should be marked "[W]" in published results, owing to the differences between the values in comparison with the Hübl method.

At the Charlottenburg Versuchsanstalt, the Hübl method, with 24 hours' exposure, is generally employed, a saving of the reagent being thereby obtained, since an excess of 40 per cent. of iodine gives accurate results, whilst the ordinary (two hour) method necessitates an excess of 70—75 per cent., a quantity which, when used under the modification referred to, gives values (in the case of linseed oil) 1—2 units lower than the smaller excess.

*Saponification Value.*—An examination of Henriques' proposed method of substitution of sodium hydroxide for potassium hydroxide, showed that the soda soaps readily separate, and that it is therefore preferable to use, as hitherto, potash. In order to avoid the error in titration due to the carbon dioxide liberated by the hydrochloric acid, the latter should not be added until, after five minutes' boiling, the liquid no longer exhibits a red coloration. This error Allen and Stange found to amount to three units.

Referring to cold saponification, he acknowledges the superiority of this method for waxes, and recognises its utility for fish and other oils that assume a brown coloration in presence of warm alcoholic alkali, though this latter difficulty can be overcome by substituting Alkali Blue for phenolphthalein as indicator. In the absence of a generally reliable method of procedure, cold saponification is inapplicable for examining mixtures of fatty oils and mineral oils.—C. S.

### *Fatty Oils, Testing: Notes on Holde's Paper.* R. Henriques.

Chem. Rev. Fett- u. Harz-Ind., 5, [3], 43—45.

*Iodine Number.* The advantage of the Waller reagent consists in its greater stability, a sample titrated at the end of 24 hours, and again after 12 weeks' standing, giving the values 45·43 and 41·08 respectively, whereas the Hübl reagent under the same conditions decreased in standard from 45·14 to 26·43.

The cause of the lower values given by the Waller method in the case of linseed oil is to be sought in an insufficiency of chloroform in the liquid to retain the addition products in solution, whereby a portion of the oil is apparently carried down and removed from the influence of the iodine.

Wolfbauer and Ulzer's direction with regard to the amount of chloroform should, therefore, be amended by the proviso that sufficient chloroform must be present to prevent precipitation during the experiment.

This question of chloroform does not appear to influence the differences noticed in the case of wood oil, the tendency to precipitation being less than with linseed oil. The differences between the values obtained by Holde and the author consequently still remain unexplained.

*Saponification Value.*—In view of the difficulties caused by the presence of alkali carbonates, the author now proposes to employ sodium hydroxide for all ordinary oils, using potash solely for solid fats and waxes forming very hard soda soaps. He regards Holde's proposal to remove the carbon dioxide by boiling, as too troublesome for practical use.

For mixtures of fatty and mineral oils the cold saponification method, performed in the same manner as for waxes, is recommended.—C. S.

### *Fatty Substances, Estimation of Ash in.* A. Delecocuillerie.

Bull. Assoc. Belge des Chim. 12, [1], 5—12.

In the usual methods a troublesome process of desiccation is necessary for the complete removal of water, since otherwise portions of the ash constituents pass away in solution in the ethereal filtrate. To obviate this source of error the author proposes to melt a weighed sample of the substance in a tared platinum capsule at a gentle heat, and then immerse in the liquid mass a small ashless filter, folded in four, the upper part of which is then ignited. By this means the fatty matter is burned off without spurting or decrepitation, sufficient heat being evolved to keep the substance fluid and to drive off the last traces of moisture. A gentle calcination completes the process.

The results obtained from various fats and waxes are concordant for each substance. In the case of fats containing large admixtures of saline matter, the substance requires heating on a water oven during the test, on account of the difficulty with which the combustion is then attended.
—C. S.

### *Fats, Testing for Rancidity.* A. Schmid.

Zeits. Anal. Chem. 37, [5], 301—303.

The author distinguishes between a rancid fat and an acid or sour fat. A fat is sour when the quantity of free fatty acid contained in it is abnormally large, the free glycerin however remaining unchanged, and rancid, whenever the free glycerin is partly or wholly oxidised into aldehydes and ketones, the free fatty acid at the same time being small in amount.

The rancidity of fats may be determined by estimating these aldehydes and ketones in the following way:—20 grms. of the fat together with 100 c.c. of water are placed in a flask and distilled in a current of steam. A 100-c.c. flask serves as a receiver, and into this, before the distillation commences, 5 c.c. of a freshly prepared solution of metaphenylenediamine hydrochloride are introduced. The distillate from fresh butter only shows a faint yellow colour, whilst rancid fats yield a deep yellow or brown distillate. The author has not yet collected sufficient data to form a table showing degrees of rancidity.—W. P. S.

### *India-Rubber, Vulcanised, The Analysis of.* C. O. Weber.

Zeits. f. angew. Chem. 1898, 313.

The following figures were obtained in the analysis of two samples of india-rubber sheet, said to be of exactly the same composition, but one of which, (*a*), had undergone decomposition, while the other one, (*b*), was sound.

| — | (a.) | (b.) |
|---|---|---|
| | Per Cent. | Per Cent. |
| Oily extract.... | 8·35 | 2·82 |
| India-rubber .. | 9·85 | 12·73 |
| Mineral compounds. | 79·90<br>Per Cent.<br>63·11 mineral compounds<br>16·79 carbon | 82·34<br>Per Cent.<br>62·81 mineral compounds<br>19·53 carbon |
| Total sulphur.. | 1·89 | 2·10 |

The correctness of these analyses, especially the enormous proportion of carbon and the extraordinarily small proportion of india-rubber, seemed to be open to serious doubts, and a critical examination of the above figures appeared to point to the carbon being a product of the analytical process, rather than an original constituent of the samples. It was suspected that in the determination of the india-rubber by the nitrobenzene method (this Journal, 1894, 484), the boiling nitrobenzene had acted otherwise than merely as a solvent, upon the india-rubber. Experiments to carry out the nitrobenzene treatment at from 170° to 190° C. proved ineffective. The above analyses were then repeated, and 3 grms. of each sample were first, for one hour, allowed to stand with 3 c.c. of chloroform. Then 50 c.c. of nitrobenzene were added and the contents of the flasks for one hour were heated to boiling point on a reflux condenser. After cooling, 100 c.c. of ether were added to each flask, and the solutions so obtained were filtered. Not a trace of carbon was now observed, the filter residues consisting only of mineral matter, 63·33 per cent., and 63·02 per cent. respectively. This appeared to show that the carbon first observed was simply produced through carbonisation of the india-rubber at the high boiling point of nitrobenzene, for it was found that the above mixture of nitrobenzene and chloroform boiled considerably lower, at from 170° to 180° C. The real cause of the carbonisation became, however, at once apparent on making a detailed analysis of the mineral constituents obtained in the above analyses.

*Nitrobenzene Treatment.*

| — | (a.) | (b.) |
|---|---|---|
| | Per Cent. | Per Cent. |
| Carbon.......................... | 17·22 | 19·93 |
| Mineral compounds:— | | |
| $CaCO_3$.......................... | 20·78 | 21·04 |
| $PbCO_3$ ....................<br>$PbO$.......................... | 40·27 | 41·83 |
| $PbS$ ..........................<br>$PbSO_4$.......................... | 2·20 | 1·00 |

*Nitrobenzene-Chloroform Treatment.*

| | | |
|---|---|---|
| | Per Cent. | Per Cent. |
| Carbon .......................... | Trace | .. |
| Mineral compounds:— | | |
| $CaCO_3$.......................... | 21·02 | 21·11 |
| $PbCO_3$..........................<br>$PbO$.......................... | 32·22 | 31·56 |
| $PbO_2$................ ...... | 9·97 | 10·05 |
| $PbS$..........................<br>$PbSO_4$ .... ................ | 1·05 | 0·88 |

The essential difference between these two series consists in the large percentage of carbon observed in the first series, and its total absence in the second, coupled with a large percentage of lead peroxide in the latter, and its absence in the first series. The disappearance of the lead peroxide in the first series makes it at once plain that the carbon observed in this series is due to the action of the lead peroxide at the temperature of boiling nitrobenzene (208° C.), which action can be prevented by lowering the boiling point of the solvent to from 170° to 180° C. by the addition of from 5 to 6 per cent. of chloroform to the nitrobenzene.—C. O. W.

*Raw Tanning Materials, Analysis of.* W. K. Alsop and J. H. Yocum. J. Amer. Chem. Soc., **20**, [5], 338—340.

The authors publish the results of a large number of exhaustive analyses of different kinds of bark. The tannins were estimated as the difference between soluble solids and non-tannins, the red colouring matter as the difference between total solids and soluble solids. Besides these constituents, the following were also determined:—Moisture, cellulose and insoluble matter, carbon, the ash as a whole, and also each separate constituent of the same.—E. H. T.

*Massecuite, Determination of Crystallised Sugar in.* E. Lallemant. Bull. de l'Assoc. des Chim. de Sucr. et de Dist, 1898, **15**, [10], 1038—1040.

The apparatus for this purpose consists simply of a copper pipe, surrounded with a jacket, which can be filled with hot water. At the bottom of the pipe a disc of the centrifugal cloth or gauze is soldered, and beneath this a small capsule is attached by a bayonet fastening. The whole is swung round like a sling by means of a cord attached to a handle at the top. From the polarisation of the massecuite and the syrup, and, if necessary, of the crystals also, the percentage of crystals is calculated.—L. J. de W.

*Sugar, Estimation of; Electrolytically.* J. Formánek. Zeits. Unters. Nahr. und Genussmittel, 1898, [5], 320—322.

Instead of filtering the precipitated cuprous oxide through asbestos and reducing it by hydrogen, the author proposes to filter through paper, wash off the filter with nitric acid (sp. gr. 1·2), add ammonia (sp. gr. 0·96) and ammonium nitrate, and to separate the copper by electrolysis. The method gives good results, and is suitable for determining the sugar in wines, milk, and liqueurs.—L. J. de W.

*Wine and Acetic Acid, Detection of and Approximate Estimation of Sulphuric Acid in.* F. Bimbi. Bull. Chim. Farm., **37**, 133—134; Chem. Centr. 1898, **1**, [13], 793.

Free sulphuric acid is usually recognised in beverages either by extraction of the residue with absolute alcohol or by means of Pollacci's method. If only little sulphuric acid has been added, both of these methods fail, since in the wine, the sulphuric acid reacts with the potassium tartrate with the formation of tartaric acid and potassium bisulphate, whilst the acetic acid is sometimes preserved in earthenware vessels, which are attacked by the sulphuric acid with formation of sulphates. For the detection of small quantities of fraudulently added sulphuric acid, the author recommends the estimation of the tartar, free tartaric acid and sulphates, and "comparison with the average values ascertained in the portion concerned."—A. S.

*Sesamé Oil in "Margarine."* M. Siegfeld. Chem. Zeit. 1898, **22**, [32], 319—321.

The author finds that the colour test for sesamé oil with furfurol and hydrochloric acid is most readily obtained when it is carried out at a temperature of 70° C. In order to approximately estimate the amount of sesamé oil in "margarine," use was made of solutions in dilute sulphuric acid, of uniformly increasing quantities of dimethylamidoazobenzene, so as to form a colour-scale. These were employed to compare with the colour produced by the oil in the margarine, having previously been compared with the colours produced by known quantities of sesamé oil.

—W. P. S.

*Iodoform, Determination of.* G. Meillière. Répert. Pharm. 1898, [3], **10**, 102.

The iodoform is placed in a flask, evaporated to dryness if in solution, and 25 c.c. of pure nitric acid added, with an excess of silver nitrate (1·7 grm. to 1 grm. of iodoform). A Liebig's potash apparatus, containing silver nitrate solution, is connected with the flask, which is then heated for 10 minutes nearly to boiling, and then boiled till no more nitrous fumes are evolved in the flask. The contents are then diluted to 150 c.c., boiled again, and the silver iodide collected and weighed. If the liquid in the potash bulbs shows any turbidity it is of course added to the contents of the flask.—J. T. D.

*Alkaloids in Tinctures and Extracts, Determination of.* J. Katz. Pharm. Zeit. 1898, **41**, [31], 273.

In the following processes the application of heat, which is deleterious to many alkaloids, is entirely obviated. 25 c.c. of a tincture containing 45 per cent. of alcohol, are extracted in a separator with 50 c.c. of ether and 1 c.c. of caustic soda solution (33 per cent.). The alkaline aqueous solution is again washed out twice with 25 c.c. of ether containing 10 per cent. of alcohol. The mixed ethereal solutions, after first washing with a very little water, are shaken with 2 to 3 grms. of dried plaster of Paris, then run into a stoppered flask, and titrated with $\frac{1}{100}$ normal HCl solution, with Iodeosin as an indicator. For the tinctures of plants containing alkaloids not readily removed by ether alone, a mixture of ether 1 and chloroform 3 is employed. The chloroform extract is washed out with a few c.c. of 30 per cent. brine. If separation of the solvent from the aqueous solution does not quickly take place, it may be accelerated by the addition of 2 or 3 grms. of sodium chloride. When the alcoholic strength of the tincture exceeds 45 per cent., it is diluted with water. Preparations containing chlorophyll are first acidulated and the precipitated colouring matter filtered off, before treating by this process. From 1 to 1·5 of an extract is dissolved in 40 to 50 c.c. of alcohol, 45 per cent., and the alkaloids are then determined as in the case of a tincture.—J. O. B.

*Atropine, Estimation of.* H. M. Gordin and A. B. Prescott. J. Amer. Chem. Soc. **20**, [5], 329—338.

Atropine and iodine combine with one another in varying proportions, according to the experimental conditions; but a definite, stable compound, atropine ennea-iodide, can be obtained if due precautions are taken. In this body, one-ninth of the total iodine is firmly combined, whilst eight-ninths are easily removed by reducing agents, so that the formula, $C_{17}H_{23}NO_3.HI.I_8$, has been given to it. Based upon these facts is the following analytical process for estimating atropine:—The aqueous solution of the atropine salt, the strength of which must not exceed $\frac{1}{2}$ per cent., is added very slowly and with constant agitation to 20—30 c.c. of $\frac{1}{10}$N iodine solution, until the turbidity first formed has given place to a dark, granular precipitate, and the supernatant liquid become transparent, though still of a dark red colour. The whole is then well shaken and diluted to 100 c.c. Of this liquid an aliquot portion is filtered off and the excess of iodine determined with $\frac{1}{10}$N sodium thiosulphate. Every c.c. of the iodine solution consumed, corresponds to 0·003608 grm. of atropine. This factor is obtained on the assumption that of the nine atoms of iodine in the ennea-iodide, eight are supplied by the free iodine of the $\frac{1}{10}$N iodine solution, the ninth atom coming from the potassium iodide of that solution. This assumption has been experimentally verified by the authors. The analytical data show that very good results are obtainable by this method, the success of which depends upon the atropine solution being sufficiently diluted.

Atropine may also be estimated by means of the iodomercurates, Atrop. $HI.HgI_2$ + (Atrop. $HI)_2.HgI_2$, which are easily obtained, and then determining the mercury and iodine contained in these bodies.—E. H. T.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Aluminium as a Reducing Agent, &c.* L. Franck. Chem. Zeit. 1898, **22**, [25], 236—245.

*Action of Aluminium on Phosphorus Compounds.*—Phosphorus vapour when led over powdered aluminium, heated to a dull red heat in a current of hydrogen, combines with it with incandescence, forming a dark greyish-black unfused mass, which is decomposed in contact with moist (normal) air, forming $PH_3$, and leaving a greyish-white powder. It is decomposed also by water, aluminium hydroxide and a brownish-black residue being left; and by acids and alkalis, which dissolve it almost completely with evolution of $PH_3$. The compound remains unaltered when heated in air.

At more or less elevated temperatures, all phosphoric acid compounds (meta-, pyro-, and ortho-salts alike) are decomposed by aluminium. Metaphosphates, however, undergo the most complete change, according to the equation—

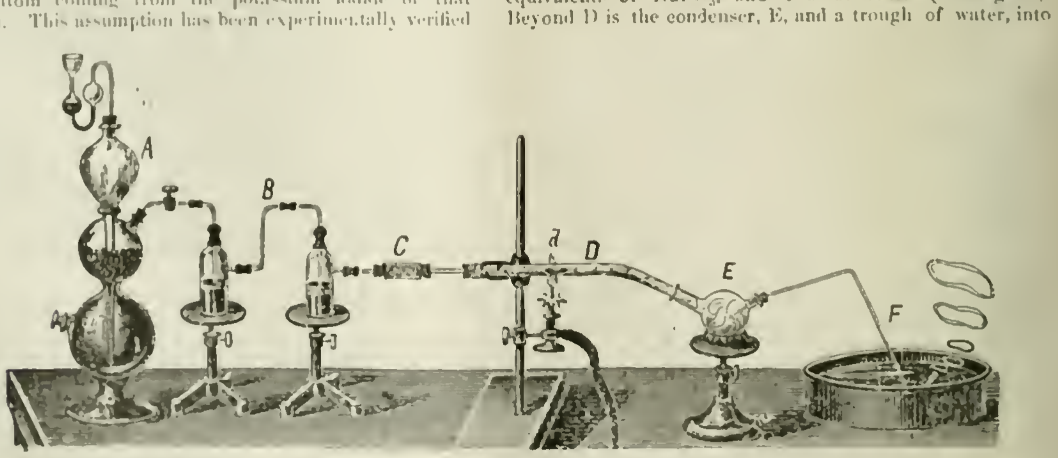
$$6NaPO_3 + 15Al = 6NaAlO_2 + 2Al_2O_3 + Al_5P_3 + P_3$$

The addition of silica effects the release of the remaining phosphorus, thus:—

$$6NaPO_3 + 10Al + 3SiO_2 = 3Na_2SiO_3 + 5Al_2O_3 + 3P_2.$$

Calcium and magnesium salts are as efficacious as those of sodium, but the superphosphates of commerce are not available for the production of phosphorus in this manner. If, however, bone ash be decomposed by hydrochloric acid instead of by sulphuric acid, a material suitable for the purpose is obtained.

Hence phosphorus may be produced, with almost quantitative completeness of yield, at relatively low temperatures, and the reduction may be demonstrated in the apparatus shown in the annexed figure. Hydrogen generated in A is dried by the sulphuric acid bottles B, and is then passed through the Fresenius safety-tube C to the combustion-tube D, which is 0·5 m. long, and contains a porcelain boat, *d*, carrying the charge of 2·1—2·5 equivalents of Al, six equivalents of $NaPO_3$, and two of silica (Kieselguhr). Beyond D is the condenser, E, and a trough of water, into which the outlet tube, F, is passed. On heating the boat at *d*, particles of phosphorus soon condense on the walls of the tube, in its cooler section, and in the condensing-bulb. If the flow of hydrogen be too rapid, some phosphorus is carried over, and is spontaneously kindled as it escapes into the air; but with a slow current no phosphorus need be lost.



*Action of Aluminium on Carbon Compounds: On Carbon Dioxide.*—On passing dry $CO_2$ over aluminium powder heated to redness, the temperature of the mass rose rapidly owing to the resulting reaction. After cooling in the same gas, the residue was found to consist of $Al_2O_3$ and amorphous carbon; but it gave a small quantity of an evil-smelling gas when treated with hot dilute HCl, so that traces of an aluminium carbide must have been formed. No carbon monoxide was detected throughout; but the reaction corresponded almost exactly with the equation $3CO_2 + 4Al = 2Al_2O_3 + 3C$. Aluminium sheet and wire heated in a current of $CO_2$ becomes covered with a black crust; at the same time it is brittle, and is partly oxidised, partly converted into a carbide.

*Action on Carbon Monoxide.*—An exactly similar result was obtained by heating aluminium powder in CO. The reaction is $3CO + 2Al = Al_2O_3 + 3C$; but, as with $CO_2$, a trace of carbide is formed.

*Action on Carbonates.*—Alkaline carbonates and aluminium mixed in equal proportions according to the equation $Na_2CO_3 + 2Al = Al_2O_3 + C + Na_2$, and heated to a red heat, became incandescent, and gave alkali metal and amorphous carbon. Lithium, sodium, and potassium were thus reduced, the latter with almost theoretical yield. No potassium carbide was observable. Calcium, barium, and strontium were reduced in the same way, and barium crystals were sometimes obtained, but never crystalline carbon. Nevertheless, some aluminium carbide and a little nitride were also formed.

*Aluminium and Carbon.*—The author confirms Guntz and Masson's statement (this Journal, 1897, 244) that the presence of aluminium iodide or chloride favours the formation of carbide when Al is heated in CO or $CO_2$, but he reaffirms the correctness of his own general equations given above. Aluminium powder heated with lampblack appears unchanged, but evolves much hydrocarbon gas when placed in water. The excess of lampblack could not, however, be removed. Much carbide may be formed by direct combination (Deville to the contrary notwithstanding), but the reaction is never complete; and Moissan's carbide, $Al_4C_3$, has not been obtained except in the electric furnace.

*Action of Aluminium on Oxides.*—Copper oxide (cuprous or cupric) mixed with the correct proportion of aluminium powder for reduction, and heated gradually, reacted suddenly, with a report like that of a gun. The glass tube was shattered and small copper shot were found. With an excess of aluminium, the residue was an aluminium bronze. Silver oxide was similarly attacked. Beryllium was also reduced at a red heat, but quietly, and with only slight incandescence. Calcium could be partly reduced from lime, and calcium alloys could thus be readily obtained. Strontium was reduced from the oxide with a more marked rise of temperature. Baryta is much more readily, and is indeed almost completely reduced, with distinct incandescence. Zinc oxide is reduced with quiet combustion and a blue-white luminous flame. Cadmium and mercury and lead oxides also yield metal, the former quietly, the second almost at once, and the last-named with explosion. Boron, silicon, phosphorus, and arsenic also, are all separated in the elementary state on heating the oxide with powdered aluminium. Iron, manganese, cobalt, nickel, and molybdenum are all partly reduced; in the electric furnace new compounds are thus produced.

*Action on Sulphates and Chlorides.*—Barium (or other) sulphate, mixed with excess of aluminium powder and heated, explodes with a loud report, shattering the glass tube in which it is held. Finely divided sulphur and sulphides are among the products of the reaction. Sodium, potassium, calcium, and barium chlorides, when melted, yield metal on the introduction of Al powder, the two latter reacting more rapidly and completely.

*Action on Sodium Peroxide.*—Aluminium powder mixed with sodium peroxide explodes at a red heat. If the mixture be moistened with water it inflames spontaneously.
—W. G. M.

### *Nitric Oxide, Absorption of, by Ferrous Salts.* V. Thomas. Bull. Soc. Chim. 1898, **19**, [8], 343—347.

It has previously been shown by the author that solutions of all ferrous salts absorb nitric oxide, but that the amount varies with the solvent employed. The experiments now recorded show that this absorption is not proportional to the amount of iron present, but is a general function of the particular salt employed. Thus, ferrous bromide in aqueous solution, absorbed for every 100 parts of iron in solution, between − 5° and 0° C., 41·73; at 0° to 7° C., 35·87; at 8° to 11° C., 24·9; and at 20·5° C., 15·43 parts of NO. With absolute alcohol as a solvent, at 17° C. the absorption is 28·33 of NO, and at 11° C. 37·94 parts. With ferrous iodide, temperature does not greatly influence the amount, the quantity of NO absorbed by an aqueous solution at 16° C. being 18·29 for every 100 of iron, and at 32° C., 20·62 parts, and nearly the same amount in an alcoholic medium.—J. O. B.

### *Iron Carbide (Cohenite) in the Terrestrial Nickeliferous Iron of Niakornak, Jacobshavn, N. Greenland; The Occurrence of.* E. Cohen. Meddelelser om Grönland, 1897, **15**, 293; Chem. Zeit. Rep. 1898, **8**, 74.

Cohen has shown that iron carbide occurs in the nickeliferous iron of Ovifak. 50·2847 grms. of the nickeliferous iron from Niakornak were dissolved in dilute HCl, with evolution of hydrocarbons, yielding 52·18 per cent. of greyish-black iron carbide, the other constituents being 46·53 per cent. of nickeliferous iron, which passed into solution; 0·06 per cent. of carbon; and 1·23 per cent. of stoney particles consisting of plagioclase, monoclinic augite, hematite, and earthy matter. If the iron is dissolved with exclusion of air, no carbon separates. The specific gravity of the cohenite is 7·5124 at 21·8° C., and the average of four analyses shows the composition of the pure mineral to be, Fe, 92·01; Ni, 1·13; Co, 0·37; C, 6·49 per cent., so that (FeNiCo):C = 3·084:1. Cohenite from terrestrial nickel-iron has thus the same composition as that from meteorites. The nickel-iron in the solution had the composition, Fe, 97·03; Ni, 2·09; Co, 0·71; Cu, 0·02; S, 0·04; P, 0·11 per cent. Therefore the iron of Niakornak consists principally of cohenite and nickeliferous iron, and its hardness is attributable to its high proportion of cohenite. A polished surface, on etching with concentrated nitric acid, shows a beautiful granular structure, the nickel-iron being dissolved out.—H. B.

### *Bismuthous Oxide.* L. Vanino and F. Treubert. Ber. **31**, [8], 1113—1118.

Attempts to prepare this oxide by Thomson's method of heating bismuth in air showed clearly that the resulting substance was a mixture of bismuthic oxide and unaltered metal, and that increased duration of the heating or a higher temperature converted it completely into $Bi_2O_3$.

No better success resulted from a repetition of Vogel's precipitation of bismuthic salts by stannous chloride and alkali. The resulting precipitate, when an excess of stannous chloride and alkali was used to precipitate a known weight of bismuthic salt, weighed less than the calculated amount of BiO, though it contained both tin and alkali; and when, before weighing, it was very thoroughly washed with weak hydrochloric acid and hot water (which completely removed the tin and alkali, while mere traces of bismuth were found in the washing liquids) the precipitate caked under a rod to metallic-looking masses, and its weight was found to agree exactly with that of the bismuth contained in the original salt. The authors represent the reaction by the equation—

$$Bi_2O_3 + 3Sn(ONa)_2 = 2Bi + 3Na_2SnO_3$$

and they regard Vogel's precipitate as a mixture of bismuth with the metahydrate of $Bi_2O_3$. They are pursuing their researches; but they point out that the different accounts of the reputed bismuthous oxide are very contradictory, and that there has been no character attributed to it which might not have been attributed to a finely-divided mixture of bismuthic oxide and metallic bismuth.—J. T. D.

### *Strontium Sulphide, Phosphorescent; Properties of.* J. R. Mourelo. Comptes Rend. **126**, (12), 904—906.

Intimate mixtures were made of barium sulphate, strontium sulphate, or calcium sulphate, with strontium sulphide, or with the materials for its production. In some instances the mixture (containing 5 to 10 per cent. of strontium sulphide) was uniformly phosphorescent without further treatment, in others it became so after being exposed to a bright red heat for some hours. Barium sulphate gave the best results, calcium sulphate the poorest. In all cases the phosphorescence, though uniformly distributed over the mixture, was but feeble in intensity, and not readily excited.—J. T. D.

### *Carbon Bisulphide, Observations on the Action of Oxygen on, and on the Chemical Influence of Light. The Work which Predetermines the Reactions.* Berthelot. Comptes Rend. 1898, **126**, [15], 1060—1066.

The action of oxygen on carbon bisulphide proceeds directly at 200° C. and upwards, but the action which takes place at ordinary temperatures, and under the influence of light, raises some general problems in chemical mechanics, as the following experiments indicate:—Three flasks, of a capacity of 2½ to 3 litres, filled with air, and each enclosing a sealed tube containing about 1 grm. of carbon bisulphide, were sealed up, and the tubes within were broken. The weight of bisulphide taken, readily vaporised in the flask, but the oxygen of the contained air was insufficient for its complete oxidation. On March 18, 1897, two of the flasks were put in a light place, to which direct solar rays could not penetrate, and the third flask was exposed to the direct action of the sun. On March 31, 1898, the flasks were removed and their contents examined. The two exposed to diffused light only, showed that no appreciable oxidation or change had occurred in the course of the year. The flask exposed to the solar rays, on the other hand, exhibited a whitish deposit within it from a few hours after it was exposed, and the deposit continued for some time to increase. Analysis of the residual gas and solid products at the end of the year, showed that of the 1·070 grm. of carbon bisulphide taken, 0·309 grm. had disappeared, while 0·160 grm. of oxygen had been absorbed. The carbonic acid and carbonic oxide in the residual gas accounted for 0·028 grm. of this oxygen, and most of the surplus oxygen (= 0·132 grm.) was combined in alkaline carbonates and sulphates formed from the alkali of the glass of the flask. The greater part of the sulphur in the carbon bisulphide decomposed, was found on the glass in an insoluble and amorphous condition, but the whitish deposit on the glass also contained a brownish-red subsulphide of carbon. The carbon bisulphide remaining yielded, on slow evaporation, a product which contained in the combined state, sulphur, carbon, and oxygen. No fixation of nitrogen was observed in this case.

A sufficiently intense luminous radiation such as the direct rays of the sun, therefore, is required to set up oxidation of the bisulphide. The oxidation at ordinary temperatures is induced by the preliminary work of luminous energy. The partial character of the reaction must not be attributed to the establishment of a chemical equilibrium, but to the physical alteration of the conditions arising from the deposit on the interior surface of the glass. The deposit absorbs and arrests the rays which produced it, and thus prevents the formation of further deposit.—J. A. B.

### *Partial Racemism.* A. Ladenburg and W. Herz. Ber. 1898, **31**, [7], 637—638.

The solubility of quinine *l*-pyrotartrate had not yet been determined. A specimen of *l*-pyrotartaric acid, which only possessed two-fifths of its proper rotatory power, was converted into quinine salt and the solution evaporated until separation of salt commenced. The acid of the separated salt had a smaller rotation than the original. Thus quinine *l*-pyrotartrate is more soluble than the i-pyrotartrate. A solution of the lævo-salt obtained from the mother liquor of strychnine *d* pyrotartrate was allowed to deposit two crops of crystals; the acid separated from the mother liquor contained about 74 per cent. of the *l* acid. The quinine salt prepared from this acid dissolved to the extent of 15 parts in 100 parts of alcohol. The corresponding salts of the *i*- and *d*-acids dissolve to the extent of 3·2 and 1·2 parts in 100 respectively. Thus the quinine salt of the inactive acid is a salt of the racemic form and not a mixture of salts of the active acids. This is then a case of "partial racemism," *i.e.*, the circumstance of the combination of two bodies which are only in part "mirror images" of one another (the base being the same in each component), the compound thus still possesses optical activity.—A. C. W.

### *Hydrazine Derivatives, Poisonous Action of.* O. Loew. Chem. Zeit. **22**, (35), 349—351.

The author has examined the action of solutions containing from ¼ to 1 per cent. of typical hydrazides which react easily with aldehydes, on various low forms of animal and plant life (infusoria, algæ, spirogyra, bacteria, yeast), as also on sprouting barley and twigs of Tradescantia They were all found to exert a powerful poisonous action, as does hydrazine itself. Semi-carbazide in the free state was found to act more strongly than amidoguanidine as nitrate, though neither of them was so powerful as hydrazine. The hydrazide of protocatechuic acid, however, exhibited this poisonous action quite as strongly as hydrazine. In some instances, the order of intensity of action was a little altered, no doubt from the fact that one or other of the substances examined was rapidly decomposed by, or entered into combination with, some constituent of the organism experimented upon.—J. T. D.

### *Chlorination of Compounds of the Aromatic Series by Means of Ferric Chloride.* V. Thomas. Comptes Rend. 1898, **126**, [17], 1211—1214.

The fact that ferric chloride alone is directly capable of effecting the chlorination of the benzene nucleus appears to have been overlooked. The action takes place at ordinary temperatures to a slight extent, and at 80° C. is energetic, with a considerable separation of carbon and formation of monochlorobenzene; similarly, by acting on phenyl chloride with ferric chloride, dichlorobenzene is produced. In a similar way, the whole series of chlorobenzenes may be obtained. By acting on the bromo-substitution products of benzene, interesting results have been obtained; thus phenyl bromide treated with ferric chloride gave the chlorobromide $C_6H_4ClBr$. Toluene is easily chlorinated with ferric chloride, but the action is strictly limited to the benzene nucleus and no benzyl chloride is formed.—J. O. B.

### *Bone, Softening of, by Means of Phloroglucol.* J. J. Andeer. Comptes Rend. 1898, **125**, [18], 1295.

It is found that an aqueous solution of phloroglucol, especially when used with hydrochloric acid, has a remarkable effect in decalcifying bones, so that anatomical or microscopical preparations may be obtained in which the most delicate organic tissues are unattacked, but the bone completely softened and decalcified. The residual portion, termed by the author "ossein," is free from any trace of calcium phosphate or carbonate. The method is not applicable to horn or ivory, nor to the siliceous matter of sponges; but the author states that by a modification of the process, concerning which further details are promised, phloroglucol may be equally useful for softening these bodies.—J. O. B.

### *Oxidation of Certain Acids in Presence of Iron.* H. J. Horstman Fenton. Proc. Chem. Soc. 1898, [191], 119.

It has been shown in several previous communications (Trans. Chem. Soc. 1894, **65**, 899; B. A. Report, 1895, &c.) that when tartaric acid is oxidised in presence of a small quantity of ferrous iron, one molecule of the acid loses two atoms of hydrogen, giving rise to dihydroxymaleic acid. The most effective oxidising agent for the purpose is hydrogen dioxide, but the result is also brought about by chlorine, hypochlorites, bromine, &c., and by atmospheric oxygen in presence of sunlight. The presence of ferrous iron is essential, but its proportion seems to bear but little relation to the yield of acid in the ordinary course of preparation, the action being, in fact, what is usually termed catalytic. It is necessary that the addition of iron shall precede that of the oxidising agent. From the fact that dihydroxymaleic acid, on heating with water, yields glycollic aldehyde, and that this readily condenses to a hexose, it is evident that the change may afford information with regard to the natural formation of carbohydrates.

The production of dihydroxymaleic acid from tartaric acid is most easily represented by assuming the removal of the two non-hydroxylic hydrogen atoms—

$$C_2H_2(OH)_2(CO_2H)_2 - H_2 = C_2(OH)_2(CO_2H)_2.$$

It is true that dextrotartaric acid would be expected, in this manner, to yield a fumaroid instead of a maleoid acid; but it is quite possible that "stereomeric" change takes place during one of the stages of preparation, and in any case this objection can hardly be considered a serious one, bearing in mind the large number of known exceptions (Michael, J. prakt. Chem. 1892, **46**, 400).

### PATENT.

*Tobacco Leaves, a Process for the Treatment of.* J. C. F. W. Feldhausen, Bremen, Germany. Eng. Pat. 4533, Feb. 23, 1898.

The tobacco-leaves are saturated "in a dilute solution of potash, for the purpose of the elimination of nitrogen and other substances." On drying the leaves, ammonia is volatilised.—E. S.

## New Books.

Metric Equivalents of Imperial Weights and Measures (based on the Board of Trade Standards), together with Thermometric Equivalents, in Degrees Centigrade, Fahrenheit, and Réaumur. [Pharmaceutical Journal Reprint.] Pharmaceutical Journal Office, 5, Searle Street, Lincoln's Inn, London, W.C. 1898. Price 1*s.* 6*d.*

"The metric system of weights and measures is now legalised in the United Kingdom for purposes of trade, and it appears desirable that the extended use of the system should be encouraged as much as possible. Several of these tables, notably those referring to length and weight, are based on those printed in the Pharmacopœia of the United States, 1890. After careful revision and extension, they are here reprinted in a more convenient form for reference."

The publication is of quarto size, and of pamphlet form.

Chemisch-technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 36. Jahrgang. 1897. Zweites Halbjahr. Zweite Hälfte. Mit in den Text gedruckten Illustrationen. Hermann Heyfelder, R. Gaertner's Verlagsbuchhandlung. Schönebergerstr. 26, Berlin, S.W. H. Grevel and Co., 33, King Street, Covent Garden, London.

The second issue for the second half of 1897 of Dr. Jacobsen's Repertory of Chemical Technology, containing reports of the progress of the following branches of chemical industry:—I. Foodstuffs. II. Paper. III. Photography. IV. Residuals; Manures; Disinfection and Sanitation. V. Soaps. VI. Explosives and Matches. VII. Preparation and Purification of Chemicals. VIII. Fine Chemicals, &c. IX. Chemical Analysis. X. Apparatus, Machinery, Electrotechnology, and Thermotechnology. XI. Appendix, &c. XII. New Books.

Jahresbericht über die Leistungen der Chemischen Technologie, mit besonderer Berücksichtigung der Electrochemie und Gewerbestatistik für das Jahr 1897. Von Dr. Ferdinand Fischer. Mit 250 Abbildungen. Verlag von Otto Wigand, Leipsic, Germany. 1898. H. Grevel and Co., 33, King Street, Covent Garden, London. Price 24*s.*

The progress and position of Chemical Industry up to the end of the year 1897 are here treated of. Trade statistics, with more special reference to the German Empire, are also given. The last Jahresbericht (1896) contained 1,183 pages; the present one (1897) contains 1,171. Alphabetical indexes of names and subject-matter are given, covering 43 pages. An index of German patents follows those of names and subject-matter.

The text is illustrated with 250 woodcuts referring to plant and apparatus. The following is a synopsis of the subjects treated of in the volume, the extent of each group and its treatment being indicated by the number of pages filled:—

Group I. Chemical Technology of Fuel (pages 5 to 158). II. Chemical Metallurgy (159 to 340). III. Electrochemistry (341 to 419). IV. Chemical Manufactures; Inorganic (420 to 560). V. Chemical Manufactures; Organic (561 to 704). VI. Glass, Pottery, Cements, Artificial Stone (705 to 775). VII. Foodstuffs (776 to 1010). VIII. Chemical Technology of Fibres (1011 to 1071). IX. Other Industries connected with Organic Chemistry (1072 to 1144). X. Apparatus. (1145 to 1162).

It was noticed in the review of the Jahresbericht for 1894, that the subject of Electrochemistry filled 85 pages; for 1895 it filled 106 pages; for 1896, 103 pages; but for 1897 it only fills 78 pages.

## Trade Report.

### PRIZES.

Prize for Matches free from Phosphorus.

The Belgian Government is offering a premium of 50,000 frs. to the inventor of a paste for matches which will be free from white phosphorus, and which will ignite on cloth or any other surface. The competition will be international in character, and will remain open until January 1, 1899.—*Westminster Gazette.*

### TARIFF CHANGES AND CUSTOMS REGULATIONS.

Portugal.

*Tariff Modifications.*

Her Majesty's Minister at Lisbon transmits copies and translations of two laws dated respectively the 2nd and 3rd May last.

The former exempts sundry chemical products specified therein, and also manures for agricultural purposes, from any Customs charges or import duties.

The latter fixes the rate of import duty on quinine salts at Reis 8,000 per kilo. of net weight.

The laws in original may be seen on application at the Commercial Department, Board of Trade, Whitehall Gardens, S.W.

Austria-Hungary.

*Importation of Saccharin and other Substances Prohibited.*

Her Majesty's Secretary of Embassy at Vienna encloses copy of the *Reichsgesetz Blatt* of the 20th April, containing Ministerial Order prohibiting the importation into Austria-Hungary of saccharin and other analogous substances.

Spain.

*Proposed Export Duties.*

Her Majesty's Ambassador at Madrid states that the commission on the budget propose to establish the duties on exports in accordance with the five following tariffs. The duties to be per ton of 1,000 kilos.:—

1. Iron ore and common salt, 0·30 peseta.
2. Stones, worked clay, minerals, except iron, combustibles of all classes, manure, and other animal deposits, 0·50 peseta.
3. Metals in ingots, bars, plates, and tubes, ordinary drugs, paints, dyes, varnishes, chemical products, glass, and porcelain, 1 peseta.
4. Raw fibres, including esparto grass, wine, oil, and other alimentary substances, 2 pesetas.
5. Hardware, pharmaceutical products, silver, and articles not included in the former classes, 3 pesetas.

*Note* 1.—Cork, linen rags, cotton, or hemp, and used effects of the same materials, are excepted from this tax, and only pay the duties imposed by the ordinary export tariff.

Galena, argentiferous lead, and litharge ores are also excepted from the export duties imposed by this law, but will pay the duties imposed by the export tariff, as the suspension of payment granted by law of July 10, 1897, will cease.

*Note* 2. —In cases of doubt, the duties of the repertory of the Customs tariff will serve as models.

The duties on exportation given in this law, if approved of by Parliament, will remain in force during the whole financial year 1898–99.

### REVISED TARIFF OF VALUES FOR BRITISH INDIA.

*Bd. of Trade J., June* 1898, 696.

| No. in Tariff schedule. | Names of Articles. | | Previous Valuation. Rs. | a. | Revised Valuation. Rs. | a. | p. |
|---|---|---|---|---|---|---|---|
| | CHEMICALS, DRUGS, MEDICINES, AND NARCOTICS, DYEING AND TANNING MATERIALS. | | | | | | |
| 10 | Chemical products and preparations: | | | | | | |
| | Acids, sulphuric ... | per lb. | 0 | 2 | 0 | 1 | 6 |
| | Alum ............... | per cwt. | 5 | 0 | 4 | 10 | 0 |
| | Sal ammoniac ...... | ,, | 37 | 0 | 26 | 0 | 0 |
| | Sulphate of copper. | ,, | 15 | 8 | 17 | 8 | 0 |
| 11 | Drugs, medicines, and narcotics:— | | | | | | |
| | Aloes, black .......... | per cwt. | 14 | 0 | 15 | 0 | 0 |
| | Asafoetida, coarse (hingra). | ,, | 21 | 0 | 25 | 0 | 0 |
| | Brimstone (amalsára) | ,, | 50 | 0 | 35 | 0 | 0 |
| | Camphor, refined cake | per lb. | 1 | 4 | 1 | 2 | 0 |
| | Cassia lignea ......... | per cwt. | 26 | 0 | 28 | 0 | 0 |
| | Cubebs ............... | ,, | 25 | 8 | 15 | 0 | 0 |
| | Pellitory (akalkára) . | ,, | 40 | 0 | 30 | 0 | 0 |
| | Peppermint crystals, from China and Japan. | per lb. | 11 | 8 | 7 | 0 | 0 |
| | Senna leaves ......... | per cwt. | 4 | 0 | 5 | 0 | 0 |
| | Storax, liquid (rose maloes). | ,, | 54 | 0 | 40 | 0 | 0 |
| 12 | Dyeing and tanning materials:— | | | | | | |
| | Alizarin dye:— | | | | | | |
| | Dry, 40 per cent. ... | per lb. | 1 | 7 | 1 | 5 | 0 |
| | ,, 50 ,, ... | ,, | 1 | 10 | 1 | 8 | 0 |
| | ,, 60 ,, ... | ,, | 1 | 15 | 1 | 12 | 0 |
| | ,, 70 ,, ... | ,, | 2 | 2 | 2 | 0 | 0 |
| | ,, 80 ,, ... | ,, | 2 | 8 | 2 | 4 | 0 |
| | ,, 100 ,, ... | ,, | 2 | 12 | 2 | 10 | 0 |
| | Liquid, 10 per cent. | ,, | 0 | 5 | 0 | 4 | 6 |
| | ,, 16 ,, | ,, | 0 | 8 | 0 | 6 | 6 |
| | ,, 20 ,, | ,, | 0 | 10 | 0 | 8 | 6 |
| | Aniline dye, liquid, indigo blue. | ,, | 0 | 9 | 0 | 8 | 0 |
| | Avar bark ........... | per cwt. | 4 | 8 | 3 | 8 | 0 |
| | Buzgand (gulpista) .. | ,, | 27 | 0 | 34 | 0 | 0 |
| | Gallnuts (myrabolams) | ,, | 4 | 0 | 3 | 0 | 0 |
| | Madder or manjit .... | ,, | 7 | 0 | 11 | 0 | 0 |
| | Orchilla weed ........ | ,, | 5 | 0 | 4 | 0 | 0 |
| | OILS. | | | | | | |
| 16 | Oils:— | | | | | | |
| | Cajeputi ............. | per qrt. | 1 | 4 | 2 | 0 | 0 |
| | Cassia ............... | per lb. | 2 | 8 | 3 | 12 | 0 |
| | Earthnut ............ | per cwt. | 16 | 0 | 18 | 0 | 0 |
| | Grass ................ | per lb. | 1 | 12 | 1 | 8 | 0 |
| | Jinjili or til .......... | per cwt. | 10 | 0 | 18 | 0 | 0 |
| | Sandalwood ......... | per lb. | 7 | 0 | 6 | 0 | 0 |
| | OTHER ARTICLES. | | | | | | |
| | Glass, glassware, and false pearls:— | | | | | | |
| | Glass, china, all colours | per 133⅓ lb. | 32 | 0 | 38 | 0 | 0 |
| | ,, crown, coloured | per 100 super. ft. | 15 | 0 | 11 | 0 | 0 |
| | ,, ,, of sizes.. | ,, | 6 | 0 | 5 | 0 | 0 |
| 59 | Gums, gum-resins, and articles made of gum or gum-resin: | | | | | | |
| | Cutch and gambier . | per cwt. | 20 | 0 | 15 | 0 | 0 |
| | Gum, Arabic ....... | ,, | 18 | 0 | 22 | 0 | 0 |
| | ,, Bysabol (coarse myrrh). | ,, | 16 | 0 | 20 | 0 | 0 |
| | ,, Persian (false) | ,, | 11 | 0 | 9 | 0 | 0 |
| | Rosin ............... | ,, | 6 | 0 | 5 | 0 | 0 |

### UNITED STATES.

*Duty on Spent Oxide of Iron.*

Oxide of iron which has been used to purify coal-gas by abstracting sulphur therefrom, dutiable at 10 per cent. *ad valorem* under section 6, Act of 1897, as an unenumerated unmanufactured article.—Protest of Nichols Chemical Co.; United States General Appraisers at New York.

*Duty on Coal-Tar Products.*

*Eng. and Mining J., May* 21, 1898, 608.

In the case of a protest of Schoelkopf, Hartford, and Maclagan against a ruling of the Collector of Customs at New York, the Board of General Appraisers holds as follows in a decision just published:—"Coal-tar products, known variously as 'dead oil,' 'tar oil,' 'liquid creosote,' 'creosote oil,' &c., are not dutiable at 25 per cent. *ad valorem* under the provisions of paragraph 60, Act of August 28th, 1894, for 'distilled oil,' or as 'chemical compounds,' but are free of duty under the provisions of paragraph 443 of said Act."

### FRANCE.

*Oxalic Acid and Alcohol.*

*Ch. of Commerce J., June* 1898, 132.

A decree, dated June 1st, fixes the legal tare at 9 per cent. on oxalic acid imported in casks or cases of wood. Other decrees have appeared relative to wines considered as over alcoholised, and on the use of denatured alcohol in industry. The text of these decrees may be seen on application at the Information Office of the London Chamber of Commerce.

### CANADA.

*Reduction of Tariff.*

We are asked by the High Commissioner for Canada to state that the rebate of 25 per cent. off the duties on certain articles, the growth, produce, or manufacture of the United Kingdom, imported into Canada, comes into operation on July 1st next, and not August 1st, as stated in a paragraph recently circulated. The reduction will apply also to similar imports from the foreign countries entitled to the Reciprocal Tariff under most-favoured nation treaties, from July 1st to July 31st. On and after August 1st, however, the rebate will be confined to goods, the growth, produce, or manufacture of the United Kingdom, and of the British colonies and possessions entitled to the benefits of the preferential tariff.

## EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.

### SULPHUR NEAR ANCONA.

*Ch. of Com. J., May* 1898, 104.

British Vice-Consul Tomassini (Ancona) states that a few years back a German syndicate commenced to search for sulphur near Sassoferrato, and after encountering great difficulties their efforts have now completely succeeded. The mine is situated at Cabernardi, and is known under the name of "Azienda Solfifera Italia." The machinery employed consists of a steam boiler of 80 h.p., a motor for the extraction of the mineral, and 35 furnaces. During 1897 the mine produced 40,000 tons of sulphur which was entirely disposed of in Italy. The number of hands employed in the interior of the mine amounts to 150 adults and 100 on the exterior, whilst work lasts for about 250 days in the year.

### SIBERIAN INDUSTRIES.

*Board of Trade J., June* 1898, 660.

A brief account of the industries carried on at the present time in Siberia may be of some interest:—

*Distilled Spirits.*—The distilling industry is chiefly concentrated in the Irkutsk government, next in the Yenisseisk, and in Transbaikalia. In the Yakutsk territory distilling is entirely absent. Of 21 distilleries in Western Siberia, nine are in the Tobolsk government, 11 in that of Tomsk, and one in the territory of Semipalatinsk. In Eastern Siberia there are 22 distilleries, and in the Littoral district but one.

Beer and mead brewing are proportionally better developed. In 1895, 67 breweries were in operation, 19 of which also produced mead. The principal breweries are located in Tomsk, Yenisseisk, Tobolsk, Irkutsk, and Akmolinsk. The total output is estimated to average about 375,000 vedros of beer and 75,000 vedros of mead (1 vedros is equivalent to 3·249 galls.).

Thus, the local production of drinks subject to excise cannot satisfy the existing demand for them, and, accordingly, they are, like spirit and vodkas, imported from various parts of the empire by land or by way of Odessa and Vladivostock.

The sugar industry is a perfectly new enterprise in Siberia. It could never rise independently, and accordingly the Government recognised the utility of offering the pioneers in this industry in Siberia certain privileges, as was done in Turkestan and the Caucasus.

The first beet sugar factory was opened in 1890, in the Minonsinsk district of the Yenisseisk government. In 1890 only 8,450 poods (1 pood equals 36 lb.) of beets were treated, but in 1891, 92,000 poods, from which 5,850 poods of white sugar were obtained. The experience of two years completely convinced the manufacturers that the conditions of soil and climate of the Minonsinsk district were perfectly adapted to the cultivation of the sugar beet, and accordingly the extension of the undertaking appears to be extremely advantageous.

Match manufacture is growing from year to year. There were, in 1894, 11 factories, three in Eastern and eight in Western Siberia. The annual output averages:—

| | Matches. |
|---|---|
| Eastern Siberia, with phosphorus..... | 430,000,000 |
| Eastern Siberia, without phosphorus.. | 128,000,000 |
| Western Siberia, with phosphorus..... | 5,000,000,000 |
| Western Siberia, without phosphorus. | 50,000,000 |

The principal factories are in Tobolsk, Tomsk, and Irkutsk.

There are, further, the following industries not subject to the payment of excise:—

| | Estimated Number of Manufactories and Works. |
|---|---|
| Hides, sheepskins, and leather goods...... | 310 |
| Metals .................................... | 16 |
| Milling.................................... | 384 |
| Tallow and soap boiling .................. | 104 |
| Timber sawing ............................ | 6 |
| Candles (tallow and wax)................. | 21 |
| Brick and lime burning.................... | 35 |
| Porcelain, faience, and glass ............. | 12 |
| Cloth, wool washing, and felt ............ | 18 |
| Saltworks and salt-grinding .............. | 14 |
| Confectionery, molasses, &c. .............. | 12 |
| Chemical, vinegar ........................ | 4 |
| Ropewalks ................................ | 5 |
| Oil mills and cheese-making .............. | 35 |
| Writing paper............................. | 3 |

It must, however, be borne in mind that most of these industries are carried on but to a small extent, which may be gathered from the fact that the total production of the Siberian manufactories and works does not reach a value of 10,000,000 roubles; the first place among the manufacturing industries belongs to milling, about 45 per cent.; the second to the leather and sheepskin trade, after which follow tallow and soap boilings, metals, &c.

### Chemical Trade of Hamburg in 1897.

*Foreign Office Annual Series, No. 2104, May 1898.*

Business in dye-woods last year was prejudicially affected by the excessive importations of the year 1896, by which this and inland markets had been considerably overstocked. Trade in these products was accordingly very quiet, especially in the various sorts of logwood, the prices of which declined seriously. Importations of Campeachy logwood amounted to only 17,800,000 kilos. in 1897, against 29,600,000 kilos. in the previous year; but those of St. Domingo and Jamaica logwood were rather more extensive, *viz.*, 8,750,000 kilos., against 7,900,000 kilos. in 1896. Prices of yellow wood, after declining during the first five months of 1897, suddenly recovered and were subsequently maintained at a high figure; the total importations amounted to 3,000,000 kilos., that is to say, to 600,000 kilos. less than in 1896. The importations of red-wood, particularly of Lima sorts, were also less extensive than in 1896, reaching altogether about 1,100,000 kilos. last year.

Of Quebracho wood no less than 71,700,000 kilos. were brought to Hamburg, against 58,200,000 kilos. in 1896. These large importations show in themselves the important part which this article now plays in the seaborne trade of this port, and they prove that Hamburg is the leading market for it. In spite of its great importance Quebracho wood cannot, however, be said to have driven oak bark out of the field. The substitute for oak bark is chiefly now oak extract, which is imported from Austria and Hungary, or chestnut extract, which comes from France and Italy; whilst Quebracho wood has, in fact, created an entirely new industry, *i.e.*, the rapid tanning process. It appears that this process would be impossible without employing Quebracho wood.

The quantity of indigo imported here last year was 5,400 cases, against 6,059 cases in 1896, the stock on hand at the end of the year having been 950 cases. In spite of the certain prospect at the beginning of 1897 of a small crop of East Indian indigo in 1897—98, business here was very limited, and prices maintained a falling tendency.

The total importations of nitrate to this port in 1897 amounted to 378,000 tons, against 536,000 tons in the preceding year. The expected improvement in the nitrate business did not take place in this market last year, and, on the contrary, matters were indeed less favourable for Hamburg importers than the year before. The attempt made in 1897 in Chili by the "Salitreros" to limit production and to keep up prices is stated to have proved a failure, owing, on the one hand, to the already large stocks existing in Europe, and, on the other hand, to the "Salitreros" themselves having, after determining to fix the future output according to the production of a given period, forced the production during this period to an unreasonable degree. In consequence, moreover, of the appearance of new producers in the nitrate market at the same time, the "ring" collapsed. The fact that prices which stood here in January 1897 at 7 marks 95 pf., for February—March 1898 delivery, fell to 7 marks 10 pf. in December of last year, sufficiently shows the unsatisfactory course which the nitrate business took in the 12 months under review.

Last year's exportations from Hamburg of the various potato products manufactured in Germany, *viz.*, potato-starch and flour, dextrine (British gum), and grape-sugar, were less extensive than in 1896, the chief reason being the low prices of competing manufacturers in Holland and of corn-starch in the United States of America. The total exportations of potato starch and flour from all Germany (Hamburg exports not yet published) were 283,036 cwt. in 1897, as against 678,728 cwt. in 1896; of dextrine, 28,614 cwt., as against 51,728 cwt.; and of grape-sugar, about 45,000 cwt. in 1897, as against 89,546 cwts. in the previous year.

Amongst the industries of this district which have attained to the comparatively greatest importance are those for manufacturing various chemical products, *viz.*, nitrates, sulphuric and nitric acid, sulphates, boracic acid, artificial manures, pharmaceutical products, dyeing and tanning extracts, essences, and particularly also different kinds of explosives. These factories now employ altogether about 4,000 workmen, against only about 1,300 10 years ago. The rapid strides made, indeed, throughout Germany by most branches of chemical industry during recent years is proved by the fact that, according to the latest trustworthy statistics the total number of chemical works registered in all parts of the German Empire was 6,144, the total number of persons employed by them being 125,440.

One of the prominent branches of chemical industry in this district is at present the manufacture of explosives, in particular of dynamite and of all kinds of gunpowder, for which several large establishments exist in this neighbourhood. An idea of the importance of these factories may be obtained from the export returns of this port, which, in 1896, show that about 465,000*l.* worth of dynamite, gunpowder, guncotton, &c., were shipped from Hamburg in 1896 to foreign countries, the larger proportion being destined for South Africa. Besides this quantity, a considerable amount of explosives were sent by rail and river to the interior of Germany.

### Palm Kernel and Linseed Crushing at Harburg.

*Foreign Office Annual Series*, No. 2104. *May* 1898.

Harburg ranks as the principal centre for the palm-kernel crushing in Europe. During the past, crushing has constituted a good source of revenue; of late, however, this trade has been most severely handicapped through the abolition of slavery on the African coast, which had the effect of raising the cost of the raw material in such a measure that, although the factories, by way of keeping working, have sold the oil below cost price, they are no longer able to compete with the substitutes of oil, and will, in all probability, have to abolish the production of palm oil and take to other products.

But this paralysis has been outweighed by the activity displayed in the linseed crushing trade. Up to 1898 there was but one factory working; the good results obtained, however, caused the erection of a second plant, and although the production was doubled and all the quantity of linseed cake found its way to the same consumers in Great Britain, prices were maintained. The production was again reduced through the new factory being destroyed by fire in 1897, but it has again been erected and recommenced operations.

### Sulphate of Copper at Zante.

*Foreign Office Annual Series*, No. 2105, *May* 1898.

A good quantity of sulphate of copper (principally in casks of 5 cwt.) is now being imported at various Greek ports, mostly from Liverpool; it costs about 17*l*. per ton; it is used to combat the "peronosporos" disease when the shoots of the vines are still young, flowers of sulphur being applied in the later stages.

### Soda-ash at Zante.

*Foreign Office Annual Series*, No. 2105, *May* 1898.

Soda-ash, used by soap-boilers, comes mostly from Liverpool; the quantity imported altogether was 3,126 casks, value equal to about 5,000*l*.

### Pyrene Oil and Soap at Zante.

*Foreign Office Annual Series*, No. 2105, *May* 1898.

Of pyrene oil, extracted from the stones and refuse of the olives by chemical process, very little was exported in its liquid state, most of it being used in producing green soap, the cost of which was about 24*l* and 32*l*. per ton f.o.b. according to quality. Olive oil soap, about 35,000 cases of 50 okes each (1 oke equal 2·84 lb.) were exported mostly for the Levant, each case costing 50 dr. or about 1*l*. 5*s*. per case.

### Soap Trade of Corfu in 1897.

*Foreign Office Annual Series*, No. 2105, *May* 1898.

The amount of soap exported in 1897 is stated to have been 187,145 okes, contained in 7,358 cases of 140 lb. each, and valued as per Annex B at 7,550*l*. sent chiefly to Albania, Dalmatia, Italy, and Egypt. Soda-ash and pyrene oil are used in its manufacture.

### Chemicals in Barcelona in 1897.

*Foreign Office Annual Series*, No. 2110, *May* 1898.

The business in chemicals from England was fairly good, but not up to that of past years. The trade in artificial manure has fallen off considerably, the farmers now buying raw materials and mixing them themselves instead of ordering expensive fertilisers. A large mill is being erected by an influential German company at a place called Fax, situated on the river Ebro, in the province of Tarragona, for the purpose of manufacturing by an electrical process caustic soda and other chemicals in common use in this country, and should the venture prove successful, it will probably interfere greatly with the importation from Great Britain.

### Sugar Industry of Baden in 1897.

*Foreign Office Annual Series*, No. 2093, *May* 1898.

In comparison to many other sugar factories, the Mannheim refinery did a tolerably good business. Notwithstanding Germany's enormous beet sugar production, sugar in Germany remains somewhat an article of luxury, the price being comparatively dear and the consumption small. This is due to the Government paying an ever-increasing premium on all sugar exported, and, to recoup itself, exacting a very heavy tax amounting at present prices to over 100 per cent. on all sugar consumed in Germany itself. Of course, this state of things is condemned, and there is a desire to get rid of both tax and premium. As long, however, as other countries adhere to export premiums there is no prospect of remedy. The situation grows acute owing to the steadily-increasing production, assisted by greater skill alike in the cultivation and manufacture of the beet.

### Chemical Industry of Baden in 1897.

*Foreign Office Annual Series*, No. 2093, *May* 1898.

The chemical industry suffered by higher cost of raw materials, while lower prices had to be conceded for most of its products. The great coal-tar colour factories were in sharpest competition; they continue to pay large dividends, and may do so for some years to come. The soda market was affected by sharper competition of English makers, and also by the opening of a new factory in Elsass. At present prices the Leblanc soda makers it is said cannot exist. An agitation of the soap manufacturers for a reduction or abolition of the duty caused, therefore, much disquiet, as such a measure, it is maintained, would ruin the German soda interest. The manufacture of sulphuric acid was but slightly remunerative, and superphosphate suffered severely by an invasion by the Belgian article, assisted by a reduction of railway freight of 20 per cent. on artificial manures. Heavy chemicals in general were hit by the United States Tariff.

### Sicilian Sulphur Exports.

*Foreign Office Annual Series*, No. 2120, *June* 1898.

*Girgenti.*

The sulphur trade has received a remarkable stimulus during 1896 and 1897, since the establishment of the Anglo-Sicilian Company, which has put a stop to the competition held by the local exporters. Although it has raised the price to a reasonable rate, the shipping of the article has been more active than in previous years, as will be seen from the following return:—

| To | Quantity. | | |
|---|---|---|---|
| | 1895. | 1896. | 1897. |
| | Tons. | Tons. | Tons. |
| United Kingdom | 11,908 | 8,548 | 12,155 |
| United States of America | 74,322 | 84,960 | 76,166 |
| France | 44,831 | 48,390 | 46,175 |
| Italian ports | 5,905 | 6,106 | 5,852 |
| Russia | 8,770 | 2,952 | 6,818 |
| Germany | 5,025 | 4,000 | 8,831 |
| Austria | 2,937 | 2,260 | 2,714 |
| Other countries | 11,848 | 14,839 | 11,054 |
| Total | 165,546 | 172,055 | 172,765 |

*Licata.*

The output of sulphur for last year gave a total of 165,704 tons, of which 94,847 tons were duly shipped, and 70,857 tons remained warehoused up to Dec. 31, as is clearly shown by the following table:—

| Quality. | Quantity. |
|---|---|
| | Tons. |
| Second best best Licata | 169 |
| Second best Licata | 8,050 |
| Second good Licata | 24,757 |
| Third best best Licata | 13,346 |
| Third best Licata | 13,024 |
| Third good Licata | 11,511 |
| Total | 70,857 |

The quantity of sulphur exported from Licata to foreign countries during 1897 was 85,741 tons; to Italy, 9,106 tons; total, 94,847 tons, amounting to 331,964*l.*, showing an increase of 20,808 tons and 124,875*l.* above the preceding year.

Sulphur carried by British vessels amounted last year to 79,365*l.*

Of the 85,741 tons of sulphur shipped for foreign countries, 31,813 tons went to France; 30,828 tons to the United States of America; 4,085 tons to Norway and Sweden; 2,367 tons to Spain; 2,339 tons to Austria; 2,232 tons to the United Kingdom (London); 1,983 tons to Germany; and 10,094 tons to other countries.

### *Catania.*

The quantity of sulphur exported from Catania was 90,590 tons, as against 95,678 tons, in 1896.

This diminution will probably be more than compensated in the coming year, the cause being that the users of the ground article have delayed their purchases in the hope of seeing lower prices.

The operations of the "Anglo-Sicilian Sulphur Company, Limited," have been a boon to the mine proprietors, who are now able to earn a good profit on their industry, with the result that several millions of lire remain in the island to their and the miners' benefit, whilst before the advent of said company the industry was at the mercy of speculators.

In June 1897 the company entered into contracts for two years with 12 (out of 15) of the refiners at Catania to work up, at fixed prices, 110,000 tons of crude sulphur into the various refined qualities which are used in commerce, with faculty to sell two-thirds of the quantity at 3 per cent. selling commission.

These contracts have also worked well, with satisfactory results to both parties.

The average price of crude sulphur for 1897, taking best thirds as a basis, has been 3*l.* 13*s.* 7*d.* per ton f.o.b. Catania, as against 3*l.* 7*s.* for 1896.

## SUMACH AND ESSENCES IN SICILY.

*Foreign Office Annual Series, No.* 2120, *June* 1898.

A decree was issued during 1897 bearing on the adulteration of sumach and essences of lemon, &c., by which the exporter was compelled to mark on the packages, invoices, &c., the quality and quantity, if any, of the adulterant used. This law has, however, not been put into force, and the general opinion is that it will remain a dead letter.

The entire production of sumach in Sicily ranges from 420,000 to 500,000 cantars (13 cantars = 1 ton). The quantity exported being about 560,000 cantars, the difference being accounted for by the adulteration with "Lentisco," and a few years ago with "Bruca," the former being imported from the Barbary Coast, and the latter from Calabria.

## IODINE AT STAVANGER, NORWAY.

*Foreign Office Annual Series, No.* 2119, *June* 1898.

A manufactory for burning seaweed into kelp, and preparing it into iodine, has been started. Enormous lots of seaweed are found along the coast, but most of it has been exported as kelp to Scotland. The iodine manufactory, "Hillevaag Keniske Fabriker," is not in the international ring or syndicate, but the owners feel sure it will prove successful.

At the Stockholm Exhibition a Norwegian engineer exhibited an invention for producing paper-glue, dressing-gum, and soap from seaweed. He said that the first manufactory in this branch would be erected in this district owing to the near access to the seaweed, but nothing has yet been done.

## ELECTRIC INDUSTRIES AT SARPSBORG, NORWAY.

*Foreign Office Annual Series, No.* 2119, *June* 1898.

Since the Kellner Partington Paper Pulp Company, Limited, of Manchester, erected their mills at the Sarpsborg Falls a few years ago, they have been yearly extending their business, so that they now employ over 1,000 workmen. The latest addition, which the company are now adding to their estate, is a large electric power station for the accumulation of motive power to be derived from the waterfall. When complete they will be able to supply the surrounding district both with electric light and motive power at a minimum of cost. The Kellner Partington Company is, however, not the only firm who are beginning to utilise the vast water-power which the Sarpfos contains. A new company, the so-called "Hafslund Aktieselskab," which has been floated chiefly with German capital, is also erecting an enormous electric power station on the opposite bank of the falls, which when complete will be able to supply 25,000 horse-power. Both companies are calculating that the cost of producing the electricity will be so trifling, that they will be able to supply the whole of Fredrikstad—a distance from Sarpsborg of about seven English miles—and the surrounding mills and factories advantageously with motive power.

The Hafslund Aktieselskab have already contracted for the supply of motive power to two new factories now in course of erection, *viz.*, one for the manufacture of aluminium and another for the production of calcium carbide used for the manufacture of acetylene gas. Besides these two new enterprises, contracts are also said to have been made for the supply of power to two other factories, the nature of which has not yet transpired. This new and sudden industrial activity is naturally a great boon to Sarpsborg, which, from being a few years back a sleepy, unimportant place, is rapidly developing into a busy manufacturing town. The large influx of workmen has necessitated the erection of more dwelling-houses, and small wooden huts are now springing up in every direction.

## BORAX IN PERU.

*Foreign Office Annual Series, No.* 2117, *June* 1898.

The borax industry appears to be assuming some importance. In 1896, 7,350 tons, valued at 36,970*l.*, were exported, whilst in 1897, 12,464 tons, valued at 62,420*l.*, were shipped from Mollendo. The deposits are found some 20 miles in the interior from Arequipa. This product has greatly increased and improved trade in that city, owing to the number of mules and donkeys employed in the carriage down from the deposits to the railway station at Arequipa, and to the impetus given to agriculture on the adjacent lands by the cultivation of alfalfa (lucerne) for the maintenance of these animals, the value of the alfalfa having increased fourfold in as many years; this is now one of the greatest interests that the department of Arequipa has to depend on, and apparently the supply of borax is inexhaustible.

## CHEMICALS AT BILBAO.

*Bd. of Trade J., June* 1898, 670.

Chemical and pharmaceutical products at Bilbao hardly come at all from Great Britain. The market is supplied partly from France and much more by Germany. Surgical instruments, syringes and similar articles sold by druggists are very largely supplied from Germany. Chemical products used in industries are mostly supplied from the United Kingdom. Belgium appears to compete in caustic soda and carbonate of soda. France, Germany, and recently Belgium have been competing with Great Britain in the supply of chloride of lime. France seems to send the most.

It is clear that if trade be thought worth having it can be best secured through a representative. The need of ascertaining the exact tastes or prejudices of the purchaser, of making sure that purchaser and seller exactly understand each other, of learning what is being done by competitors, are the more obvious reasons. The advantages of travel were experienced a few weeks ago by an agent who went to Bilbao to see if there was any market for a certain commodity. He was at the moment not prepared to accept orders, but in one or two places his wares were so much wanted that orders were pressed on him. He was told that no British traveller had presented himself for many years. In one place he was shown an article that had come from Germany. Inspection showed that it had been made by the house which he represented. It had been first sent to Hamburg, re-made up for sale into packages containing a slightly different quantity, and then sent as German. In another place he found his goods disguised as French. Sold in the first instance in Paris, they had been submitted to a simple process and re-sold as French.

### QUICKSILVER AND COPPER IN OREGON.

*Foreign Office Annual Series, No.* 2115, *June* 1898.

At Black Butte Lane County, Oregon, a valuable deposit of cinnabar has been opened, which reports say can be worked very economically at a cost of 10 cents per lb. This is probably an understatement, but the deposits are said to be very large.

There are said to be immense deposits of copper in the Seven Devils district in Idaho, which it is claimed rivals the richness of the mines of Butte, Montana, but it is a difficult country to open up, although the mines have been worked more or less for years. It is said that 120,000*l.* was paid during the year for one property. There are copper deposits all over these States embraced in this Consular district, but none of them are developed to any extent.

### TEXTILES FROM PEAT-FIBRE IN SWEDEN.

*Bd. of Trade J., June* 1898, 730.

According to a report of the French Minister at Stockholm, the industry of textiles made from peat fibre has just been introduced into Sweden. The fibres, produced from peat by a mechanical process, can be mixed in the proportion of 75 per cent. with pure wool, for the manufacture of yarn similar in appearance to common woollen yarn.

### SOAP INDUSTRY IN JAVA.

*Foreign Office Annual Series, No.* 2095, *May* 1898.

The soap industry is in a flourishing condition, and the production as well as the consumption of native-made soap is yearly increasing to the detriment of the cheaper qualities of soap of British manufacture, of which only small imports have taken place during 1897.

### FERTILISERS IN JAVA.

*Foreign Office Annual Series, No.* 2095, *May* 1898.

Imports of sulphate of ammonia have been effected in considerable quantities from Europe and Australia, chiefly for use in the sugar fields. Some planters, however, prefer to use the native or imported bungkil, the chemical properties of which become assimilated more slowly but more thoroughly with the soil. Dried blood as a fertiliser has been imported on a large scale from Australia, its use having been found advantageous on many coffee estates. The trial of nitrate from the west coast of South America, referred to in an earlier report, has not been repeated.

### CINCHONA IN JAVA.

*Foreign Office Annual Series, No.* 2095, *May* 1898.

The cinchona of Java, which island produces about two-thirds of the world's supply, has for years been regularly shipped to Holland, where it has been sold at public auction. The large quinine manufactories, mostly situated in Germany, who supply themselves with the raw material in the Dutch market, have, however, during the last five or six years combined to keep prices at such a low level as to render the cinchona cultivation unprofitable, notwithstanding the fact that large dividends have been derived from these manufactories, part of which should rightly have found their way into planters' pockets. In order to counterbalance the influence of this ring of quinine manufacturers, it was decided, as mentioned in an earlier report, to establish a manufactory in Java. This quinine manufactory has since been erected at Bandoeng, in the Preangar Regencies, and delivered last year its first product, which is said to be of first-rate quality, and in all respects equal to the best European brands. Some of the planters during the early part of the year were tempted to ship their bark to Europe rather than to consign it to the Java manufactory, in view of the sudden and important rise which took place in cinchona in Europe about the time that the Bandoeng manufactory was started. Latterly, however, they appear to have seen that this policy, which could only result in the eventual downfall of the Java manufactory, was a mistaken one, and a number of them are now affording the local manufactory good support, and are giving it a considerable share of their bark for manipulation, so that the enterprise is confidently expected to be a success.

## *GENERAL TRADE NOTES.*

### THE DRESSING OF PHOSPHORIC MAGNETITE AT LULEA.

Mr. T. Beckert, in the *Zeitschrift des Vereins Deutscher Ingenieure* (1897, p. 1307), says that the iron ore deposits of Gellivara are almost entirely worked for export. In order to utilise the more highly phosphoric varieties a large dressing and concentrating plant has been erected at Lulea by the Norbotton Ore Improvement Company, with a capacity of 100,000 tons. The treatment includes breaking in a rock-breaker, drying in Oxlad calciners, crushing by rolls to 0·5—1 mm. size, and electro-magnetic separation by the Ball and Wingham separator, which removes the clean magnetite. The waste, consisting of silicates and apatite, is dressed by jigging and washing, the apatite, when recovered, being ground to an impalpable powder in ball-mills lined with porcelain with flint grinding balls. This powder is then mixed with calcined soda-ash and heated to low redness until it is slightly fritted in a double-stage calciner similar to that used in chloridising pyrites residues. The product, when finally ground, is a fertiliser of very high value, containing about 30 per cent. of phosphoric acid in a readily soluble form. In composition it is a quadribasic phosphate, similar to the slag of the basic converter, but with one molecule of calcium replaced by sodium or $Ca_3Na_2P_2O_9$. The method, which is due to Prof. Wiborgh of Stockholm, is the result of some attempts to reproduce the slag phosphate by treating apatite with caustic lime, which, it was found, could only be effected at the melting point of wrought iron; but when sodium carbonate is substituted a much lower heat, about 700° C., is sufficient. (See also *ante*, p. 590.)

### QUICKSILVER IN NEW ZEALAND.

*Eng. and Mining J., June* 1, 1898, 665.

The Government of New Zealand has offered a bounty of 4*d.* (8 cents) per lb. for the first 100,000 lb. of quicksilver produced in the colony; but one-third of that quantity at least must be produced before March 31st 1900, and the remaining two-thirds by March 31st 1901. The bounty will amount to 8,000 dols. The existence of cinnabar in New Zealand is known. It has been found in several places, and in some of them, apparently, in sufficient quantities to warrant further exploration; but no workings have ever been undertaken.

### ALKALI IN RUSSIA.

*Chem. and Druggist, June* 11, 1898, 940.

Like the United States, Russia is slowly giving up British alkali, and makes her own by the Solvay process. Ljubimow and Solvay have three works, producing 1½ million poods (1 pood = 36 lb.) of soda annually, including one million poods of caustic soda. The imports of soda fell from 2,011,000 poods in 1885 to 887,000 poods in 1894. There is a special reason for so rapid a diminution in the imports. The purification of one million poods of naphtha-products used to require two million poods of caustic soda annually. For a long time the soda residue was worthless waste, until improved processes were devised, and a regeneration of the residue in reverberatory furnaces succeeded. Baku now requires 300,000 poods for naphtha-purification, and half of this is recovered. Glauber salt is found in vast quantities near Tiflis, but is little utilised. Most of the hydrochloric acid made in the Le Blanc process is transformed into zinc chloride for the impregnation of railway-sleepers; and bleaching-powder manufacture by the Weldon process is an increasing industry.

### Japanese Purchases.

*Chem. and Druggist, June* 11, 1898, 964.

Among the drugs and chemicals imported into Japan during 1897 were:—Alcohol, 100,974*l.*; 1896, 52,660*l.*; bicarbonate of soda, 12,044*l.*; 1896, 13,026*l.*; carbolic acid, 4,239*l.*; 1896, 13,132*l.*; caustic soda, 23,915*l.*; 1896, 9,218*l.*; chlorate of potash, 51,840*l.*; 1896, 46,928*l.*; amorphous phosphorus, 29,232*l.*; 1896, 19,129*l.*; and salicylic acid, 20,490*l.*

### American Sulphur.

*Chem. and Druggist, June* 18, 1898, 1001.

The recent high prices of sulphur in the United States have called attention to the resources there for the production of brimstone in Colorado. It is undoubtedly the case that in the Western States there are several valuable deposits, and in the mining section of the Chicago Exhibition were exhibited several excellent specimens, and information was given that the deposits only lacked capital to work them. It is said that in Colorado there is in sight about 3,000,000 tons of almost pure sulphur which is of a greyish colour, but the nearest railroad is about 40 miles distant, and the cost of transportation about 14 dols. per ton. An attempt to get this worked by New York houses has recently failed. It is also stated that the Western Chemical Company is operating the Vulcan mine in Gunnison County, Col., and has already shipped six carloads of 85 per cent. sulphur to Denver.

### Peppermint Oil.

*Chem. and Druggist, June* 18, 1898, 1000.

The production of peppermint oil in the United States is approximately 250,000 lb. a year, the bulk coming from Michigan. For many years Wayne County, N.Y., took the lead as a producer, with an annual output of 150,000 lb., but the production there has now decreased to 25,000 lb. or 30,000 lb., owing to many farmers finding prices unremunerative. The output of peppermint oil in Japan in 1897 was estimated at 187,000 lb. In 1896 Germany imported Japanese oil to the extent of 53,000 lb.; in 1895, 44,000 lb. In Thuringia, Germany, the plant has been cultivated on a moderate scale, but only a small quantity of oil has been so far produced. China, Spain, and France each grow enough peppermint to show a small surplus for export. At the low prices now ruling for American peppermint oil it is not possible for any but the largest growers to secure a profit, and the tendency is to drive out the small growers.

### Kauri Gum.

*Chem. and Druggist, June* 18, 1898, 978.

A Commission appointed by the New Zealand Government has reported upon this industry. They find that, although the yield was over 8,000,000*l.*, practically nothing was returned to the revenue of the colony, while it involved a large destruction of soil. The work is mostly done by Austrians, who send their wages out of the country. The Commission recommend that no one be allowed a licence to dig gum until after 12 months' residence in the colony; also that an export duty be levied of from 3*l.* to 5*l.* per ton.

### Ore Discovery at Dungannon.

*Ironmonger, June* 16, 1898, 518.

During the past week one of the most valuable deposits of ore and fireclay ever found in Ireland has been discovered at Dungannon. The mine belongs to the Ranfurley Coal Co. Three seams of iron ore, 6, 18, and 42 ins. respectively in thickness, containing a large percentage of metallic ore and several beds of fireclay, were found. The discovery of iron ore in this district opens up a new field of industry, as no ore was known to exist in the locality. Arrangements are being made to commence mining operations at once.

### Bermuda Arrowroot.

*Kew Bulletin, No.* 135, *pp.* 50—51.

One reason for the superior character of Bermuda arrowroot is the scrupulous care taken in every stage of the manufacture. The mode of culture is very similar to that of the common potato. The roots, after being collected, are washed, and their outer cuticle completely removed. This process requires care, for the cuticle contains a resinous matter which imparts colour and a disagreeable flavour to the starch, and cannot be removed by subsequent treatment. The pared roots are then well washed, and crushed to a pulp between powerful rollers. The pulp is thrown into perforated cylinders, where it is agitated by revolving wooden paddles, while a stream of pure water carries off the fecula from the coarser fibres of the pulp, and discharges it through the perforated bottom of the cylinder. Thence it is conveyed in pipes, and passed through fine muslin strainers into reservoirs, in which it is allowed to settle, and the supernatant water drawn off. After repeated washings, it is again allowed to settle for some time, when the surface is skimmed with palette knives of German silver. The rollers and cylinders are made of brass and copper. The drying is conducted with equal care and cleanliness. The substance, spread in flat, copper pans placed on rollers, is covered with white gauze and run under glass-covered sheds when there is danger from rain or dew. When dry it is packed with German silver shovels in new barrels lined with paper, which is stuck in with arrowroot paste. The barrels are exported on the decks of vessels under cover, since, if placed in the hold, the arrowroot might be tainted by the effluvia of other freight.

### The Mineral Production of Turkey.

*Eng. and Mining J., June* 11, 1898, 696.

The Ministry of Agriculture, Mines, and Forests of the Turkish Empire has issued a statement showing the "average annual production" of the mines of the empire. The quantities are in metric tons. This is believed to be the first official statement ever made of the mineral production of Turkey. The table is as follows:—

| | Tons. | | Tons. |
|---|---|---|---|
| Chromo ore | 36,500 | Argentiferous lead | 9,500 |
| Manganese ore | 55,300 | Lead | 2,500 |
| Emery | 12,500 | Gold and silver ore | 15 |
| Arsenic | 500 | Quicksilver | 2 |
| Antimony | 2,300 | Boracide | 16,800 |
| Copper ore | 10,000 | Borax | 3,900 |
| Zinc | 1,800 | Sulphur | 500 |
| Calamine | 650 | Lignite | 15,000 |

The production of lignite does not include that of the mines of Heraclea, which are exploited by the Ministry of Marine.

### Action of Quicksilver on Aluminium.

*Eng. and Mining J., June* 11, 1898.

According to *La Revue Technique* of Paris, M. C. Margat finds that if the surface of a plate of aluminium, which has been well sandpapered, is rubbed lightly with amalgam of mercury, there will be produced on it, within a very short time, an efflorescence of alumina. The surface of the metal will be soon covered with a growth of the alumina. But heat applied to the plate suffices, by reason of the volatilisation of the mercury, to arrest the phenomenon entirely. It is probable that the mercury induces catalytic action, and that it then allows the aluminium disintegrated by its presence to be more readily attacked by the oxygen of the air. The plate during the action becomes heated to a degree appreciable to the touch.

### Potash Salts in Germany.

*Eng. and Mining J., June* 18, 1898, 730.

At Seesen, in Brunswick, potash salts have been discovered at a depth of 2,190 ft Of the bed 27 ft. have been bored through, the cores yielding on analysis 24·14 per cent. of potassium chloride.

### Sulphate of Ammonia Production.

*Eng. and Mining J., June* 18, 1898, 730.

The production of sulphate of ammonia in Great Britain in 1897 was as follows:—Gas works, 132,724 long tons; iron works, 17,779; shale-oil works, 37,153; coke ovens and producers, 10,624; total, 198,280 long tons, an increase of 7,371 tons over 1896. In spite of the increase in production the price has risen. The increased consumption of sulphate of ammonia in making fertilisers has, however, interfered with the imports of nitrate of soda. The production of sulphate of ammonia in the United States in 1897 amounted to 3,111 tons, chiefly from coke-oven gases.

### Coke Ovens at Blast Furnaces.

*Eng. and Mining J., June* 11, 1898.

An experiment of considerable importance to large consumers of coke has been in progress for some time at the Newport Ironworks of Sir B. Samuelson and Company, Limited, Middlesborough—the erection of coke ovens close to the blast furnaces, instead of at the collieries—and a correspondent of the London "Colliery Guardian" says, that so satisfactory has been the result, that the firm are proceeding to double the number of ovens at the Newport Works, so that before long the whole of the 6,000 tons of coke, which they use weekly in their furnaces, will be manufactured within 1,000 ft. of the furnaces in which it is consumed. Some of the German ironmasters have adopted this system with advantage, and, so far, the above-named firm is said to be the only one in England that has ventured to make this new departure; but it will not long occupy this position, for other Cleveland blast-furnace proprietors, who are also colliery owners, are reported to be considering the matter, and others would be among the number if they could get the necessary land in proximity to their furnaces.

The main advantage claimed for having the coke ovens near the blast furnaces where the coke is to be consumed, is that the quality, as fed into the furnace, is better, and the quantity needed per ton of iron made is smaller than if the coke has to be brought from the colliery. There will be less moisture in the coke, especially in wet weather or in winter time. Being put into the furnace drier, the latter has less work to perform, as the moisture that is acquired on the journey has not to be evaporated, and the smelting of the ore is effected at a quicker rate, thus increasing the production of the furnaces. There is also less handling, as the coke is taken direct from the ovens to the tops of the furnaces in the ordinary barrows. By this, labour is saved, and what is of even more importance, is that the coke is much less liable to be broken. It is thus in a state better calculated to support the materials in the furnace, and more appreciated by the blast furnace manager, who calls for a hard, dry coke, which cannot easily be crushed in the furnace. The objections to the manufacture of the coke away from the colliery, it is contended, are much more than counterbalanced by the advantages. It is true that the coal which has to be carried from the pit to the coke ovens at the furnaces outweighs by between 30 to 40 per cent. the tonnage of coke which would have to be conveyed if that coke had been made at the colliery. Further than that, as a rule, land on which to erect coke ovens near a colliery is cheaper than it is near the blast furnaces, and labour is cheaper. But when ovens are used in which the recovery of the by-products forms a feature, the ovens near the furnaces are, in the Cleveland district at least, considerably less distant from the chemical works in which the by-products are utilised than are those at the collieries; but that is not a matter of paramount importance, for the railway carriage would not amount to much in either case. Besides, the Cleveland pig iron makers seem to favour the old beehive oven rather than the newer forms of oven—at least they consider the coke better. The experiment made by Sir B. Samuelson and Company is, however, with the patent ovens, and, as is stated above, the firm have reason to be satisfied with the result of their enterprise.

### Mineral Production of the United States in 1897.

*R. P. Rothwell. Engineering and Mining Journal, May* 25, 1898, 637.

| No. | Products. | Customary Measures. | 1896. Quantity. Customary Measures. | 1896. Quantity. Metric Tons. | 1896. Value at Place of Production. Totals. | 1896. Value at Place of Production. Per M. Ton. | 1897. Quantity. Customary Measures. | 1897. Quantity. Metric Tons. | 1897. Value at Place of Production. Totals. | 1897. Value at Place of Production. Per M. Ton. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Non-Metallic. | | | | | | | | | |
| | Abrasives— | | | | Dols. | Dols. | | | Dols | Dols. |
| 1 | Carborundum | Sh. T. | 595 | 539 | 365,612 | j 0·68 | 621 | 563 | 153,312 | j 0·27 |
| 2 | Corundum | „ | 250 | 227 | 35,000 | 154·19 | 230 | 209 | 32,200 | 154·07 |
| 3 | Crushed steel | „ | 326 | 296 | 50,624 | 171·00 | 324 | 294 | 51,824 | 176·00 |
| 4 | Diatom. earth | „ | 3,540 | 3,211 | 34,200 | 10·65 | 3,000 | 2,721 | 30,400 | 11·17 |
| 5 | Emery | „ | 1,550 | 1,406 | 108,500 | 77·17 | 1,500 | 1,361 | 105 000 | 77·15 |
| 6 | Garnet | „ | 2,440 | 2,214 | 85,400 | 38·57 | 2,260 | 2,050 | 79,100 | 38·57 |
| 7 | Grindstones | „ | 31,301 | 28,396 | 294,338 | 10·36 | 36,651 | 33,250 | 366,675 | 11·00 |
| 8 | Pumice | „ | .. | .. | .. | .. | 1,700 | 1,542 | 8,500 | 5·51 |
| 9 | Quartz crystal | „ | 6,000 | 5,432 | 16,500 | 3·04 | 6,000 | 5,432 | 16,500 | 3·04 |
| 10 | Tripoli | „ | 1,405 | 1,275 | 4,823 | 3·79 | 1,631 | 1,479 | 5.475 | 3·70 |
| 11 | Whetstones | .. | .. | .. | 105,201 | .. | .. | .. | 80,220 | .. |
| 12 | Alum | Sh. T. | 14,090 | 12.782 | 422,700 | 33·07 | 15,456 | 14.021 | 463,680 | 33·07 |
| 13 | Aluminum sulphate | „ | 42,240 | 38,319 | 1,056,000 | 27·56 | 46,355 | 42,053 | 1,158,875 | 27·56 |
| 14 | Ammonium sulphate | „ | 257 | 233 | 10,280 | .. | 3,111 | 2,822 | 124,440 | .. |
| 15 | Asbestos | „ | 716 | 650 | 12,670 | 19·49 | 770 | 698 | 15,400 | 22·06 |
| 16 | Asphalt | „ | 20,414 | 18,519 | 362,590 | 19·58 | 27,397 | 24,854 | 486,620 | 19·58 |
| 17 | Asphaltic limestone | „ | 5,000 | 4,536 | 55,000 | 12·12 | 2,390 | 2,168 | 11,450 | 5·28 |
| 18 | Bitumin's sandstone | „ | 51,956 | 47,134 | 132,500 | 2·81 | 41,185 | 37,363 | 125,555 | 3·36 |
| 19 | Barytes | „ | 21,900 | 19,867 | 87,600 | 4·41 | 27,316 | 24,781 | 109,264 | 4·41 |

MINERAL PRODUCTION OF THE UNITED STATES IN 1897—*continued*

| No. | Products. | Customary Measures. | 1896. | | | | 1897. | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Quantity. | | Value at Place of Production. | | Quantity. | | Value at Place of Production. | |
| | | | Customary Measures. | Metric Tons. | Totals. | Per M. Ton. | Customary Measures. | Metric Tons. | Totals. | Per M. Tons. |
| | NON-METALLIC—*cont.* | | | | Dols. | Dols. | | | Dols. | Dols. |
| 20 | Bauxite | L. T. | 17,096 | 17,369 | 42,740 | 2·46 | 20,590 | 20,919 | 41,180 | 1·97 |
| 21 | Borax (crude) | Sh. T. | 13,320 | 12,084 | 266,400 | 22·04 | 19,400 | 17,599 | 388,000 | 22·05 |
| 22 | Bromine | Lbs. | 559,285 | 253 | 143,074 | *j* 0·57 | 487,149 | 221 | 136,402 | *j* 0·62 |
| 23 | Calcium carbide | Sh. T. | 860 | 780 | 48,000 | 61·54 | 1,925 | 1,746 | 134,750 | 77·17 |
| 24 | Cement, natural hydraulic | Bbls.*a* | 7,407,311 | 1,007,980 | 4,385,962 | 4·35 | 7,781,377 | 1,058,883 | 4,127,124 | 3·90 |
| 25 | Cement, Portland | ,, *b* | 1,577,283 | 286,181 | 2,502,479 | 8·74 | 2,272,971 | 412,405 | 3,578,839 | .. |
| 26 | Cement, slag | ,, *c* | .. | .. | .. | .. | 40,000 | 6,350 | 60,000 | 9·45 |
| 27 | Chrome ore | L. T. | 702 | 713 | 7,775 | 10·90 | 50 | 51 | 550 | 10·78 |
| 28 | Clay products | Sh. T. | .. | .. | 65,000,000 | .. | .. | .. | *q* 60,000,000 | .. |
| 29 | Coal, anthracite | ,, | 48,133,930 | 42,667,101 | 86,682,719 | 2·03 | 52,645,133 | 47,759,665 | 85,857,717 | 1·79 |
| 30 | Coal, bituminous | ,, | 139,468,659 | 126,525,967 | 113,401,602 | 0·89 | 147,557,599 | 133,864,599 | 120,505,982 | 0·89 |
| 31 | Coal, cannel | ,, | 54,661 | 49,588 | 146,491 | 2·95 | 56,511 | 51,267 | 153,145 | 2·98 |
| 32 | Coke | ,, | 10,369,015 | 9,406,770 | 17,311,823 | 1·84 | 12,742,340 | 11,563,673 | 23,367,879 | 2·02 |
| 33 | Cobalt oxide | Lbs. | 12,825 | *j* 5,817 | 17,314 | *j* 2·98 | 19,300 | *j* 8,754 | 32,810 | *j* 3·75 |
| 34 | Copperas | Sh. T. | 11,170 | 10,133 | 52,662 | 5·19 | 11,924 | 10,818 | 56,565 | 5·23 |
| 35 | Copper sulphate | Lbs. | 48,732,840 | 22,150 | 1,953,225 | 88·18 | 51,012,945 | 23,139 | 2,040,518 | .. |
| 36 | Feldspar | L. T. | 24,907 | 25,305 | 124,251 | 4·91 | 20,900 | 21,234 | 113,773 | 5·36 |
| 37 | Fluorspar | Sh. T. | 6,000 | 5,432 | 48,000 | 8·83 | 9,025 | 8,187 | 74,456 | 9·09 |
| 38 | Fullers earth | ,, | 11,326 | 10,275 | 68,476 | 6·66 | 17,049 | 15,467 | 91,634 | 5·93 |
| 39 | Grahamite(*k*) | ,, | 1,282 | 1,163 | 38,460 | 33·07 | 1,756 | 1,592 | 52,680 | 33·09 |
| 40 | Graphite, crystalline | Lbs. | 405,006 | *j* 183,709 | 18,225 | *j* 0·10 | 993,138 | *j* 450,487 | 44,691 | *j* 0·10 |
| 41 | Graphite, amorphous | Sh. T. | 574 | 520 | 3,850 | 7·40 | 1,200 | 1,090 | 11,400 | 10·45 |
| 42 | Gypsum | ,, | 195,553 | 177,405 | 583,136 | 3·29 | 223,061 | 202,360 | 711,952 | 3·52 |
| 43 | Iron ore | L. T. | 16,000,056 | 16,256,057 | 31,200,889 | 1·93 | 18,316,967 | 18,610,038 | 31,138,844 | 1·67 |
| 44 | Litharge | Sh. T. | 6,500 | 5,897 | 540,300 | 91·57 | 9,900 | 8,981 | 899,100 | 100·11 |
| 45 | Magnesite | ,, | 2,067 | 1,875 | 9,715 | 5·12 | 1,907 | 1,730 | 7,628 | 4·41 |
| 46 | Manganese ore | L. T. | 162,526 | 165,126 | 339,083 | 2·05 | 156,787 | 159,296 | 332,700 | 2·09 |
| 47 | Mica, ground | Sh. T. | 570 | 517 | 9,687 | 18·73 | 2,692 | 2,442 | 38,218 | 15·65 |
| 48 | Mica, sheet | Lbs. | 17,630 | *j* 7,997 | 12,528 | *j* 1·57 | 92,335 | *j* 41,883 | 45,615 | *j* 1·09 |
| 49 | Mineral wool | Sh. T. | 5,853 | 5,309 | 61,614 | 11·60 | 5,667 | 5,141 | 45,494 | 8·81 |
| 50 | Monazite | Lbs. | 17,500 | 8 | 875 | 109·37 | 40,000 | 18 | 2,000 | 111·11 |
| 51 | Natural gas | .. | .. | .. | 10,000,000 | .. | .. | .. | 10,000,000 | .. |
| 52 | Paints, metallic | Sh. T. | 31,865 | 28,908 | 342,167 | 11·15 | 36,293 | 32,925 | 370,594 | 11·26 |
| 53 | Paints, ocher, &c. | ,, | 17,835 | 16,179 | 178,793 | 11·05 | 11,151 | 10,116 | 110,165 | 10·89 |
| 54 | Paints, venetian red | ,, | 5,908 | 5,309 | 93,586 | 17·62 | 4,596 | 4,169 | 55,690 | 10·96 |
| 55 | Paints, white, red lead | ,, | 95,955 | 87,049 | 7,868,310 | 90·39 | 103,235 | 93,654 | 9,291,150 | 99·21 |
| 56 | Paints, zinc oxide | ,, | 15,863 | 14,391 | 1,189,725 | 82·67 | 26,262 | 23,825 | 2,100,960 | 83·18 |
| 57 | Petroleum (crude) | Bbls.*d* | 55,254,795 | 7,730,425 | 65,753,206 | 8·50 | 56,985,643 | 7,972,579 | 44,804,962 | 5·62 |
| 58 | Phosphate rock | L. T. | 937,372 | 952,370 | 2,812,116 | 2·95 | 906,080 | 920,577 | 2,718,240 | 2·95 |
| 59 | Precious stones | .. | .. | .. | 200,000 | .. | .. | .. | 101,000 | .. |
| 60 | Pyrites | L. T. | 109,282 | 111,030 | 292,626 | 2·63 | 128,468 | 130,523 | 379,699 | 2·83 |
| 61 | Salt | Bbls.*e* | 15,707,908 | 1,995,017 | 5,328,250 | 2·67 | 13,153,524 | 1,670,592 | 4,859,364 | 2·91 |
| 62 | Silica, sand, and quartz | L. T. | 720,390 | 731,925 | 1,076,038 | 1·47 | 750,000 | 762,000 | 1,125,000 | 1·48 |
| 63 | Slate, roofing | Squares | 699,100 | .. | 2,260,862 | *n* 3·23 | 895,372 | .. | 2,695,580 | *n* 3·01 |
| 64 | Slate, manufactures | .. | .. | .. | 467,578 | .. | .. | .. | 547,645 | .. |
| 65 | Soapstone | Sh. T | 14,350 | 13,018 | 143,500 | 11·02 | 18,974 | 17,213 | 189,740 | 10·91 |
| 66 | Soda, natural | ,, | 3,000 | 2,722 | 65,000 | 23·88 | 5,000 | 4,536 | 110,000 | 24·25 |
| 67 | Soda, manufactured | M. T. | .. | 157,475 | 3,621,925 | 23·00 | .. | 277,072 | 5,774,656 | 20·84 |
| 68 | Stone for building | .. | .. | .. | 50,599,804 | .. | .. | .. | 30,000,000 | .. |
| 69 | Stone, limestone (flux) | L. T. | 3,794,175 | 3,854,882 | 1,669,437 | 0·43 | 4,247,688 | 4,315,651 | 1,868,983 | 0·43 |
| 70 | Strontium sulphate | Sh. T. | .. | .. | .. | .. | 40 | 36 | 200 | 5·55 |
| 71 | Sulphur | L. T. | 3,800 | 3,861 | 72,200 | 18·70 | 1,690 | 1,717 | 34,814 | 20·27 |
| 72 | Sulphuric acid | Sh. T. | 1,019,501 | 924,885 | 17,331,517 | 18·74 | 1,128,741 | 1,023,987 | 21,446,079 | 19·19 |
| 73 | Talc, common | ,, | 7,098 | 6,439 | 63,585 | 9·87 | 9,563 | 8,675 | 82,795 | 9·54 |
| 74 | Talc, fibrous | ,, | 51,816 | 47,007 | 256,080 | 5·45 | 58,836 | 53,376 | 288,185 | 5·39 |
| 75 | Uranium ore | ,, | .. | .. | .. | .. | 17 | 15 | 9,010 | 600·66 |
| 76 | Zinc ore, exported | L. T. | 2,324 | 2,361 | 47,408 | 20·08 | 9,251 | 9,399 | 211,350 | 22·48 |
| 77 | Estimated product unspecified | .. | .. | .. | 5,000,000 | .. | .. | .. | 5,000,000 | .. |
| | Total non-metals | .. | .. | .. | 484,999,136 | .. | .. | .. | 481,692,497 | .. |
| | METALS. | | | | | | | | | |
| 78 | Aluminium | Lbs. | 1,300,000 | *j* 589,676 | 520,000 | *j* 0·85 | 4,000,000 | *j* 1,814,400 | 1,400,000 | *j* 0·77 |
| 79 | Antimony | ,, | 1,226,000 | 556 | 84,717 | 152·37 | 1,500,000 | 680 | 107,250 | 157·72 |
| 80 | Copper(*m*) | ,, | 479,806,183 | 217,639 | 51,003,897 | 234·35 | 510,190,719 | 231,421 | 56,325,055 | 243·39 |
| 81 | Gold | Ozs.*f* | 2,558,423 | *j* 79,576 | 52,886,209 | *j*664·60 | 2,864,576 | *j* 89,092 | 59,210,795 | 664·60 |
| 82 | Iron, pig | L. T. | 8,623,157 | 8,761,097 | 91,577,610 | 10·45 | 9,652,680 | 9,807,123 | 92,677,312 | 9·45 |
| 83 | Iridium | Ozs. | .. | .. | .. | .. | 20·25 | .. | 606 | .. |
| 84 | Lead, value at New York | Sh. T. | 174,792 | 158,479 | 10,411,643 | 54·07 | 197,718 | 179,368 | 11,784,093 | 65·73 |
| 85 | Nickel | Lbs. | 17,170 | *j* 7,788 | 4,464 | *j* 0·57 | 33,700 | *j* 15,286 | 11,668 | *j* 0·76 |
| 86 | Platinum | Ozs.*f* | 200 | *j* 6·21 | 2,800 | 450·89 | 200 | *j* 6·21 | 3,000 | 482·39 |
| 87 | Quicksilver | Flks.*g* | 29,863 | 1,036 | 1,104,997 | 1066·58 | 26,079 | 965 | 991,002 | 1026·94 |
| 88 | Silver, commercial value | Ozs.*f* | 58,488,810 | *j*1,819,208 | 39,245,992 | *j* 21·58 | 56,457,292 | *j* 1,756,004 | 33,755,815 | 19·22 |
| 89 | Zinc | Sh. T. | 77,637 | 70,432 | 6,117,796 | 86·86 | 100,387 | 91,070 | 8,271,889 | 90·83 |
| | Total metals | .. | .. | .. | 252,959,625 | .. | .. | .. | 264,538,485 | .. |
| | Grand totals | .. | .. | .. | 727,958,761 | .. | .. | .. | 746,230,982 | .. |

(*a*) Barrels of 300 lbs. (*b*) 400 lbs. (*c*) 200 lbs. (*d*) 42 galls. (*e*) 280 lbs. (*f*) Troy ozs. (*g*) Flasks of 76½ lbs. (*h*) Bituminous coal includes brown coal and lignite. The anthracite production is the total for Pennsylvania, Arkansas, and Colorado. (*i*) Estimated (*j*) Kilograms or per kilogram. (*k*) Including bitumen from Texas. (*m*) The value of the copper production is calculated at 0·25 cents per lb. less than the average price of Lake copper at New York. (*n*) Value per square. (*p*) Value per cubic foot. (*q*) This figure is only approximate, and will be revised.

Abbreviations: Sh T., short tons (2,000 lbs.); L. T., long tons (2240 lbs.); M. T., metric tons (2204·6 lbs.) Squares (100 sq. ft., lapped and laid).

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 31st May. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,693,518 | 1,797,374 |
| Chemicals and dyestuffs | 481,832 | 442,359 |
| Oils | 574,728 | 564,129 |
| Raw materials for non-textile industries. | 4,143,172 | 3,609,859 |
| Total value of all imports | 36,346,348 | 37,706,378 |

### Summary of Exports.

| Articles. | Month ending 31st May. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 2,954,991 | 2,733,343 |
| Chemicals and medicines | 743,510 | 667,962 |
| Miscellaneous articles | 2,888,967 | 2,825,138 |
| Total value of all exports | 19,322,146 | 17,891,354 |

### Imports of Metals for Month ending 31st May.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| Copper:— | | | £ | £ |
| Ore ........ Tons | 1,863 | 4,911 | 16,057 | 60,591 |
| Regulus ........ ,, | 7,340 | 7,053 | 161,345 | 187,882 |
| Unwrought ........ ,, | 6,383 | 5,440 | 316,355 | 288,107 |
| Iron:— | | | | |
| Pig ........ ,, | 11,739 | 8,527 | 39,555 | 29,565 |
| Ore ........ ,, | 495,831 | 426,031 | 368,848 | 314,514 |
| Bolt, bar, &c. ........ ,, | 6,229 | 4,632 | 48,138 | 37,043 |
| Steel, unwrought ........ ,, | 3,483 | 3,191 | 35,586 | 18,267 |
| Lead, pig and sheet ,, | 13,216 | 17,273 | 156,700 | 241,072 |
| Pyrites ........ ,, | 52,423 | 62,697 | 80,902 | 106,219 |
| Quicksilver ........ Lb. | 58,678 | 63,260 | 5,598 | 6,235 |
| Silver ore ........ Value £ | .. | .. | 102,513 | 79,460 |
| Tin ........ Cwt. | 34,954 | 37,862 | 104,891 | 121,606 |
| Zinc ........ Tons | 6,296 | 6,788 | 105,633 | 119,730 |
| Other articles ... Value £ | .. | .. | 153,397 | 187,083 |
| Total value of metals | .. | .. | 1,693,518 | 1,797,374 |

### Imports of Chemicals and Dyestuffs for Month ending 31st May.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali ........ Cwt. | 25,343 | 19,623 | 9,888 | 10,394 |
| Bark (tanners', &c.) ,, | 30,687 | 32,732 | 11,291 | 12,040 |
| Brimstone ........ ,, | 40,590 | 20,216 | 9,374 | 4,789 |
| Borax ........ ,, | .. | 96 | .. | 110 |
| Chemicals ...... Value £ | .. | .. | 132,208 | 113,574 |
| Cochineal ........ Cwt. | 286 | 883 | 1,863 | 5,127 |
| Cutch and gambier Tons | 1,241 | 1,940 | 23,830 | 28,784 |
| Dyes:— | | | | |
| Anilin and other Value £ | .. | .. | 39,034 | 49,845 |
| Alizarin ........ ,, | .. | .. | 18,558 | 20,196 |
| Indigo ........ Cwt. | 2,503 | 2,033 | 36,986 | 35,321 |
| Nitrate of potash . ,, | 38,672 | 24,782 | 29,088 | 18,425 |
| Valonia ........ Tons | 4,510 | 1,310 | 45,326 | 13,396 |
| Other articles... Value £ | .. | .. | 124,396 | 130,358 |
| Total value of chemicals | .. | .. | 481,832 | 442,359 |

### Imports of Oils for Month ending 31st May.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut ........ Cwt. | 19,570 | 25,626 | 22,604 | 30,609 |
| Olive ........ Tuns | 2,145 | 1,888 | 70,001 | 62,390 |
| Palm ........ Cwt. | 88,842 | 66,081 | 89,81 | 70,107 |
| Petroleum ........ Gall. | 13,414,437 | 12,785,918 | 244,726 | 227,354 |
| Seed ........ Tons | 2,428 | 2,876 | 48,984 | 52,677 |
| Train, &c. ........ Tuns | 1,246 | 551 | 17,999 | 13,213 |
| Turpentine ........ Cwt. | 1,526 | 16,216 | 1,578 | 19,905 |
| Other articles .. Value £ | .. | .. | 79,027 | 87,874 |
| Total value of oils... | .. | .. | 574,728 | 564,129 |

### Imports of Raw Material for Non-Textile Industries for Month ending 31st May.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 3,742 | 4,909 | 11,465 | 9,540 |
| Bristles ........ Lb. | 359,597 | 280,753 | 49,605 | 31,516 |
| Caoutchouc ........ Cwt. | 30,191 | 37,812 | 330,506 | 416,942 |
| Gum:— | | | | |
| Arabic ........ ,, | 5,659 | 6,650 | 10,056 | 10,526 |
| Lac, &c. ........ ,, | 17,597 | 4,859 | 73,890 | 15,573 |
| Gutta-percha ........ ,, | 3,615 | 7,338 | 41,207 | 68,710 |
| Hides, raw:— | | | | |
| Dry ........ ,, | 54,384 | 44,955 | 135,219 | 119,871 |
| Wet ........ ,, | 43,113 | 46,178 | 94,731 | 95,184 |
| Ivory ........ ,, | 629 | 723 | 21,916 | 27,224 |
| Manure:— | | | | |
| Guano ........ Tons | 2,112 | 1,538 | 13,589 | 10,324 |
| Bones ........ ,, | 3,899 | 1,496 | 14,460 | 18,333 |
| Nitrate of soda ........ ,, | 19,393 | 17,147 | 145,209 | 126,535 |
| Phosphate of lime ,, | 26,947 | 22,788 | 39,476 | 31,967 |
| Paraffin ........ Cwt. | 49,071 | 52,104 | 45,167 | 43,720 |
| Linen rags ........ Tons | 2,912 | 1,667 | 26,232 | 15,690 |
| Esparto ........ ,, | 19,356 | 9,620 | 75,914 | 37,832 |
| Pulp of wood:— | | | | |
| Chemical ........ ,, | 34,318 | 15,536 | 181,689 | 98,175 |
| Mechanical ........ ,, | | 14,294 | | 40,102 |
| Rosin ........ Cwt. | 74,516 | 126,796 | 17,904 | 28,389 |
| Tallow and stearin ,, | 175,281 | 173,542 | 163,502 | 177,962 |
| Tar ........ Barrels | 5,635 | 6,798 | ,696 | 3,673 |
| Wood:— | | | | |
| Hewn ........ Loads | 260,395 | 146,026 | 519,725 | 322,459 |
| Sawn ........ ,, | 441,780 | 312,010 | 1,109,236 | 790,102 |
| Staves ........ ,, | 7,053 | 7,209 | 38,815 | 41,794 |
| Mahogany ........ Tons | 4,681 | 4,982 | 43,055 | 45,993 |
| Other articles .... Value £ | .. | .. | 937,908 | 981,419 |
| Total value ........ | .. | .. | 4,143,172 | 3,609,859 |

Besides the above, drugs to the value of 81,807*l*. were imported, as against 67,787*l*. in May 1897.

### Exports of Metals (other than Machinery) for Month ending 31st May.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass ........ Cwt. | 8,280 | 7,770 | 37,952 | 35,074 |
| Copper:— | | | | |
| Unwrought ........ ,, | 39,308 | 46,004 | 101,651 | 124,453 |
| Wrought ........ ,, | 25,568 | 24,203 | 77,171 | 75,810 |
| Mixed metal ........ ,, | 15,624 | 14,066 | 37,481 | 34,264 |
| Hardware and cutlery. Value £ | .. | .. | 179,593 | 157,246 |
| Implements ........ ,, | .. | .. | 110,590 | 103,264 |
| Iron and steel ... Tons | 343,515 | 293,641 | 2,125,265 | 1,914,913 |
| Lead ........ ,, | 6,597 | 4,873 | 82,487 | 66,759 |
| Plated wares ... Value £ | .. | .. | 27,125 | 28,811 |
| Telegraph wires . ,, | .. | .. | 61,226 | 41,944 |
| Tin ........ Cwt. | 11,405 | 14,719 | 36,685 | 50,010 |
| Zinc ........ ,, | 11,723 | 17,112 | 9,223 | 14,389 |
| Other articles .. Value £ | .. | .. | 68,583 | 86,390 |
| Total value ........ | .. | .. | 2,954,991 | 2,733,343 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST MAY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 501,695 | 321,355 | 128,264 | 87,030 |
| Bleaching materials „ | 91,246 | 91,074 | 29,463 | 24,388 |
| Sulphate of copper Tons | 8,086 | 6,592 | 136,653 | 106,222 |
| Chemical manures „ | 23,670 | 25,980 | 134,362 | 131,660 |
| Medicines....... Value £ | .. | .. | 92,710 | 88,508 |
| Other articles... „ | .. | .. | 222,058 | 230,154 |
| Total value .......... | .. | .. | 743,510 | 667,962 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST MAY.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 512,800 | 330,500 | 12,650 | 6,012 |
| Military stores.. Value £ | .. | .. | 127,081 | 233,388 |
| Candles.......... Lb. | 2,036,100 | 1,649,900 | 28,196 | 23,142 |
| Caoutchouc ..... Value £ | .. | .. | 94,255 | 113,560 |
| Cement.......... Tons | 41,038 | 25,955 | 67,380 | 48,196 |
| Products of coal Value £ | .. | .. | 163,465 | 141,592 |
| Earthenware ... „ | .. | .. | 167,298 | 130,152 |
| Stoneware ...... „ | .. | .. | 14,700 | 17,779 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 146,513 | 175,158 | 7,588 | 7,575 |
| Flint........... Cwt. | 6,851 | 8,399 | 15,238 | 18,058 |
| Bottles......... „ | 69,711 | 67,594 | 32,233 | 31,880 |
| Other kinds.... „ | 20,628 | 21,773 | 15,247 | 17,824 |
| Leather:— | | | | |
| Unwrought .... „ | 12,468 | 13,120 | 114,567 | 113,754 |
| Wrought ..... Value £ | .. | .. | 31,058 | 28,492 |
| Seed oil.......... Tons | 5,490 | 4,046 | 91,261 | 69,019 |
| Floorcloth ..... Sq. Yds. | 2,376,800 | 2,095,400 | 96,404 | 87,635 |
| Painters' materials Val. £ | .. | .. | 146,875 | 146,120 |
| Paper ........... Cwt. | 89,424 | 83,800 | 130,383 | 124,781 |
| Rags............ Tons | 4,451 | 5,526 | 26,589 | 27,562 |
| Soap............. Cwt. | 65,487 | 82,306 | 70,563 | 73,715 |
| Total value .......... | .. | .. | 2,888,967 | 2,825,138 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

11,135. W. F. Berner. Improved means for heating furnaces. May 16.

11,243. F. T. Andrews and H. F. Wilkinson. Improvements in boiler cleaners. May 17.

11,364. P. Reuss. Improvements in centrifugal machines. Complete Specification. May 18.

11,392. J. H. Riley and F. Riley. Improvements in rotary pumps for corrosive liquids. May 19.

11,481. W. Gimblett and T. Williams. An annealing pot. May 20.

11,544. S. Dreyfus and The Clayton Aniline Company, Ltd. Improvements in and apparatus for use in the concentration of sulphuric acid or the like. May 23.

11,555. W. B. Johnson.—From The Carnegie Steel Company, Ltd., United States. Improvements in blast furnace feeding apparatus. May 23.

11,826. C. Butters. Improvements in the manufacture of of discharge doors for vats. Complete Specification. May 24.

11,987. W. A. E. Crombie. Improvements in apparatus for evaporating or drying. May 26.

12,181. L. C. Reese. Improvements in distilling apparatus. May 28.

12,273. E. Kain. Improvements in or relating to crucible furnaces. May 31.

12,396. W. Weir and J. R. Richmond. Improvements in and connected with apparatus for removing oil and fatty matters from feed water for steam boilers. June 2.

12,501. I. Qurin. Tank waggon for fluid gases, especially for carbonic acid. June 3.

12,543. T. P. Richards. Improvements in drying apparatus. June 4.

12,572. H. Lowenfeld. Improvements in or relating to vessels employed to contain gaseous liquids. June 4.

12,750. H. H. Lake.—From the firm of N. O. fu Alessandro, Italy. Improvements in evaporating and distilling apparatus for use on board ship. Complete Specification. June 7.

13,108. T. Molinard. Improvements in refrigerators. Complete Specification. June 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

4744. R. Moodie. Apparatus for washing or leaching granular or powdered substances and separating the same from the liquids used. May 25.

11,872. J. Hill. Filter presses. May 25.

15,105. S. C. Davidson. Apparatus for drying or partially drying or otherwise treating tea leaf, tobacco leaf, coffee grain, malt, wool, and other substances. May 25.

15,384. W. J. Baker. Apparatus for purifying water and steam, and for heating and softening water. June 8.

17,475. F. Abbott and Lennard, Ltd., and L. Gaster. Apparatus for use in subjecting air or other gases or liquids or solids to the action of air, gases, or liquids; applicable also for mixing, cooling, heating, and other analogous operations. June 8.

17,714. J. Buchanan, jun. Appliances for discharging coke and like ovens. June 15.

19,718. T. R. Berkley. Refuse-consuming furnaces. June 1.

1898.

4518. B. H. Scheibler. Apparatus for carbonising rags. June 8.

5325. La Société Anonyme Du Temple and H. Brillié. Boilers, evaporators, or heaters. June 15.

6431. E. Schliemann and E. von Boyen. Process of and apparatus for continuous extraction. June 8.

8069. A. T. Welch. Drying apparatus. June 15.

9143. W. Pilkington. Metallic cylinders or bottles for containing gas under high pressure, and for other purposes. May 25.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

11,107. W. Smith. Improvements in the manufacture of peat fuel. May 16.

11,161. W. W. Strode and G. H. White. Improvements in or relating to the production of acetylene gas. May 16.

11,248. R. T. Nicholson. Improvements in connection with the generation of acetylene gas. May 17.

11,261. F. A. Keiffer. Improvements in acetylene gas generators. Complete Specification. Filed 17 May. Date applied for, 11 Dec. 1897, being date of application in France.

11,283. M. Otto. Apparatus for the production of ozone and other purposes. May 17.

11,285. A. E. E Cantley and E. Springborn. An improved manufacture of peat fuel. May 17.

11,297. S. Chandler, junior, and J. Chandler. Improvements in incandescent gas burners. May 18.

11,367. J. Shackleton and A. Ross. Improvements in apparatus for generating and purifying gas. May 18.

11,377. T. R. Fowler. Improvements in apparatus for the manufacture of acetylene or the like. May 19.

11,389 J. White. Improvements in apparatus for generating, purifying, and storing acetylene gas. May 19.

11,422. G. Hope. Improvements in or relating to generators for acetylene gas. May 19.

11,565. H. Ely. Improvements in acetylene gas generators. May 23.

11,566. H. Ely. Improvements in apparatus for the production and storage of acetylene gas. May 23.

11,619. T. Steenberg and E. Steenberg. An improved method or process and apparatus for the treatment of peat. May 23.

11,676. E. Evans. Improved apparatus for washing and purifying gas. May 23.

11,683. T. T. Cowell. Improvements in incandescence burners. May 23.

11,781. E. Francetti-Schieroni. Improvements in apparatus for the manufacture of acetylene gas. May 24.

11,799. E. Rischgitz. Improvements relating to the treatment of peat. May 24.

11,802. W. B. Hartridge. Improvements in apparatus employed in the manufacture of fuel blocks. May 24.

11,874. F. W. Golby.—From E. Caplat. Improvements in apparatus for producing acetylene. May 25.

11,910. J. Bartlett. An improved acetylene gas generator. Complete Specification. May 25.

11,917. A. Beard and A. E. Beardmore. Improvements in the utilisation of gas for the combined purpose of heating low-temperature furnaces and the like, and producing a high temperature for heating steam boilers or other purpose. May 26.

11,949. R. Cremer.—From H. L. J. Dorenfeld, Germany. Preparation of or method for employing the spent liquor from Esparto-straw wood pulp and similar mills as fuel. May 26.

11,964. C. Bellé.—From P. Dreske, Germany. Improvements in apparatus for generating acetylene. Complete Specification. May 26.

11,970. British Pure Acetylene Gas Syndicate, Limited, and T. Keene. Improvements in acetylene gas generators. May 26.

11,971. R. Cremer.—From L. J. Dorenfeld, Germany. Improvements in apparatus for or in the method of utilising the spent liquor from Esparto-straw wood pulp and similar mills as fuel. May 26.

12,022. J. K. Rigby. Improvements in or relating to apparatus used in the generation of acetylene. May 26.

12,061. J. E. Scarth and W. K. Ferno. Improvements in or relating to lamps for generating and burning acetylene gas. May 27.

12,173. R. Williams. Improvement in acetylene generators. May 28.

12,182. P. M. Justice.—From V. Sard , Italy. Improvements in apparatus for the manufacture of acetylene gas. May 28.

12,198. A. J. Mottlau. Improvements in apparatus for the continuous production of acetylene gas. May 28.

12,199. M. Graham. Improvements in and apparatus for charging inclined gas retorts. May 31.

12,210. T. Thorp. Improvements in and connected with apparatus for the production of acetylene. May 31.

12,233. G. Boulard. An improved apparatus for the manufacture of acetylene gas. May 31.

12,250. W. P. Thompson.—From The Geselschaft für Heiz- und Beleuchtungswesen, Germany. An improved acetylene generator. May 31.

12,251. J. Gööz. Improvements in or relating to smokeless firing for boiler furnaces and the like. May 31.

12,295. E. Evans. Improved combined apparatus for generating and storing acetylene gas. June 1.

12,356. W. H. A. Sieverts. An improved method of and apparatus for forming incandescent bodies for gas lighting. Complete Specification. June 1.

12,401. J. E. Scarth. Improvements in or relating to apparatus for the generation of acetylene gas. June 2.

12,469. E. L. Levetus. Improvements in or relating to acetylene generators. June 3.

12,491. A. J. Boult.—From Societe Italiana Pel Carburo di Calcio Acetilene ed altri gas, Italy. Improvements in or relating to acetylene gas generators. Complete Specification. June 3.

12,500. W. Sassmann. Acetylene gas generator. June 3.

12,510. O. Ernst and A. Philips. Improvements in apparatus for producing acetylene gas. Complete Specification. June 3.

12,530. A. Hallet. A new or improved acetylene gas generator. June 4.

12,586 C. D. Abel.—From Société Anonyme Continentale Pour la Fabrication des Compteurs a Gaz et Autres Appareils, France. Improvements in apparatus for feeding gas retorts. June 4.

12,698. A. J. Boult.—From J. Desforges, France. Improvements in or relating to acetylene gas generators. June 7.

12,701. B. Fröhlich. Improvements in briquettes or fuel blocks. Complete Specification. June 7.

13,029. F. A. Jackson. Improvements in and relating to apparatus for generating acetylene gas. June 10.

13,070. Sir C. S. Forbes, Bart. Improved apparatus for the generation and supply of acetylene gas. June 11.

13,135. G. Hilgenstock. Apparatus for opening and closing the doors of coke ovens. Complete Specification. June 11.

13,149. T. C. Lecca. Improvements in apparatus for generating acetylene gas. June 11.

### Complete Specifications Accepted.

### 1897.

8485. E. Bouchaud-Praceiq. Apparatus for carburetting air. June 8.

12,263. A. Preston. Apparatus for the manufacture of acetylene gas. May 25.

13,905. E. Soxhlet, F. V. Bergh, A. Borremans, and V. Borremans. Process of obtaining lighting, heating, and motive power, and apparatus therefor. June 8.

14,505. H. H. Lake.—From the firm Piatti and Co., Italy. Manufacture of illuminating gas. June 1.

14,535. T. Watkins. Oil or spirit vaporising apparatus June 1.

14,700. H. R. Bean and H. Ringwood. Purifier and enricher for acetylene gas. May 25.

14,742. T. R. Fowler. Apparatus for the manufacture of acetylene gas. June 8.

15,125. J. W. Scarth. Apparatus for the generation of acetylene gas, and the like purposes. May 25.

15,350. A. Zendroni. Manufacture of artificial fuel or briquettes. June 1.

16,344. J. Ackroyd and B. Ackroyd. Apparatus for making acetylene gas. June 8.

17,327. W. B. Hartridge. Fuel blocks. May 25,

25,750. J. Zimmerman. Gas-producing materials. May 25.

25,870. A. H. Barthez. Apparatus for the automatic production of acetylene gas. June 8.

27,416. A. A. Dickson. Process and apparatus for forming cold peat into blocks for fuel. June 8.

28,439. V. Sockeel, A. Luaby, C. H. Drisse, and —Sneck. Apparatus for the production of acetylene gas, also called "Lumière Soleil." May 25.

28,798. B. Richard. Apparatus for generating and purifying acetylene. June 1.

### 1898.

1584. H. de Thiersant and W. A. Coulson. Apparatus for the production of acetylene gas. June 15.

7070. E. Edwards.—From L. Steiner, Roumania. Acetylene gas apparatus. June 1.

## IV.—COLOURING MATTERS AND DYES.

### Applications.

11,259. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the manufacture of mixtures of ortho-chlor- and ortho-nitro-benzylalcohol, or mixtures of the ethers of these two substituted alcohols, and the separation of such mixtures. May 17.

11,260. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the manufacture of ortho-chlor-benzaldehyde and its separation from ortho-nitro-benzaldehyde. May 17.

11,437. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of azodyestuffs from picramic acid. May 19.

11,585. J. E. Bedford and C. S. Bedford. Improvements in the treatment of logwood colouring matter. May 23.

11,643. O Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of penta- and hexa-oxyanthraquinonedisulphonic acids. May 23.

11,736. Read Holliday and Sons, Ltd., J. Turner, and H. Dean. Improvements in the manufacture of aniline and naphthylamine derivatives and colouring matters therefrom. May 24.

12,085. Read Holliday and Sons, Ltd., J. Turner, and H. Dean. Improvements in the manufacture of intermediate products and colouring matters therefrom. May 27.

12,118. I. Levinstein, R. Herz, and Levinstein, Ltd. The manufacture of a new sulphonic acid of the naphthalene series and of colouring matters therefrom. May 28.

12,119. I. Levinstein, R. Herz, and Levinstein, Ltd. Manufacture of sulphonic acids of the naphthalene series and of colouring matters therefrom. May 28.

12,427. A. G. Green, A. R. Wahl, and The Clayton Aniline Company, Ltd. Improvements in the production of fast colouring matters of the triphenylmethane series. June 2.

12,466. A. Ashworth and J. Bürger. Improvements in the production of colouring matters. June 3.

12,596. K. Elbs and A. Wülfing. *See* Class XI.

12,679. O. Imray.—From The Society of Chemical Industry in Basle, Switzerland. Manufacture of naphthacetaldi-sulphonic acids and of new colouring matters derived therefrom. Complete Specification. June 6.

12,937. O. Imray.—From The Basle Chemical Works, Bindschedler, Switzerland. Manufacture of new dyestuff from 1:8 dinitronaphthalene-3-sulphonic acid. June 9.

12,938. O. Imray.—From The Basle Chemical Works Bindschedler, Switzerland. Manufacture of 1:4-amido-chloronapthalene-8-sulphonic acid. June 9.

### Complete Specifications Accepted.

### 1897.

14,043. J. E. Bedford and C. S. Bedford. Treatment of logwood colouring matter. May 25.

15,353. S. Pitt.—From L. Cassella and Co., Germany. Producing diazo salts in a durable form. June 1.

15,377. W. H. Claus, A. Rée, and L. Marchlewski. New polyazo colouring matters. June 15.

15,478. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture of dyestuffs. June 1.

15,479. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture of colouring matters. June 15.

15,890. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning. Manufacture of ortho- and para-nitrobenzylaniline sulphonic acids and their homologues. June 1.

16,483. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of dyestuffs of the triphenylmethane series fast to alkali. June 1.

16,495. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Production of blue mordant-dyeing colouring matters from dinitro-anthraquinones. June 15.

16,713. M. Prud'homme. Manufacture of colouring matters having acid action. June 1.

17,328. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of azo dyestuffs fast to washing light. June 15.

18,002. J. Imray. — From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of green and blue-green colouring matters from triphenylmethane. June 15.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

11,419. E. Ungnad. Improvements relating to the treatment of fibres, yarns, and fabrics to imitate silk. May 19.

11,727. J. M. Collins. Improvements in and connected with frames for use when mercerising yarns, said frames being also adapted for use when dyeing silk or other yarns. May 24.

11,728. J. M. Collins. Improvements in machinery for dyeing slubbing or wool and other yarns, said machinery being also adapted for soaping yarns. May 24.

11,841. E. Sykes and D. Redfearn. New or improved apparatus for and means to be employed in preparing or cuttling yarn in the warp for dyeing, scouring, or bleaching. May 25.

12,027. W. N. Packer. Method of and means for defibrating ramie. May 26.

12,066. J. D. Tomlinson and J. Porter. Improvements in or applicable to machines for winding, gassing, and the like operations on yarn or threads. May 27.

12,161. A. J. Boult.—From F. A. Jone, Germany. An improved fabric or material. May 28.

12,214. A. Debuisne. An improved process and apparatus for clearing wool. May 31.

12,484. C. Weygang. Improvements in the treatment of fibrous materials. June 3.

12,669. L. S. van Westrum. An improved process for mercerising cotton. Complete Specification. June 6.

12,837. J. L. Bottomley and S. C. R. Gauntlett. The manufacture of an improved yarn. June 8.

12,867. E. Müller. A process for the manufacture of fabrics from nitrated yarns, chiefly designed for filtering purposes. Complete Specification. June 8.

13,017. W. G. Heys.—From H. David, France. Improvement in and connected with the production of lustrous effects upon vegetable textile materials. Complete Specification. June 10.

13,124. O. Venter. Improvements in apparatus for treating yarns or threads with liquids. June 11.

### Complete Specifications Accepted.

### 1897.

13,274. E. Neuweiler. Manufacture of yarns. May 25.

15,381. G. B. Garlach, of the firm of Kettling and Braun. Process of and means for finishing or dressing textile fabrics and other goods. June 8.

17,302. J. Wetter.—From Herbelier and Co., Switzerland. Treatment of cotton yarn and other vegetable textile material for imparting to it a silk-like gloss. May 25.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### APPLICATIONS.

11,436. C. D. Abel.—From The Actien-Gesellschaft für Anilin Fabrikation, Germany. New process of dyeing. May 19.

11,601. C. L. Horack. Improvements in methods of and apparatus for colouring or printing yarns. Complete Specification. Filed May 23. Date applied for, October 29, 1897, being date of application in the United States.

11,653. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in printing with indigo. May 23.

11,951. H. Gotliffe. Improvements in means for printing waterproof fabrics. May 26.

12,252. H. C. Meyer. An improved process for bleaching and dyeing glazed or smooth cane. May 31.

12,379. W. P. Thompson.—From B. Cohnen, Germany. Improvements in machines for mordanting, sizing, or mercerising hanks of yarn. Complete Specification. June 2.

12,483. W. Shaw. Improvements in method of and apparatus for colouring yarns. Complete Specification. June 3.

12,635. S. Pitt.—From L. Cassella and Co., Germany. Improvement in dyeing in dark blue shades. June 6.

12,764. W. P. Thompson.—From H. C. Mayer, jun., Germany. An improved process for bleaching and dyeing glazed or smooth cane. June 7.

12,900. J. J. Jackson. Improvements in the construction of kiers for boiling or bleaching goods. June 9.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

10,213. E. Bentz. Production of white and coloured patterns on noilire black grounds in calico printing. June 1.

14,639. W. Woodcock, J. Longthorpe, and W. Dargue. Machinery for dyeing, sizing, and washing hanks of yarn and slubbing. June 1.

1898.

3775. F. F. Stimpson. Method of printing on fabrics. May 25.

5475. J. Bundgens and L. Bündgens. Dyeing vats. May 25.

8029. R. C. W. A. Sieverts. Processes for bleaching or dyeing Spanish cane (*calamus rotang*), bamboo, and other plants which have a siliceous coating. June 1.

## VII.—ACIDS, ALKALIS, AND SALTS.

### APPLICATIONS.

11,137. J. Turner and Co., O. Rosenheim, and P. Schidrowitz. Improvements in the production of tannic acid. May 16.

11,541. S. Dreyfus and the Clayton Aniline Company, Ltd. *See* Class I.

11,564. H. Ely. Improvements in storage and transport vessels for calcium carbide. May 23.

11,633. J. Lagache. A process for the manufacture of sulphate of copper. Complete Specification. May 23.

11,650. G. W. Johnson.—From Chemische Fabrik Griesheim, Germany. Improvements in the purification of caustic alkalis. May 23.

11,780. F. G. Welch. Improvements in the manufacture of nitrate of soda, potash, and other soluble salts, and in apparatus employed therein. May 24.

12,075. H. Precht. Improvements in packing caustic alkalis. May 27.

13,134. Electric Reduction Company, Ltd.—From W. T. Gibbs, Canada. An improvement in the treatment of mineral phosphates. June 11.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

15,976. E. M. Polet. Process relating to the treatment and production of phosphates. May 25.

17,476. R. H. Davidson and The United Alkali Company, Ltd. Apparatus for the manufacture of flowers of sulphur. June 15.

18,417. O. Luupe. Process for the recovery of perchloride of iron from the solutions of perchloride of iron used for etching purposes. June 15.

29,384. F. J. Bergmann. Process for the production of calcium carbide in blast furnaces. June 1.

1898.

7,574. W. F. Thompson.—From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuchel, United States. Method and apparatus for producing calcium carbide. June 1.

8554. E. Tooth. Manufacture of carbide of calcium. June 15.

9730. F. Henkel. Process for the manufacture of solid but easily soluble alkaline silicate. June 15.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### APPLICATIONS.

12,014. W. M. Edge, W. S. M. Edge, and S. W. Dean. An improved process for reproducing engravings upon the surface of pottery and other articles. Complete Specification. May 26.

12,078. P. Wood, Ltd., and H. P. Nash. Certain improvements in continuous kilns for burning bricks, tiles, sanitary ware, and other clay goods, and for salt glazing. May 27.

12,344. J. Sossick. Improvements in the manufacture of moulded decorative tiles, slabs, panels, or blocks. June 1.

12,746. W. Drake. Improvements in the manufacture of glass bottles and the like. June 7.

12,852. W. van den Berg and T. Haass. An improved process for the manufacture of water- and acid-proof non-conducting articles of clay. Complete Specification. June 8.

12,885. G. Pelmear, W. H. Cummings, and W. R. Meggeson. A substitute for glass. June 9.

### COMPLETE SPECIFICATION ACCEPTED.

1897.

16,062. E. F. W. Hirsch. Glass-melting plant. June 15.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

11,113. J. R. Cooper. Improved decorative process in plaster or cement. May 16.

11,170. A. Taylor. Improvements in and in connection with hydraulic presses, and in the manufacture thereby of artificial stone slabs or the like. May 16.

11,960. C. J. Lomax. Improved method of making and strengthening cement concrete for pavements. May 26.

12,902. J. Goodwin, jun. An improved composition for covering the roofs and (or) walls of buildings. Complete Specification. June 9.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

16,661. H. H. Lake.—From J. Seidler and A. J. C. Graf, Germany. Manufacture of artificial blocks or plates for building and other purposes. June 1.

1898.

7105. G. F. Lebioda. Apparatus for impregnating and dyeing wood. May 25.

## X.—METALLURGY, MINING, Etc.

### Applications.

11,180. J. Colquhoun. Improvements in treating copper matte. Complete Specification. May 17.

11,191. J. Dick. Improvements in and relating to the obtaining or recovering of gold or gold amalgams. May 17.

11,265. W. P. Thompson.—From W. J. Wilder, United States. An improved process and preparation for coating iron or steel. Complete Specification. May 17.

11,507. C. T. J. Vautin. An improved process for the reduction of argentiferous or non-argentiferous complex sulphide ores of lead and zinc. May 20.

11,556. J. Hall. Improvements in the manufacture of mild steel. May 23.

11,689. W. M. Theobald. Process of finishing sheet iron or steel plates. Complete Specification. May 23.

11,801. F. Siemens. Improvements in protecting iron and steel from oxidation during heating. May 24.

11,823. F. Ellerhausen. Improvements in condensing and collecting fumes obtained in the treatment of ores. May 24.

11,895. J. Armstrong. Improvements in the treatment of sulphide complex or other ores or mattes, and the recovery of metals therefrom. May 25.

11,940. R. Threlfall. Improved method of treating flue dust obtained from sulphide ores. May 26.

11,992. W. P. Thompson.—From G. Behr and T. Walffisch, Germany. An improved process for hardening steel. Complete Specification. May 26.

12,003. W. P. Thompson.—From F. H. Cartland, France. An improved furnace for melting and retaining in a molten state metal, alloys of metal, and other materials which liquefy when heated. May 26.

12,082. J. A. Mays. Process of and apparatus for reducing and separating metals from lead and zinc oxides. May 27.

12,163. R. A. Poore.—From P. Poore, South African Republic. Improvements in the cyanide process for the obtaining of gold and other precious metals, and in apparatus therefor. May 23.

12,249. W. Morris. Improvements in or connected with machinery or apparatus employed in the manufacture of tin, terne, or other plates. May 31.

12,287. P. Rogers and J. Player. Improvements in machinery or apparatus for coating metal plates or sheets with tin, lead, or other metals or alloys. Complete Specification. May 31.

12,436. W. Skaife. Improvements in the separation of soft spelter from hard spelter produced in the process of galvanising iron. June 2.

12,481. C. Heap and T. Oddy. Improvements in the production of aluminium and aluminium alloys, and in furnaces to be used for such production and for other purposes. June 3.

12,503. G. Schwahn. Improvements in reducing aluminium. June 3.

12,651. J. Gordon. Improvements in processes of dephosphorising steel and other metals. Filed June 6. Date applied for Nov. 12, 1897, being date of application in United States.

12,838. F. E. Elmore. A process and apparatus for separating the metallic from the rocky constituents of ores. June 8.

12,946. W. Baines, jun. Improvements in or relating to armour-plating or the like. June 9.

13,123. L. Budzinski and B. Schonwaloff. Improvements relating to the treatment of steel. Complete Specification. June 11.

### Complete Specifications Accepted.

1897.

10,829. J. Swinburne. Treatment of sulphide ores. June 8.

10,829A. J. Swinburne. Treatment of sulphide ores. June 8.

14,392. C. Wetherwax. Method of and means for the preliminary treatment of gold and silver ores. June 15.

15,115. S. Alley.—From J. Campbell and J. W. H. James, United States. Furnaces for treating ores containing precious metals, and other ores. May 25.

15,203. S. Alley. Furnaces for treating ores containing precious metals, and other ores. May 25.

19,338. J. Riley. Utilising slag or cinder from steel-melting furnaces. June 15.

23,278. J. Bedford and J. Ashton. Production of compound steel bars for making cutting tools and the like. June 8.

26,063. A. Reynolds. Manufacture of crucible steel. May 25.

29,751. G. F. Redfern.—From P. Argall, United States. Roasting and drying ores, and apparatus therefor. June 15.

30,463. J. T. Wainwright. Art of manufacturing steel armour plate. June 15.

1898.

2567. G. Hammond and T. Flint, jun. Solder and flux for aluminium. June 1.

3810. W. P. Thompson.—From B. Talbot, United States. Process of refining metals. June 1.

7008. O. Imray.—From The Pennsylvania Salt Manufacturing Company, United States. Smelting plant June 1.

8245. F. Schadeloock. Manufacture of crucible stee and its alloys. May 25.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

11,155. F. King. Improvements in secondary batteries or accumulators. May 16.

11,340. E. A. Jahncke. Improvements in or connected with electric batteries. May 18.

11,570. G. Bell and G. W. Bell. Improvements in or connected with electrolytic cells. May 23.

11,604. E. Stassano. Improvements in and connected with the electro-metallurgic production of iron, steel, and their alloys with chromium, tungsten, nickel, manganese, and the like. Complete Specification. May 23.

11,640. F. Heimel. Improvements in accumulators or secondary batteries and apparatus for their manufacture. May 23.

11,678. W. Dunn and W. J. Twining. Improved apparatus for use in the electro-deposition of metals. May 23.

11,858. F. King. Improvements in the manufacture of elements or plates for secondary batteries or electric accumulators. May 25.

12,019. H. Tobler and J. H. Graeber. Manufacture of active material for accumulator batteries. Complete Specification. May 26.

12,313. F. King. Improvements in secondary batteries or electric accumulators. June 1.

12,321. E. Edwards.—From P. A. Emanuel, United States. Improvements in electric batteries. Complete Specification. June 1.

12,322. E. Edwards.—From P. A. Emanuel, United States. Improvements in electric batteries. Complete Specification. June 1.

12,323. E. Edwards.—From P. A. Emanuel, United States. Improvements in electric batteries. Complete Specification. June 1.

12,596. K. Elbs and A. Wülfing. A process for the electrolytic reduction of nitro-benzol, nitro-anisol, nitro-aniline, and their homologues to azo and hydrazo compounds. June 4.

12,754. F. E. Elmore.—From J. O. S. Elmore, India. Improvements in apparatus for the generation and electrolytic application of electric currents. June 7.

12,765. W. P. Thompson.—From M. Hazard-Flamand, France. Improvements in or relating to the electrolysis of liquids and apparatus therefor. June 7.

12,825. J. B. de Alzugaray. Improvements in obtaining metals from their fused ores, oxides, and salts by electrical action. June 8.

13,121. H. W. Cobb. Improvements in electrical accumulators. June 11.

### Complete Specifications Accepted.

1897.

9803. E. E. de Rufz de Lavison. Voltaic battery having its depolarisation effected by heat. March 16.

12,665. H. Tee. Combined generation and application of electricity supply and the manufacture of salt. May 25.

13,735. W. Rowbotham. Primary batteries. June 1.

14,022. R. Memmo. Electric furnace for the manufacture of carbide of calcium with continuous operation, and with water covering for the gas. June 15.

15,850. J. B. Brooks and J. Holt. Certain apparatus to be employed in connection with the electro-deposition of metals. June 1.

15,879. W. J. S. Barber-Starkey. Plates for secondary batteries. May 25.

15,903. A. D. Seton and J. L. Dobell. Electric batteries. June 8.

16,797. G. J. Erlacher and M. A. Besro. Controlling apparatus for accumulator batteries. June 8.

16,816. H. Leitner. Construction of secondary batteries. June 15.

17,158. W. Rowbotham. Electric batteries. June 1.

1898.

3368. D. d'Arbel. Secondary batteries. June 15.

4910. C. Koenig. Primary batteries. June 1.

7193. A. J. Boult.—From J. Cerpaux and A. Wilbaux, Belgium. Primary electrical batteries. June 1.

7575. W. P. Thompson.—From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuchel, United States. Electric furnaces for manufacturing calcium carbide, &c. June 1.

7577. N. B. Stubblefield. Electrical batteries. May 25.

9637. J. W. Richards and C. W. Roepper. Process for the production of chemical compounds by electrolysis. June 15.

9638. J. W. Richards and C. W. Roepper. Process for manufacturing metallic sulphides electrolytically. June 15.

## XII.—FATS, OILS, and SOAP.

### Applications.

11,509. N. Schwartz and J. Rayman. Improved composition for cleaning wall papers and distempered surfaces. May 20.

12,494. E. L. Blosse.—From G. I. de Richardon, Belgium. Improvements in the manufacture of washing and cleansing compounds. June 3.

12,508. S. Rosenblum and The Commercial Ozone Syndicate, Ltd. Improvements in the production of chemical compounds from a certain oil or oils or the fatty acids of same and the employment of such compounds in the manufacture of drying oils, varnishes, enamels, lacquers, antifouling compositions and the like. Complete Specification. June 3.

12,990. J. Stork. Improvements in the method of extracting oil and grease from woollen and mixed woollen yarns. June 10.

13,098. L. Rivière. A new or improved process of saponification of fatty substances by bases in the native state. June 11.

### Complete Specification Accepted.

1898.

7932. W. P. Thompson.—From E. Sieglin, Germany. Manufacture of soap powder. June 8.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

11,234. R. Norwood. The manufacture of an improved coating compound or paint. May 17.

11,264. G. F. Newman. A composition to be used for rendering garments and other articles waterproof. Complete Specification. May 17.

11,447. T. C. Sanderson. Improvements in or relating to the manufacture of white lead paint. May 19.

11,522. T. C. Sanderson. Improvements in or relating to the treatment of white lead. May 20.

12,038. T. Cardiff. A new or improved damp-resisting washable sanitary water composition or paint. May 27.

12,184. G. Wegelin. An improved method of and means for the manufacture of lamp black and other carbon of great density. Complete Specification. May 28.

12,211. J. R. Wood. Varnish. May 31.

12,327. — Répin. Improvements in the treatment of india-rubber, gutta-percha, and other natural elastic substances. Complete Specification. June 1.

12,348. Saxby's White Lead Syndicate, Ltd., and H. G. Percival. Improvements in chambers for the manufacture of white lead. Complete Specification. June 1.

12,508. S. Rosenblum and The Commercial Ozone Syndicate, Ltd. *See* Class XII.

### Complete Specifications Accepted.

1897.

13,829. H. C. P. Graves. Compound for use as a substitute for india-rubber, leather, or other substances, or for any other purposes to which it may be applicable. May 25.

14,272. P. H. Hansen and J. K. Westengaard. Manufacture of casein compounds for electric insulating and other purposes. June 15.

14,911. P. Reidel and E. Kuhn. Manufacture of a compound or material applicable for use as a substitute for leather and india-rubber, and for floor coverings and the like, and apparatus therefor. June 1.

15,269. F. Fenton. Improvements in the production of artificial india-rubber and gutta-percha-like substances. June 8.

1898.

110. W. Peel. Anti corrosive composition or paint as a preventative against corrosion on metals. June 8.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

12,274. F. Grognet. An improved process for the preparation of skins. Complete Specification. May 31.

12,549. J. T. Wood. Improvements in and relating to the means used for the bating of hides and skins. Complete Specification. June 4.

12,553. W. W. McLaurin. A new or improved substitute for leather, and process of preparing same. June 4.

### Complete Specifications Accepted.

1897.

12,712. H. Bremer. Process for the solution of organic albumenous substances and for the production of soluble preparations of albumen. June 1.

1898.

5164. A. Stephan. Apparatus for the continuous treatment of size by boiling. May 25.

8099. D. Young.—From the Vaughan Machine Company United States. Machines for the treatment of hides or skins. June 15.

8218. V. Quaedolieg. Process and apparatus for the production of belting leather butts. June 15.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

11,511. E. Beanes. Improvements in the manufacture of sugar. May 20.

11,599. J. Welcker. An improved process for the production of milk-like solutions of albumen. Complete Specification. May 23.

11,753. A. J. Boult.—From C. M. Higgins, United States. Improvements in adhesive compounds. Complete Specification. May 24.

11,813. R. Kommerell. An improved process and apparatus for producing sugar in the form of bars, cubes, or other shapes. Complete Specification. May 24.

12,020. F. Virneisel. Improved process for producing soluble starch. May 26.

### Complete Specifications Accepted.

1897.

13,357. W. Powell. Manufacture of sugar. May 25.

1898.

8561. R. A. Chesebrough. Process of and means for refining sugar. May 25.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

11,373. T. T. Power. Improvements in the process of storing, maturing, and preserving whisky and such like liquors. May 19.

11,532. G. Souter and J. S. Souter. A new or improved method for the disposal of distillers' or brewers' refuse. May 23.

11,724. J. Watt and J. F. Watt. Improvements in and relating to the vaporising or treatment of by-products in distilleries and the like. May 24.

12,129. W. K. Fulleylove. A process for preventing the deterioration of beers, wines, or other fermented liquors in the barrel during the time of consumption. May 28.

12,143. J. Radley. Improvements in apparatus for carbonating malt liquors, wines, and other beverages. May 28.

12,334. K. Michel, jun. An improved apparatus for producing beer free from alcohol. June 1.

12,574. R. W. Perry. Improvements in or relating to the manufacture and bottling of champagne or other sparkling liquors. June 4.

12,660. G. Kreutzer and J. Schmitz. Improvements in or relating to brewing, and apparatus therefor. Complete Specification. June 6.

12,803. C. A. Stevenson. *See* Class B.

13,019. T. H. Nash. Improved apparatus for drying hops, malt, and the like. June 10.

13,053. A. Collette, Fils. Improved process for the manufacture of alcohol by saccharification and fermentation by mucedineæ, and apparatus therefor. Complete Specification. June 10.

### Complete Specifications Accepted.

1897.

13,312. P. F. Van der Gucht and A. Kreps. Malting apparatus. May 25.

19,161. C. O'Sullivan. Treatment of the by-products of brewers' and distillers' yeast, or either of them, whereby a substance is obtained that can be used as a food by man, and for other purposes. June 8.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

*A.—Foods.*

11,266. W. P. Thompson.—From J. H. Kellogg, United States. New or improved alimentary product made from edible nuts, and process of producing the same. Complete Specification. May 17.

11,414. L. Buchhold. Process for preserving preparations or objects. Complete Specification. May 19.

11,697. O. de Santa Cruz. A new or improved process and apparatus for the preservation of meat and other food or alimentary substances. May 24.

11,811. H. A. Hobson. Improvements in the production of concentrated foods. May 24.

12,068. E. Camphausen. Improvements in the preparation and production of essences of gravies and the like. May 27.

12,355. M. E. O. Liebreich. Improvements in the manufacture of artificial butter. June 1.

12,612. F. Grognet. An improved process for obtaining sterilised raw meat in the condition of powder. Complete Specification. June 4.

12,685. G. F. Thompson.—From J. S. Cochran, United States. A new or improved material as food for animals, and the method of preparing the same. June 7.

12,729. A. J. Bellamy. Concentrated tablet (blanc mange and custard). June 7.

12,869. N. Bendixen. Improvements in the method of and apparatus for sterilising milk. Complete Specification. June 8.

13,032. A. Denaeyer. A process for transforming yeasts into soluble alimentary matters. June 10.

*B.—Sanitation.*

11,463. F. M. Spence and D. D. Spence. Improvements in the treatment of sewage. May 20.

11,574. F. M. Spence and D. D. Spence. Improvements in the treatment of sewage. May 23.

12,489. R. Wilder, J. Wilder, and H. Wilder. Improvements in sludge or sewage tanks. June 3.

12,602. H. A. Hobson. Purifying agent for water sewage and other foul liquids. June 4.

12,803. C. A. Stevenson. Means for the disposal of waste products from distilleries or other works. June 8.

*C.—Disinfectants.*

12,601. H. A. Hobson. An improved deodorising disinfecting agent. June 4.

12,646. H. Noerdlinger. Improved process of manufacturing disinfectants rich in formaldehyde. June 6.

### Complete Specifications Accepted.

*A.—Foods.*

1897.

7220. A. S. Poyser. Method of treating milk and obtaining therefrom a gelatinous proteid substance and thus forming a nourishing food for invalids and others. May 25.

13,141. H. Higgins. Treatment of separated milk for alimentary purposes. May 25.

13,142. H. Higgins. Treatment of separated milk for alimentary purposes. May 25.

13,143. H. Higgins. Treatment of separated milk for alimentary purposes. May 25.

16,338. E. Wadham. Improved medicinal and nutritive compound or preparation. June 15.

19,161. C. O'Sullivan. *See* Class XVII. June 8.

29,189. A. Bernstein. Manufacture of a substitute for eggs. May 25.

*B.—Sanitation.*

1897.

12,984. F. P. Candy. Purification of sewage and polluted water. June 8.

13,400. W. D. Scott-Moncrieff. Purification of sewage and apparatus therefor. May 25.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

11,456. C. G. Hagemann and F. O. C. Zimmermann. Improved manufacture of celluloid. May 20.

11,949. R. Cremer.—From L. J. Dorenfeld, Germany. *See* Class II.

11,971. R. Cremer.—From L. J. Dorenfeld, Germany. *See* Class II.

12,040. R. Cremer.—From V. B. Drewsen, United States, and L. J. Dorenfeldt, Germany. A method or process for disposing of or destroying and utilising the spent liquor from sulphite pulp mills. May 27.

12,096. A. Aberg. Improvements in or relating to apparatus for treating cellulose or paper pulp and the like. Complete Specification. May 27.

13,022. M. L. Jonas. The manufacture or production of articles of paper or other suitable material chiefly for advertising purposes. June 10.

### Complete Specifications Accepted.

1897.

16,315. H. Schulk. Process of and apparatus for incorporating dissolved and suspended substances into paper, cardboard, or pasteboard in long sieve or cylinder machines. June 8.

17,736. T. Schiffer. Process for producing waterproof and washable glazed chromo-paper. May 25.

1898.

4945. W. Curtis. Settling and recovering apparatus for paper and pulp manufacture. May 25.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

11,891. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. A new pharmaceutical compound. May 25.

12,535. A. Heidenhain. Process for the neutralisation of the unpleasant odour of the vapours of formaline. June 4.

12,676 C. D. Abel. From The Actien Gesellschaft für Anilin Fabrikation, Germany. A new process for manufacturing compounds of acetamide. June 6.

12,956. E. Haas. The manufacture of improved articles of perfumery. June 9.

### Complete Specifications Accepted.

1897.

13,055. W. Simpson. Method of preparing fruit or other essences and extracts in tablet form. June 8.

19,014. A. J. Boult.—From E. Vial, Belgium. Extraction of quintate (quintine).

23,355. P. E. M. Jamain. Apparatus for compressing pharmaceutical and other powders for the manufacture of tabloids. June 15.

1898.

4736. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or preparation of derivatives of purine containing sulphur. June 1.

## XXI.—PHOTOGRAPHY.

### Application.

11,420. P. Mercier. Improvements in the preparation of argentiferous sensitive surfaces for photographic purposes. May 19.

12,272. E. Neuhaus. Process for production of relief photographs without the use of mould or block. May 31.

12,845. R. E. Liesegang. Improved method of preparing or preserving sensitive materials for the production of chromate or bichromate photographs. June 8.

### Complete Specification Accepted.

1897.

19,208. A. E. Wade. Photographic solutions as used for toning and fixing prints. June 8.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

11,496. G. Pinder. A new or improved explosive. May 20.

11,674. E. S. Clark. Improvements in or relating to safety tamping for explosives used for blasting operations in coal and other mines, and in apparatus for use in connection therewith. May 23.

11,856. A. A. Craig. Improvements in safety igniting composition. May 25.

11,927. L. L. Béthisy. Uninflammable nitro-cellulose products. May 25.

12,001. R. T. Maguire and D. P. Maguire. Improvements in or relating to lucifer matches. May 26.

12,141. J. Williamson. Improvements in the manufacture of gunpowder and blasting powder, and the treatment or preparation of carbonaceous materials therfor. May 28.

12,264. O. Imray.—From A. B. Markham, France. An improvement in percussion fuses for shells. May 31.

12,613. C. E. Bichel. A method of manufacturing explosives. June 4.

12,760. E. A. G. Street. Improved manufacture of explosives. June 7.

12,761. E. A. G. Street. Improved manufacture of explosives. Filed June 7. Date applied for March 22, 1898, being date of application in France.

12,962. A. de Pont. Improvements in the manufacture of soluble nitrocellulose, and in solvents therefor. June 9.

13,127. C. S. Bailey. Improvements in matches and manner of making same. June 11.

### Complete Specification Accepted.

1898.

10,489. J. G. Dixon. Detonators for railway fog signalling. June 15.

## PATENTS UNCLASSIFIABLE.

12,275. A Krefting. Improvements in the process of manufacturing valuable organic products from seaweed. Complete Specification. May 31.

12,277. A. Krefting. Improvements in the manufacture of valuable organic products from seaweed. Complete Specification. May 31.

12,416. A. Krefting. An improved system or apparatus for treating seaweed for the manufacture of products therefrom. Complete Specification. June 2.

12,599. W. A. C. Hünkerpöller. Improved artificial whalebone. Complete Specification. June 4.

13,151. A. Krefting. Improvements in the manufacture of organic products from sea-weed. Complete Specification. June 11.

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## NOTICES.

The Society's Office will be closed from September 2nd to September 17th next, and the General Secretary will feel obliged if members who have changes of address to communicate will send them in before that date.

### Annual General Meeting.

The group photograph, taken at University College, Nottingham, is a very successful picture. Members who desire a copy should send the sum of 4*s*. 6*d*. to Arthur Neale, Studio, St. Peter's Church Walk, Nottingham. Terms :—Cash with order.

### Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, half of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer, who is making every effort to complete this vast work.

The prices are as follows :—

| | |
|---|---|
| To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List. | Each copy 10*s*. |
| To Subscribers .................... | „ 12*s*. 6*d*. |
| To others...................... | „ 15*s*. |

Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

### Telegrams.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

## LIST OF MEMBERS ELECTED 13th JULY 1898.

Bhattacharyya, Haripada, Dakinsore, P.O. Kamarhatty, 24, Pergunnahs, Bengal, Chemist.

Cosby, Chas. D., Eidsvold, Norway, Mining Engineer and Assayer.

Forgie, Jas. T., Viewfield, Bothwell, N.B., Civil and Mining Engineer.

French, Wm., M.A.. 135, Walmersley Road, Bury, Lancashire, Science Teacher.

Galewsky, Paul, c/o C. H. Boehringer Sohn, Nieder Ingelheim a/Rhein, Germany, Colour Chemist.

Goodrich, Chas. G., Akron, Ohio, U.S.A., Rubber Manufacturer.

Hall, Wm. F., Barrett G.M. Co., Godwaan River, S.A.R., Cyanide Manager.

Hart, F. Cyril, 12, Western Road, Shoreham, Sussex, Chemist (Chemical Works).

Meyer, Dr. Franz, Somerset Chemical Co., Bound-Brook, N.J., U.S.A., Chemist and Manager.

Smith, John, Hartley Street Dyeworks, Dewsbury, Yorks, Dyer.

Van Epenhuysen, Cornelis, Zwijndrecht, Holland, Chemical Manufacturer.

Young, Thos., Stubbins Printworks, Ramsbottom, near Manchester, Calico Printer.

## LIST OF MEMBERS ELECTED 25th JULY 1898.

Bird, Frank W., Vinegar Works, Cambridge, Vinegar Brewer.

Hall, Clarence A., Sault Ste. Marie, Mich., U.S.A., Chemist.

Hall, Frederick W., 25, Oak Bank, Harpurhey, Manchester, Dyer.

Parker, Charles H., The Copthornes, Copthorne Road, Wolverhampton, Chemist.

Taylor, Newman, 19, Cleveland Road, Crumpsall, Manchester, "Water Specialist."

## CHANGES OF ADDRESS.

Bradburn, J. A., l/o Saltville; retain Journals until further notice.

Brown, F. W., l/o Maida Hill; 56, Grange Park, Ealing, Science Coach.

Brown, Jas., l/o Mountpottinger; Avoneil Distillery, Belfast.

Claudet, F. G., l/o Dulwich; Ennismore, Willesden Lane, N.W.

Dick, F. Burnett, l/o City Road; c/o J. M. Bell, 5–6, Great Winchester Street, E.C.

Fairlie, H. C., l/o Glasgow; Camelon Chemical Works, Falkirk, N.B.

Hamilton, Robt., l/o Shotts; c/o Bestwood Chemical Co., Ltd., near Nottingham; and (Journals) 138, Gregory Boulevard, Nottingham.

Hosie, G. H., l/o Innellan; 136, Cambridge Drive, Kelvinside N., Glasgow.

King, C. M., l/o London; Stahlhammer o S., Germany.

Leather, Dr. J. Walter, l/o London; Dehra Dun, N.W.P., India.

Lucas, Alf.; Journals to English Pension, Esbekieh, Cairo, Egypt.

McLellan, J. Y.; Journals to Cogoletto, Liguria, Italy.

Mansbridge, Wm., l/o Lee; Colgate, near Horsham.

Marchlewski, Dr. L., l/o Higher Broughton; Lynton House, Rushford Avenue, Levenshulme, Manchester.

Mayfield, H. B., l/o Fairview House; Normanhurst, Mundy Street, Heanor, near Nottingham.

Mordle, F. Dare, l/o The Park; 38, The Ropewalk, Nottingham.

Morrison, G. R., l/o Plaistow; c/o E. Ryan and Co., Ltd., Pope's Quay, Cork.

Paget, R., l/o Leeds; Guildford House, London Road, Croydon.

Powter, P., l/o London; c/o Pauling and Co., Beira, East Coast of Africa.

Prentice, Manning, l/o Stowmarket; Anglesea House, Felixstowe.

Sedding, G. H. P., l/o River Junction; c/o Florida Syndicate, Jacksonville, Fla., U.S.A.

Simon, Dr. A., l/o Saarbrucken; 55–56, Bishopsgate Street, E.C.

Tasker, G., l/o Brunswick; c/o Cuming Smith and Co., Chemical Works, Yarraville, Melbourne, Victoria.

Vanderpoel, Dr. F., l/o Roseville Avenue; 153, Center Street, Newark, N.J., U.S.A.

White, W. Gilchrist, l/o The Hodge; 75, West End Villas, Broadbottom, near Manchester.

Young, J. W., l/o Westmoreland Terrace; 4, Portland Terrace, Newcastle-on-Tyne.

## CHANGE OF NAME AND ADDRESS.

Trevaille-Williams, T. (formerly Williams, Thos.), 5, Madeira Villas, Horrabridge, Devon; Journals as before.

## Deaths.

Oushkoff, P. K., Elabouga, Government of Viatka, Russia.

Pankhurst, Dr. R. M., 10, St. James' Square, Manchester. July 5.

# PROCEEDINGS

OF THE

# SEVENTEENTH ANNUAL MEETING.

President: Prof. F. CLOWES, D.Sc.

## FIRST DAY.—Wednesday, July 13th.

The seventeenth annual meeting of the Society of Chemical Industry was held in Nottingham on Wednesday, July 13th, in the Lecture Theatre of the University College, Prof. F. Clowes, D.Sc., President of the Society, in the chair.

The minutes of the previous meeting were read, confirmed, and signed by the President.

### Appointment of Scrutators.

Dr. G. Schack-Sommer moved the appointment of Mr. Gibson and Mr. Canziani as scrutators.

Mr. Hemingway seconded the motion, and it was adopted.

The President then declared the ballot closed.

### Report of Council.

The Secretary read the report of the Council, which was as follows:—

We have the honour to report that the number of members on the register is 3,185, as compared with 3,037 at the last Annual Meeting. During the year 311 new members have been elected, as compared with 226 last year. This is the largest number since 1885, when 345 elections were recorded, though 1890, with 298, runs it close. The loss has been 152, as compared with 162 last year. Among the latter are 28 deaths: Dr. M. Alsberg, L. Ashworth, J. J. Bowrey, Charles Cochrane, Sir Henry Doulton, S. E. Field, G. Fletcher, J. W. Freestone, W. A. L. Hammersley, Henry Hills, J. H. Hodgkinson, Thos. Holliday, Dr. F. Hurter, Thos. Jackson, Alfred Julien, Jas. E. King, Horace Koechlin, H. W. Langbeck, Dr. H. Lassing, Robt. Mitchell, Jas. Napier, Dr. R. M. Pankhurst, W. S. Pritchard, Harold Rawcliffe, Chas. L. Stanley, Edmund Tonks, J. Ivens Vaughan, and G. I. J. Wells.

During the past session, 87 papers have appeared in the Journal, as compared with 104 last year. The excess of revenue over expenditure, as may be seen from the Treasurer's Statement in the last number of the Journal, amounted last year to 737*l.* 12*s.* 5*d.*, as against 1,011*l.* 3*s.* 11*d.* the year before. In 1896 the receipts from the Collective Index were 347*l.* 5*s.*, against 7*l.* 0*s.* 6*d.* in 1897; while the expenditure on Index account was almost the same in each year. This accounts for the smaller surplus in 1897.

The Collective Index is nearly finished, and will very shortly be in the hands of members. In revising the proof it was found that certain alterations in the arrangement of the matter, which could not have been foreseen, would materially add to the value of the Index. These have now been effected, and the whole MS. is now ready to be placed in the printer's hands. It was also found that no firm of printers had a sufficient stock of type of the kind required to keep the whole in type during revision. Our thanks are due to the special committee which, to relieve the pressure on the indexer, has undertaken the entire work of revising page proof.

The sanction of the meeting is asked for a slight modification of the byelaws, notice of which was given in the April number of the Journal. The first three changes concern the taking of the ballot at annual meetings, and bring the general byelaws into more perfect harmony with those of the Sections. It is proposed that, when the number of nominations by members does not exceed the number of vacancies on the Council, no ballot shall be taken, but that when the nominations exceed the vacancies, the existing practice shall be followed. The proposed change, besides saving money, will expedite the business of annual meetings when, as on the present occasion, there is no contest. The remaining amendment is a purely verbal one to render a vague expression clear.

The following chairmen of Sections retire this year:—Prof. C. F. Chandler (New York), Mr. George E. Davis (Manchester), Mr. Thos. Fairley (Yorkshire), Dr. Chas. A. Kohn (Liverpool), and Dr. Rudolph Messel (London). They are succeeded by Messrs. Thomas J. Parker, David Watson, D.Sc., Christopher Rawson, Alfred Smetham, and Boverton Redwood. Mr. John Heron retires, and is succeeded by Mr. A. R. Ling, as Hon. Secretary of the London Section. Prof. H. R. Procter has been induced to retain his post for another year.

Reference may be made to the part played by the President in the Joint Conversazione given by the Presidents of Institutes of Chemistry, Society of Public Analysts, and our own Society in London in May last. The Council desires to place on record its appreciation of his hospitality on that occasion.

The President mentioned that there were one or two clauses officially added to the report that morning, which were not in the printed statement in the hands of members.

Dr. W. H. Perkin moved the adoption of the report, remarking that it was a matter for great congratulation to the Society to find such a large accession of members during the past year, namely, an addition of about 311. It showed, he thought, the vitality of the Society, and an appreciation of its value, when they got such a large number of new members. He remembered being at the first meeting held to inaugurate the Chemical Society, and he little thought then that the Society would ever possess over 3,000 members, as it does at the present time. It was now the largest Society in the kingdom which had to do with chemistry.

Mr. J. M. C. Paton seconded the motion. A report of that character, he observed, must, of necessity, be somewhat stereotyped, unless something special took place. The increase in the number of members showed that the Society was appreciated, and was doing good work. Some of their members had passed away, but there were others rising to take their places. That the index was making progress and would shortly appear was good news. The chairmen of Sections of course changed, but he thought the members who were put forward on this occasion were good men. Altogether he regarded the report as a satisfactory one.

The report was then adopted.

### Finance.

The President said he had to apologise for the absence of Mr. E. Rider Cook (the honorary treasurer of the Society), owing to indisposition.

The Secretary read the Treasurer's report. In the year 1897 they had received in respect of 2,818 subscriptions a total sum of 3,524*l.* 12*s.* 11*d.* They also received 226*l.* entrance fees, and two life composition fees, but those sums were not put to revenue at all; they were regarded as capital, and therefore did not go into the total receipts of the year. They received 7*l.* on account of the Collective Index, and also a sum of 262*l.* from the invested savings of the Society. From the "Journal" they received 576*l.* for advertisements and 368*l.* 10*s.* 8*d.* for sales to others than members, and of odd numbers. That gave a total of 944*l.* received from the "Journal." Therefore from the four departments named—subscriptions, 3,524*l.* 12*s.* 11*d.*; on account of the Collective Index, 7*l.*; investments, 262*l.*: and the "Journal," 944*l.*—they had received altogether 4,738*l.* 16*s.* 8*d.* With regard to the expenditure, first came the "Journal," which was an important item. They had spent in printing and issuing the Journal (including postages), 1,598*l.* 2*s.* 2*d.*, and in editing it, 1,209*l.* 7*s.* 10*d.*; which, with insurance, 3*l.* 5*s.*, made a total of 2,802*l.* 5*s.* spent on the "Journal." Other expenses were:—For printing, 68*l.*; sectional expenses, 306*l.*; secretary's salary (portion not included in the "Journal" editorial expenses), 337*l.* 10*s.*; expenses in connection with the annual meeting, 89*l.* 6*s.* 10*d.*; honorary treasurer's assistant, 62*l.* 10*s.*; stationery, 46*l.* 15*s.* 4*d.*; office expenses, 113*l.* 5*s.* 4*d.*; binding books, 5*l.* 15*s.*; auditor's fees, 10*l.* 11*s.* 6*d.*; Collective Index, paid on account, 100*l.* 10*s.*; bank charges, 4*l.* 3*s.* 8*d.*; treasurer's petty cash, 14*l.* 3*s.* 6*d.*; secretary's petty cash, 49*l.* 10*s* 7*d.*; leaving a balance in hand of 737*l.* 12*s.* 5*d.*, as mentioned in the report.

### Vote of Thanks to the Treasurer.

Mr. George E. Davis proposed a vote of thanks to the treasurer for his statement. They would all, no doubt, be aware, he remarked, that Mr. Rider Cook had been with them from the time of inception of the Society, and had performed a labour of love in looking after the finances of what had been a very successful business. They must look at it in this way: when the Society was formed in the year 1881, he was in charge of the books of a Society having 300 customers. To-day he is in charge of all the accounts, which, as they had just heard, amounted to some 4,000*l.* or 5,000*l.* in the year, and he had now over 3,000 customers to look after. They would see, therefore, that their treasurer had a very large amount of work to do and to supervise; and, having regard to the fact that he did it entirely for the love he had for the welfare of the Society, the very least they could do was to give him a very hearty vote of thanks. Moreover, they were now ramifying over the whole surface of the world; their expenses were increasing, as also was their revenue, and the number of members seemed to be getting larger and larger every year. He, for one, should not be satisfied until he saw the Society ramifying over the whole world.

Mr. Forbes Carpenter seconded the motion, remarking that like Mr. Davis, he had some knowledge of the work the treasurer had to do. Their Society was one to put by money every year, and the treasurer had had the responsibility of advising as to advertisements. He had been associated with Mr. David Howard in regard to those investments, and the thanks of the Society were due to both gentlemen in this connection.

The motion was adopted.

## THE PRESIDENT'S ADDRESS.

In the summer of 1890, under the distinguished presidency of Sir Lowthian Bell, and during the local chairmanship of Sir John Turney, the Society of Chemical Industry held its annual meeting in the town of Nottingham, the University College, in which we are now again assembled, being its head quarters. In acknowledging the courtesy which the Mayor and Town Council have again extended to the Society by permitting its use of the College buildings, we desire to congratulate the town of Nottingham upon having been raised since our last visit to the dignity of a city, the present Mayor of Nottingham upon having received academic distinction, and the Town Clerk upon having received the honour of knighthood. We believe that the willingness shown by Nottingham to receive our Society must be connected with the progressive and hospitable spirit which the city has always shown, and with the existence in it of a large and successful University and Technical College, and of a flourishing Section of our Society of Chemical Industry.

### Origin and Development of the Society.

In acknowledging the welcome accorded to our Society, it is natural to compare our present position with our origin and past. We are informed in the Report of our Council that the membership of the Society has now reached a total of over 3,000, and it must be remembered that this large enrolment has been secured in a period of only 17 years. We are yet several years removed from attaining our majority. Towards the end of June 1881 the first General Meeting of the Society was held in London under the presidency of Sir Henry Roscoe. The Society was then only three months old, and numbered about 300 members. It was in every sense a national society, but distinguished continental chemists were willing to help the young association by taking up its membership. The first president stated that "the main object of the Society is to bring together at definite intervals those who are interested in or possess knowledge concerning the utilisation of chemical action on a large scale, and have charge of, or are connected with, those large branches of industry which are dependent upon chemical principles. To the interchange of ideas brought about by such personal contact may well be added the publication of the proceedings of our meetings, as well as of information or intelligence interesting to our members. The Society cannot fail in this way to contribute to the advancement of those important branches of national industry which are dependent on chemical principles. This we hope to do, not only by bringing forward new processes, or discussing the advantages or disadvantages of old ones, but also by acting as a bond of union hitherto non-existent between individuals engaged in cognate pursuits, giving them a personal interest in one another, and thus encouraging that good fellowship which is not an unimportant factor in the smooth working of our social machinery."

Thus spoke in 1881 the first President of the Society; and those of us who have, with him, noted from those earliest days the development of his scheme, cannot but see how completely his intentions and predictions have been fulfilled. The Journal, referred to by Sir Henry Roscoe, has come into existence, and is one of the most complete and valuable records of technical chemistry in the world. Those interested in the advancement of applied chemistry in this country have become to a large extent united in their efforts, and therefore more successful in attaining their objects.

The members of the Society value the organisation on account of the information brought together in their Journal. But they have further advantages afforded them by the increased opportunity of meeting together, owing to the establishment of local Sections. The existence of these local Sections is an important and somewhat special feature of our Society. These Sections are now existent and active in London, Manchester, Liverpool, Newcastle, Nottingham, Leeds, Glasgow, and New York; and the existence of a New York Section is an indication that the importance of the work of the Society is felt outside our own country; this is further proved by the very large number of foreign general members who have been enrolled.

Those who have watched the gradual rise and development of these local Sections of the Society will acknowledge the very important service which has been rendered by our Provincial Colleges. The great importance and advantage of the movement, which has brought higher university instruction into our large provincial centres, are now universally conceded. But there is cause for congratulation, amongst those who are endeavouring to promote the advancement of applied science in this country, that the foundation of a University College in a provincial centre has not only led to the introduction of higher instruction in pure science, but has also advanced in a most important way the interest and education in applied science. One of the evidences of this gratifying result is the fact that in the centres in which our University Colleges are established and successful, we find arising Sections of the Society. Leading manufacturers associate themselves with those who are promoting science educationally, and with those who have passed through the classes of the College, and together they maintain a useful Section of our Society.

My lengthy experience as a chemical teacher in Nottingham leads me to lay stress upon the advantages resulting from the establishment of a local Section of our Society in connection with the local College. In 1881 the Town Council of Nottingham, supported by the public spirit of its inhabitants, had responded to a challenge thrown out by an anonymous donor; and, by supplementing his handsome gift by public funds, had built the stately and convenient buildings in which we are now assembled; they had also appointed a staff of teachers, amongst whom I represented the branch of chemistry and metallurgy; and they had founded a curriculum of instruction in the principles and applications of science, which was in a great measure a response to the general demand for the provision of technical instruction throughout our country. It was felt desirable that the provision of general higher-class culture should be the main object of this University College, but that instruction should also be provided which would be of value to those equipping themselves for industrial pursuits in an industrial centre. The teachers in the college have therefore always borne in mind that whilst they teach scientific principles first and foremost as the true foundation of all industrial success, they should also in some considerable measure illustrate their teaching by pointing out the industrial applications of those principles.

What could be more suitable and advantageous than the establishment, in connection with the Chemical Department of a College possessed of these aims, of a Section of the newly founded Society of Chemical Industry? In 1885 a little band of chemists of Nottingham and the neighbourhood started the Nottingham Section of this Society. I was honoured by being its first chairman. The early existence of this Section naturally depended very largely upon support from those connected with the College, either as members of the Management Committee or as chemical teachers and senior and past chemical students. Even from the earliest times, however, those engaged in industrial enterprise and pursuits in the town and neighbourhood rallied in rapidly increasing numbers around this original nucleus, and the Section soon felt that it was fairly representative of the chemical work and industry of the district. I am glad to say that the Section is now as active as at any period of its career, and its activity is shown not only by its frequent contribution of important matter to the Journal, and by its regular and frequent meetings, but by its eagerness to entertain for a second time the general Association of which it is a constituent part. There is no doubt that this Section has been of advantage to those concerned in the applications of chemistry in the district. It has certainly had the effect of maintaining an interest in chemistry amongst the past students of our college, and of reminding the teachers of the college that they must consider in their chemical instruction not only the paramount importance of chemical principles and of pure science, but also of chemistry in its connection with industrial applications.

I have been tempted to speak at some length of the experience of the Nottingham Section—the Section with which I have had the longest experience and have been for many years personally identified—not because I desire to place it in an unduly prominent position; but rather because the origin and development of the Section are, I believe, typical, and represent what has already occurred in most, if not in all, of the other Sections of the Society. May I add that we hope that the Society is destined yet to see a similar rise and development of new Sections in other important industrial centres of this country? And may we not further hope that the existence of a branch of the Society in New York proves that Sections of this Society are destined to arise generally in the industrial English-speaking communities of the world, and to lead thus to a federation of interest in the advancement of industrial chemistry, which may even antedate that more general world-wide federation in British interests which our politicians are desirous of fostering?

A glance down the names of those who have served the Society since its foundation, shows that its interests have been promoted by men who stand prominently before the chemical world, not only as industrial chemists, but also as teachers, as investigators, and as professional chemists; and the stimulus given by such men, who, whilst leading a busy life, have found time to help forward the objects of the Society, probably accounts for its continued and increasing progress. During the year which you have allowed me the honour of serving you, I have been gratified to hear of larger accessions to our membership, in the monthly list which is presented at our Council meetings, than have occurred during any recent year.

### Obituary Notices.

The gratification which is naturally felt in seeing the Society thus largely increasing in numbers and in usefulness is, however, somewhat alloyed by the notification of the loss by death of no less than 27 members, and we have to note with regret in the list of recently deceased members the names of such men as Sir Henry Doulton, Horace Koechlin, Dr. F. Hurter, and Charles Cochrane; whilst amongst those who are not members, but whose names are classical, are further to be noted Sir Henry Bessemer, Paul Schützenberger, and August Kekulé. It is only right that we should unitedly avow our indebtedness to men who have exerted so important an influence in furthering the objects which our Society has in view.

It is unnecessary to refer at any length to the distinguished and useful careers of these distinguished chemists who are now numbered with the majority, since our own Journal and the Journal of the Chemical Society record in their obituary notices and memorial lectures the principal features of their scientific and industrial work. There is some satisfaction to be derived from the fact that most of these eminent men had attained the usual limit in the span of human life, and had remained long enough amongst their fellow-men for the importance of their work to receive a fair measure of appreciation.

Ferdinand Hurter may, however, be considered to form an unfortunate exception, in that he has been removed from us in the prime of his intellectual vigour, and at a time of life when the matured experience of an active spirit is usually productive of valuable results. It is our Liverpool Section which suffers the loss of the stimulating effect of the presence amongst them of Hurter's personality. But the whole Society will suffer the loss of his original communications, the most important of which have been printed in our Journal. An account of Hurter's career appears in a recent number of our Journal, and will serve as a permanent memorial of the affection and esteem in which he was held by those who knew him best—his fellow-members of the Liverpool Section. A friend who had long been associated with him states that: "He had the advantage of a sound and wide education during his student years; but to his credit it may be said that by strenuous and unremitting effort he continued his education during his whole life. He was an intellectual athlete, and kept himself in splendid mental training by increasing practice. The faculties of his mind were under complete control, and he possessed a most retentive memory, so that whenever occasion required, he could set himself to work, bring out of his treasury things new and old, and solve technical problems with amazing rapidity and accuracy. Amongst the most marked elements of his success as a technical chemist were the ability he possessed in a remarkable degree of devising new apparatus for new problems and his insight into the nature of the technical and commercial problem before him. He was never lured into any side-issue, however interesting from a scientific point of view; he kept but one end in sight—the solution of the problem in hand, and his grasp of the situation, both technically and commercially, was always complete and clear."

Paul Schützenberger, though not enrolled among our members, was one who largely influenced the advancement of industrial chemistry by his researches on the natural dyestuffs and colouring matters. His original investigations, however, were extended to many other branches of our science. As a teacher, his influence was also felt. Commencing as a lecturer in the Realschule and the Hochschule of Mülhausen, he became subsequently Ballard's assistant at the Collège de France, then second director of the chemical laboratory at the Sorbonne, and professor in the Collège de France. In 1882 he was finally appointed director of the newly-founded School of Chemical Industry and Physics. His text-book and other contributions to chemical literature are too well known to require any special mention.

It is to Schützenberger that our recently deceased member, Horace Koechlin, owed his early chemical education and inspiration. His many investigations in bleaching, dyeing, printing, and in the production of dyestuffs indicate, not only the effect of his teacher's influence, but also the effect of his progenitors' occupation, since Horace Koechlin represented the third generation of those who had interested themselves successfully in these pursuits.

August Kekulé represents, amongst those who are no longer with us, the great master in chemistry; the man who promotes the advancement of the science, not only by his own brilliant researches and theoretical conceptions, but also by his systematic teaching and by the publication of students' literature, and more especially by the stimulating and inspiring effect which he exerts upon all those chemists who come into contact with him. I will not attempt to sketch Kekulé's career; this has been done already in the account of his scientific work by Dr. Japp, which has been published in a recent number of the Journal of the Chemical Society. It cannot be doubted that members of our Industrial Society will heartily appreciate and honour our great chemical masters, and accord to them the very high position which they should hold in their esteem.

The important work of DOULTON and of BESSEMER has been before the world so long, that it is known, not only to the chemist and metallurgist, but to the general public. The names of these creators of industry have practically become incorporated into our language, and every educated person knows the meaning of the terms "Doulton ware" and "Bessemer steel."

### THE MEDALLIST: WILLIAM HENRY PERKIN.

Men of the stamp of Doulton and Bessemer are peculiarly English; it is the glory of England to have reared many such sons; and amongst them—though fortunately he is still hale and hearty and actively working in our midst—we must certainly number the man whom your Council has this year selected for the award of its medal: I refer, of course, to WILLIAM HENRY PERKIN.

While I am treating of the biographical part of my subject, which unfortunately has been thus far a sketch of those whose labours in this world are ended, I am sure that you will allow me to sketch in a few words the career of our distinguished medallist, to whom I shall, before we separate, have the pleasure of handing, as your representative, a permanent mark of our esteem and appreciation You are probably all aware that, like Doulton and Bessemer, Perkin has been the founder of a great and important industry. The production of artificial colouring matters from coal-tar products owes its origin, and in large measure its development, to Perkin. As industrial chemists you will all undoubtedly be able to appreciate the great importance of this department of Perkin's work. You will also undoubtedly wish that his devotion to the interests of our Society should be acknowledged. We find him actively supporting this Society in in its foundation and in the critical early years of its existence. In 1881 he was elected to serve as its President—his immediate predecessors in that office having been Walter Weldon, Sir Frederick Abel, and Sir Henry Roscoe. Of the very great services which Perkin has rendered on the Council and committees of this Society many here could join me in speaking in the highest terms. But I am sure that you will wish me to add to the causes of our appreciation which I have already mentioned, the very great additions to our science which have resulted from his chemical researches. On looking through the lists of chemical papers in the catalogue of scientific papers edited by the Royal Society, Perkin's name stands very high in the honour list of indefatigable and successful investigators, and he ranks in this respect with the leading chemists of our times. It is only right to take every opportunity of emphasising this fact, since, owing to Perkin's personal modesty and retiring disposition, and to the fact that he has held no public scientific appointments, the very great importance and extent of his scientific work is apt to be lost sight of, even by members of our Society. As he has told me himself, he has almost continuously, since his early schooldays, been following scientific research as a recreation; and this was the case even during the years when he was founding and conducting a large chemical manufactory and engaging in manufacturing and mercantile pursuits.

I have referred to Perkin's schooldays. You will, I trust, pardon me if I venture to refer to my own early connection with our medallist, and say that I rank it as an honour, shared amongst others by Edward Rider Cook, John Spiller, William Thorp, F. J. M. Page, and William Garnett, to have been a schoolfellow of Perkin. We all of us owe a great debt of gratitude to the City of London School, then situated in Milk Street, Cheapside, but since removed to the Victoria Embankment. That school was then under the headmastership of the Rev. Canon Mortimer, D.D., and was the pioneer amongst the schools in giving instruction in natural science.

The science-master of those days was to most of us for a time also our form-master. Thomas Hall—still affectionately termed by us "Tommy"—was the man who gave us our early teaching in science, and who by his infectious enthusiasm led us to look upon natural science as an entrancing pursuit. To those of us who were juniors to Perkin at the school, Perkin, who had just made his discovery of "mauve," was freely held up as an illustration of what advantages might be secured by a scientific training. In fact, Thomas Hall, who otherwise does not appear amongst the discoverers of his day, may be said to have made one all-important discovery: he discovered Perkin. It was while Perkin was in Hall's form, and before he had shown special aptitude for any department of study, that his form-master discovered in him an enthusiasm for experimental chemistry, which the master wisely fostered and encouraged to the utmost. In 1851 we find young Perkin assisting in the preparation of the chemistry lectures at the school; and two years later, when he was only 15 years old, he went by Hall's advice to the newly instituted Royal College of Chemistry with the intention of continuing his chemical studies under Dr. A. W. Hofmann. Perkin's rapid advance is marked by the fact that two years later he was acting as assistant to Dr. Hofmann in his research laboratory. In the following March he communicated his first research to the Chemical Society; and during the Easter recess (1856), while he was attempting to produce quinine artificially, he discovered "aniline purple" or "mauve"—a discovery which laid the foundation of the extensive coal-tar colour industry of the present time.

And here it is only right to refer to the enthusiasm which the late Dr. Hofmann aroused, as a teacher and investigator, in the students and assistants who were so fortunate as to be associated with him; and many men who hold prominent positions in our Society owe their early instruction and impetus to this great and genial master.

Experiments made at Messrs. Pullars' dyeworks at Perth encouraged Perkin, at the early age of 18, to devote himself to the development of his discovery, and to extend the laboratory preparation of mauve to an attempt at its manufacture. The manufacture involved the preparation of various substances on the large scale, which had hitherto only been made in small quantities in the laboratory: it was further necessary to devise entirely new plant. But the scientific knowledge and inventive and practical resource of Perkin were equal to surmounting these difficulties. Working in association with his father and brother, he manufactured the new dye and actually sold it to the silk dyer in 1857—the year immediately succeeding its discovery. The success achieved by Perkin in making and applying the new dye was rapidly followed by the introduction of additional coal-tar colours, to the discovery and application of which Perkin himself contributed. The credit remains to Perkin of producing alizarin synthetically from anthracene, almost simultaneously with Graebe and Liebermann, but by a practicable method, and thus leading to the manufacture of the madder-dye taking the place of its preparation from the cultivated madder-root.

In 1873, Perkin retired from strictly technical pursuits, and he has since devoted himself indefatigably to scientific research, with the most important results. He is still continuing a research on magnetic rotation, and many instalments of his results have already been published. The name of our medallist is thus associated both in the past and at the present time with the progress of chemical knowledge and industry; and we may reasonably indulge in the hope that we have yet many more important researches to receive from his laboratory in the future. We have further the satisfaction of knowing that when the time arrives, which we all sincerely trust may be long deferred, for this active researcher to cease to contribute to the advance of science, the family name will still figure largely in the scientific records of the time, since Perkin has sons who are fully imbued with the capacity for and importance of scientific research, whose names are already ranged honourably alongside of that of their father, and who will prevent the lustre of the good name from becoming dim.

It has not been possible, within the limits of a paragraph of a presidential address, to do adequate justice, even in outline, to Perkin's work and activity. Those who wish to pursue the sketch further should refer to the Hofmann memorial lecture delivered in May 1893 before the Chemical Society, in which much of Perkin's work is followed out in greater detail; whilst a sketch of Perkin's career appears, under "Names of Note," in the *Dyer and Calico Printer* of Nov. 15, 1889. But a full appreciation of his researches can only be secured by studying them in detail in the Journal of the Chemical Society and in other scientific and industrial journals in which they have been published.

It is commonly said that the secret of the success of the English soldier in war lies in the fact that he never knows when he is beaten. One rises from a study of Perkin's career with the impression that he pursued his work of peace in a similar spirit, and triumphed over the many difficulties which beset him as pioneer and discoverer by never acknowledging an apparent defeat. By conferring our medal on a man of this type—a man also of untiring activity and industry—we feel that we are honouring our Society in the endeavour to honour him.

While we trust that the mark of our appreciation which we to-day bestow upon Perkin will meet with his acceptance, and will be treasured as an heirloom and memorial in later days, it would not be right to overlook the fact that the institution of the award of our medal is very recent, and that the medal is now only awarded for the second time. Accordingly, it naturally occurs that we try to confer honour upon a man already laden with the honours of his fellow chemists. And in attempting to confer one more honour, it is only right to record the recognition which Perkin's work has already received.

In 1859 the "Société Industrielle" of Mülhausen awarded a silver medal, and some time afterwards a gold medal, to Perkin for his discovery of the mauve. Then we note his election to the Chemical Society (1856), the Royal Society (1866) at the early age of 28, to the Honorary Secretaryship of the Chemical Society (1869), to the Presidency of the Chemical Society (1883), to the Presidency of our own Society (1884), and to the Honorary Membership of the German Chemical Society in the same year. Our own medal is an addition only to others already in the possession of Perkin: these include the Royal Medal of the Royal Society (1879), the Longstaff Medal of the Chemical Society (1888), the Davy Medal of the Royal Society (1889), the Albert Medal of the Society of Arts (1890), and the Birmingham Medal of the Gas Institute (1892).

Academic distinctions have also been accorded to this truly eminent man, and are evidence of the fact that his honours came from widely different sources. The University of Würzburg, well known to me as my *alma mater*, conferred the title of "Philosophiæ Doctor" (Ph.D.) upon Perkin at its centenary celebration in 1882, and the University of St. Andrews gave him the honorary degree of LL.D. in 1892.

This is a long but, I fear, still an incomplete list of the honours already conferred on our medallist of this year.

### Some Technical Matters.

In concluding my address, I will now only briefly allude to some matters of applied science which have of late years been prominently under my notice.

Living for upwards of 20 years as a teacher in mining districts, I have had my attention constantly directed to the improvement of scientific methods applied to coal-mining, and more especially to methods at once delicate, exact, and trustworthy, for detecting and measuring the firedamp or methane in the air of the mine. Careful experiments, which were widely extended both in the laboratory and in the coal-mine, led me to infer that no simple and practicable method equalled in these respects the hydrogen flame when it was safely and suitably applied. The "flame-cap" over the standard hydrogen flame, as it is seen in the adapted safety lamp, not only detects, but measures firedamp with great precision. This standard test-flame has since been adapted to the detection and measurement of coal-gas in air, and, in conjunction with Mr. Boverton Redwood, our newly-elected London chairman, I have applied it also to the detection and measurement of petroleum vapour in the air. A full summary of our work in these directions, which includes and somewhat extends our papers in the Journal of this Society, appears as a monograph published by Messrs. Crosby Lockwood and Son under the title of the "Detection and Measurement of Inflammable Gas and Vapour in the Air."

The new illuminant, acetylene, has claimed in some measure in recent years the attention of most of us. It cannot be maintained that acetylene is a newly discovered gas, or even that the material from which it is now produced is new to science. But it may certainly be claimed that the cheap production on the large scale of the calcium carbide, from which acetylene is most economically prepared, is a recent discovery. And it is this discovery which has brought acetylene to the front as a possible and powerful illuminant. An inspection of the flame of acetylene, when it is suitably produced from an appropriate burner, is sufficient to satisfy anyone that it is most desirable to secure the service of this brilliantly burning gas. Apparently the general adoption of acetylene for illuminating purposes is being delayed by the feeling that the gas is dangerous; and there certainly are not wanting instances of dire disaster arising from its preparation and storage.

In the liquid condition acetylene can undoubtedly, owing to its endothermic character, undergo most violent explosive decomposition, yet Raoul Pictet strongly urges that the liquid is safe if it is suitably prepared and stored. In the gaseous form we are assured by Vivian Lewes that the gas does not undergo this explosive decomposition when it it is stored under a pressure less than two atmospheres. Lewes further states that this gas may be burnt in such a way, if it is properly prepared, as not to rapidly cause obstruction in the burners—a difficulty which has caused trouble in the past; he further states that while it is not of use for enriching coal-gas, it is a valuable enricher of oil-gas.

It is to be hoped that the public may ultimately receive satisfactory assurance that they can employ acetylene as an illuminant with success and safety under suitable conditions. It will be remembered that other endothermic substances, such as nitroglycerine or dynamite, and nitrocellulose or guncotton, have now attained this desirable condition, after having in the past worked such disaster as to cause them to be looked upon by the public as hopelessly unsafe. It must be remembered in any case that acetylene when mixed in very small proportion with air furnishes an explosive mixture, but danger arising from this cause must certainly be looked upon as preventable.

The enrichment of coal-gas, which it was hoped might have been effected by acetylene, is now usually secured by the indirect employment of water-gas. The large amount of poisonous carbon monoxide which is thus introduced into the enriched gas has naturally led to apprehension, whilst the use of carburetted water-gas has certainly given rise to serious loss of life. The substitution of a comparatively non-poisonous and highly illuminating gas, such as acetylene, for enriched coal-gas may be one of the solutions of the attempt to furnish the public with gas of high illuminating power.

Those who are interested or concerned in another important problem of applied science—the disposal of our town sewage—will watch with interest the development in recent years of the process of treatment of sewage by artificial filtration through coke or other suitable substances. This filtration is known to secure the oxidation of the putrescible matter of the sewage indirectly through the intervention of bacteria. Perhaps the nearest parallel cases are the change of sugar into alcohol and carbon dioxide under the influence of the yeast cell; followed by the production of vinegar from the alcohol of malt liquor or "must," under the influence of the *mycoderma aceti*, which has long been known to be the organism necessary to transfer the atmospheric oxygen to the alcohol. Indeed, just as it is found necessary that the saccharine liquid shall be sown with the yeast cells, and the alcoholic liquid shall become sown with the *mycoderma*, before alcoholic fermentation and acetification can take place with rapidity; so it is found that the bacterial filter for sewage must become infected with the bacteria necessary for the liquefaction of the suspended organic matter of the sewage, and with those suited to effect the oxidation of the dissolved organic substances, before it is in a condition to purify the sewage. In experiments conducted on the small scale, a double filtration of raw London sewage through two small coke bacterial filters has in this way effected upon the raw sewage a purification of 95 per cent. It is hoped and anticipated that experimental filters of a similar kind, but on a larger scale, which are now being started, may produce a similarly successful result, and that their action may be continuous and permanent.

### Conclusion.

And now the time is at hand when I shall delegate my duties as President to my successor. In congratulating him and assuring him of my best wishes, I take the opportunity of thanking the members of this large Association for the entirely unexpected and unsolicited honour which they conferred upon me at their annual meeting last year. I have served the Society for many years in the past, on its Council and committees in London, as well as in the important city in which we are now assembled. By electing me as your President you have conferred upon me the opportunity of higher, if not more arduous, service. No one here is conscious, as I am myself, of the extent to which the performance of these higher duties has fallen below the ideal which I have placed before me; but I trust and believe that after electing me to a position of honour which I have highly valued, you will one and all feel that I have wished and attempted to fulfil the duties which you have entrusted to me.

Mr. George Beilby said it was his very pleasant duty to propose a hearty vote of thanks to Dr. Clowes for the address he had just given them. That address contained a number of most noteworthy features, and among them the Society must be gratified at the allusions to the very successful connection in Nottingham, through the Nottingham University College, between applied science and pure science. One of the great objects of the Society was to bring into complete touch the followers of applied science with teachers and followers of pure science; and when they found a college in a town like Nottingham taking the lead in the matter of chemistry, through Dr. Clowes, who had directed largely matters of applied science, and who had all along been keenly interested in it, and was now more keenly interested in it than ever, in the position he now held in London, they felt that the Society was fortunate in having had such a president as Dr. Clowes. The President had referred all too briefly to his extremely beautiful testing arrangement for the detection of inflammable gas in coal mines—the enormous advantage of which, from the coal-miners' point of view, was already well known—and, in conjunction with Mr. Boverton Redwood, to the application of this process to the detection and measurement of petroleum vapours in the air.

Mr. William Thorp seconded. There was, he said, one point on which he would like to say a word or two, and that was in connection with their medallist. The President had pointed out to them that he and their medallist and several other members of the Society came all from the same school; and perhaps they might think that it was pressing a personal question a little too far to speak too much of this school. But he thought there was good ground for it, for that school was 20 years in advance of any other school in giving scientific instruction, and the master who gave it was a competent teacher, and not one who had simply "got up" a subject for the purpose of giving a few lessons upon it. He won, as the President had said, the affection of his pupils. In his own time, which was contemporaneous with that of their President, their teacher was always holding up to them Perkin as the great model for them to imitate, and he sent his old scholars on to a good school therefore. He supposed there was not in the world at that time a better teacher of pure and applied chemistry than Hofmann. With regard to the important underlying thread in the President's address, namely the connection between pure chemistry and technology at the present time, he remarked that there was a great deal of nonsense talked and done as to the teaching of technology. A great many of their public bodies had command of very considerable sums of money which they intended to devote to technology, but he was afraid a great many of them, with infinite care and labour, succeeded only in throwing a great deal of that money away. A great deal of the slight elementary teaching that was given even in evening schools was, he thought, pure and simple waste. They might succeed, perhaps, in making a more or less skilful carpenter or bricklayer, but that would not make an architect. They had in their medallist an instance to the contrary. He became, first of all, a thoroughly sound and competent chemist under the guidance of that great teacher, and afterwards he applied his chemistry to technical work. That, he thought, was the proper way to develop technical instruction and teaching. But to give young lads a mere smattering of teaching, and then expect them to be scientific men, was, obviously, asking too much of them. It was not their fault; it was the fault of those who put them in that position. It simply came to be a question whether it was possible to make a lad, in comparatively a few hours, do work which had taken a man, with equal and even with much greater ability, five or six years of steady work to accomplish. It was unreasonable to expect it of the young man, and the result would be unreasonable to the nation.

Mr. Earp (Cheshire County Council) said, as a representative of those public bodies who spent money in the way referred to by Mr. Thorp, he wished to dissent entirely from the remarks of that gentleman. He thought that if Mr. Thorp was himself connected with any of those public bodies he would see how difficult and troublesome the work was. As a matter of fact they did everything they could to bring about scientific and technical education for the classes who needed it.

The resolution was adopted, and

The President, replying, said he thought that Mr. Thorp probably did not allude to the body which Mr. Earp had at heart. There were public bodies and public bodies, and Mr. Thorp very judiciously did not name any of them.

### Presentation to the Medallist.

The President said it was now his pleasant duty to hand over to their medallist, Dr. Perkin, the medal to which reference had already been made. Speaking for a moment personally, he might say that his year of office as President of the Society had been a year of pleasure and gratification throughout. By residing in London, he had been able to attend to the formal business of the Society better than if he had been living in a provincial centre. He could assure them that the performance of his duties had given him the greatest possible gratification, but he must range far above that the pleasure he felt in handing over to his old schoolfellow, Dr. Perkin, the medal of the Society. If they had chosen their medallist to suit the year of his presidency they could not have chosen him more happily than they had done. In handing the medal to Dr. Perkin, he was quite sure he might add to the remarks which he had already made, the pleasure it would give every member of the Society to think that the medal rested in such very worthy hands.

Dr. W. H. Perkin, in returning thanks, expressed his very high appreciation of the honour they had done him, and at the same time he also thanked the President for the very kind manner in which he had referred to his early career and his work in connection with scientific and industrial chemistry. From his earliest acquaintance with chemistry, and whilst a schoolboy, the idea of working at experimental research, and the possibility of making scientific discovery, became a leading thought in his mind, and since then he had found practically how important was research, not only from a scientific point of view, but also from a technical, so that he had as far as in him lay sought to impress others with its value and the great importance of cultivating it. As he had mentioned on previous occasions, one of the greatest fears he had, when he was led into technical work, was that it might make it impossible for him to continue to follow scientific investigation, though he determined to do so if possible, because in those days very little research, properly so called, was carried out in chemical works, very few even possessing laboratories. But now it was gratifying to find that its practical importance was by many manufacturers more fully realised, so that it was getting understood that it was absolutely necessary, if progress was to be made, that a chemical works should be a place of research as well as a factory. This state of things had been brought about by the coal-tar industry, which was the most remarkable evidence of the value of scientific research to technical work that ever existed; in fact, had it not been for research, the very raw materials required in this industry would not have been known. Its birth also took place during the prosecution of research and its wonderful development had also been due to research. It was also very useful to remember that whilst this industry had been so greatly benefited by this means, it had in return most bountifully furthered science by handing back to it new materials for investigation which could not otherwise have been obtained, and also by bringing forward new problems to be solved, and by doing this had been again enriched with fresh fruit from science, so that technical and scientific work had gone together, hand in hand, to an extent never before experienced. This condition of things had now spread to other chemical industries. There was also another interesting matter connected with the influence of the coal-tar colour industry on technical chemistry, and that was in relation to chemical plant. At the time when this industry was commenced the plant used in chemical works was mostly of a crude nature, and none of it was suitable for this new industry, where the products were costly, the operations delicate, and in many cases the chemicals used were of a very active and corrosive character. This necessitated the invention of new plant of a much higher order than that in use at that date, requiring for its construction a good knowledge of chemistry and physics, as well as of engineering, &c., and the use of this higher kind of plant in the colour industry had had a large influence in improving the character of that now used in chemical works generally. Much more might be said in relation to the subjects he had referred to, but this was not the occasion to do so. He alluded to them, however, because it was a great satisfaction to him to see the wonderful spread of scientific work and the great advancement of technical work which had taken place, directly and indirectly, by the development of the coal-tar colour industry, the foundation of which fell to his lot.

The President said he was sure they would all regret that Dr. Perkin had shortly to leave them in order to return to London. They would, however, be pleased to hear that he was to receive a further mark of esteem that evening; he was to be presented by the Leather Sellers Company, of which he was a Past Master, with his portrait, as a token of their esteem for him.

## The Council.

The Secretary announced that the election of the Council had resulted as follows :—

*President :*

George Beilby.

*Vice-Presidents :*

Prof. F. Clowes, D.Sc.
George E. Davis.
John Heron.
David Howard.
Prof. A. K. Huntington.
Dr. Charles A. Kohn.
Ivan Levinstein.
A. Gordon Salamon.
Dr. W. Jay Schieffelin.
Dr. Edward Schunck, F.R.S.
Wm. Thorp, B.Sc.
Thomas Tyrer.

*Ordinary Members of Council :*

R. Forbes Carpenter.
Sir John Evans, K.C.B., F.R.S.
Sir David Gamble, Bart., C.B.
W. Winwood Gossage.
Oscar Guttmann.
Prof. J. J. Hummel.
Dr. Rudolph Messel.
B. E. R. Newlands.
John Pattinson.
Sir Robert Pullar.
Walter F. Reid.
E. C. C. Stanford.

*Honorary Treasurer :*

E. Rider Cook.

*Hon. Foreign Secretary :*

Dr. Ludwig Mond, F.R.S.

Mr. G. Beilby, in acknowledging his election to the presidency, said he looked upon the honour as the very highest that could be paid to a follower of applied chemistry, and he accepted it very gratefully as such. He looked forward with very great diffidence to the year that was before him, and he did not think he would have undertaken it at all but for the very hearty way in which his colleagues on the Council had always received him. Their kind reception that day encouraged him to try to do his best for the Society during the next 12 months.

## Auditors.

On the motion of Mr. G. N. Stoker, seconded by Mr. R. B. Batty Messrs. Miall, Wilkins, and Co. were re-appointed auditors at a fee of 10*l.* 10*s.*

## Imperial Institute Representative.

Mr. Boverton Redwood proposed the re-appointment of Mr. E. Rider Cook as the Society's representative on the Imperial Institute. They all knew, he said, that in Mr. Rider Cook they had one of the best representatives that the Society could possibly have.

Dr. Kohn seconded, remarking that Mr. Cook had always been their representative on the Imperial Institute, and they could not replace him by a better man.

The motion was adopted.

## Next Year's Meeting.

Mr. Newall, speaking on behalf of the Newcastle Section as its chairman, invited the Society to meet next year in Newcastle. It would be, he said, 15 years since the previous meeting of the Society took place in Newcastle, and there had been many changes in the Society since then. He trusted they would be able to show the Society that there were important industries in the North of England.

Mr. Saville Shaw said, as secretary of the Newcastle Section it was his pleasure to support the invitation which its chairman had conveyed to them. He thought that members of the Society who visited Newcastle 15 years ago had retained so pleasant memories of the town that they would like to repeat their visit.

The invitation was accepted, the President remarking that he was one of the members who visited Newcastle 15 years ago, and he might say they saw more in regard to the chemical industry in its most varied forms than in any Section he had since visited. He had not the slightest hesitation in saying that the members would not only be admirably treated as guests, but they would see chemical industries there on a scale and in a variety such as they would scarcely see in any other part of the Kingdom.

## Amendments to Byelaws.

The President proposed the adoption of amendments to byelaws, of which notice had been given.

The Secretary, at the request of the President, read the proposed amendments. He explained that if the number of persons nominated to fill vacancies on the Council exceeded the number of vacancies, a contest would take place as at present, and a ballot would be taken. But it had happened for some three or four years that there had not been sufficient members nominated even to fill the vacancies. Under those circumstances, the change in regard to the amended byelaws would then come into operation.

Mr. Tyrer seconded the President's proposition. The proposed change, he said, practically meant a saving to the Society of over 10*l.* They did not want contests in connection with the Society, and they did not want ballots that were unnecessary.

Mr. Carulla said he rose with considerable diffidence and regret. When he came to the meeting it was with the idea that the proposed change would be a grave mistake. He had ascertained that others shared that opinion, and that had determined him to throw himself into the breach. There was a danger, if the proposals were adopted, of the rights and privileges of the ballot being lost sight of.

Mr. Beilby said he thought they could not take too much care that the Society should remain as it had been in the past—truly representative; that every member, whether in the provinces or in London, should feel that he was contributing to the arrangements for the government of the Society. He should be the last to advocate anything in the nature of a change from that. But it had never occurred to him that the proposal before the meeting tended in that direction. The point he wished to make was that two months before the meeting every member received an intimation that it was open to him to join with others in nominating persons for the vacant places. He was duly apprised so that the opportunity could not drop out of sight in the way suggested.

Mr. Thos. Tyrer wished to point out to Mr. Carulla that there was no such place as London in regard to the Society of Chemical Industry. He contended that a ballot test was an absurdity if it was not necessary. He hoped they would hear the last of any differentiation between London and the provinces.

Mr. Wm. Thorp looked upon the question of elections as of very great importance. He agreed that it should be perfectly fair and open, but on the same ground he would like to put it in another way. He would point out that the proposed alteration made no change whatever in their procedure until they came to the point whether ballot papers should be sent out or not. Up to that point they would proceed precisely as they have done hitherto. At that point the Council had to consider whether they should send out ballot papers, which circumstances had shown would be practically useless. It was now proposed to give them the opportunity of avoiding the expense. At the same time if any considerable body of members thought there was anything mysterious about it, or that any harm would be done, he would say by all means let them go on as they had been going.

Mr. G. E. Davis regarded the matter from a financial point of view. The proposed alteration would make no difference in regard to the procedure and method of electing the Council, but it would save the Society from 10*l.* to 15*l.* per year. They wanted to accumulate as large a reserve fund as possible with their small subscription and their large expenditure on the Journal, and he certainly thought if they could do it they ought to do so.

Mr. Redwood said in what was proposed there was no possible curtailment of the existing rights and privileges of members, and therefore, with that assurance, he trusted the resolution would be carried unanimously.

Mr. Carulla: Perhaps it will be well if I say, after this discussion, I am convinced of the mistake of opposing the resolution.

A Member suggested that in future the ballot lists should be sent out under a separate cover, instead of as hitherto, between the pages of the Journal.

The resolution was then put to the meeting, and adopted.

1. Rule 7. To add rule 19 to the list of exceptions.
2. Rule 19. To add "and no ballot shall be taken. If four persons only be nominated, no ballot shall be taken."
3. Rule 20. To add at beginning "If the number of persons nominated, as provided by Bye-law 18, be more than four—"
4. Rule 56. To replace the words "be at the discretion" by the words "subject to the sanction."

### Vote of Thanks.

The President proposed a vote of thanks to the authorities of the University College, Nottingham, for the use of the building in connection with the annual meeting.

Mr. James O'Sullivan (Chairman of the Nottingham Section) seconded the proposition, and it was adopted.

### The Decimal System.

The President said they had reached the end of the formal business, but there was one announcement he wished to make. The Council, at their meeting to-day, had before them a request from the Decimal Association in favour of the adoption of the decimal system in this country, and asking that members of the Society who were interested in the matter should send a petition in support of it. He might say, a petition had been already signed by every member of the Council, and he hoped this second petition now lying on the table would be signed by many more members.

This concluded the proceedings.

### Visits to Works and Places of Local Interest, &c.

After the morning's business, lunch was provided by the Section, at the George Hotel, and then a large number, with ladies, were conveyed to the residence of Alderman Sir John Turney, J.P., Springfield, Alexandra Park.

### Sir John Turney's Reception and Garden Party.

On arrival, the party was received most cordially by Sir John Turney, and entertained in his beautiful grounds, which are placed on successive terraces high up in a ravine. Some passed the time in conversation or going through the grounds, whilst others listened to the band or the singing of a quartet party. The pleasure of this, as of all the succeeding entertainments, was largely enhanced by the charming weather which prevailed.

### Reception at Nottingham Castle by His Worship the Mayor of Nottingham and the Mayoress.

*Conversazione.*

In the evening the members, together with a large number of other guests, were entertained by his Worship the Mayor, Alderman G. H. Fraser, D.C.L., J.P., and the Mayoress, at a conversazione at the Art Museum, The Castle. The guests were received by Alderman Fraser and Mrs. Fraser, together with the Misses Fraser, Lady Johnson, and Mrs. Bright, after which they were at liberty to inspect the rich art and curio treasures of the Castle, Mr. Alfred R. Watson's orchestral band meanwhile contributing a tasteful and well-selected programme of music. Music was also provided on the green by the police band for those who cared to enjoy a stroll through the grounds, the instrumentalists, under Mr. Albert Redgate's direction, performing a number of familiar selections. Light refreshments were provided during the evening in the water-colour gallery and in the refreshment room on the ground floor.

Among the guests of the evening were their Graces the Duke and Duchess of Portland.

## SECOND DAY.—Thursday, July 14th.

### Visit to Sheffield Steel Works, &c.

By special train from Nottingham (Midland Station) at 9.30 a.m., the members proceeded to Sheffield, arriving about half-past 10. On arrival they were divided into four parties, which were conducted to the following works:—

### The Atlas Works of Sir John Brown and Co., Ltd.

*Conductor:* Mr. S. J. Pentecost.

Here the members were shown the manufacture of armour plates—pressing, rolling, and finishing; the manufacture of marine forgings; tyres, springs, buffers, &c.; and also the blast-furnaces.

The Atlas Works are situated in the Brightside district of Sheffield, about a mile from the centre of the city, and cover an area of about 32 acres.

These works were originally purchased by John Brown and Co. in the year 1854, and ten years later the present Limited Company was formed, with Sir John Brown as chairman.

Messrs. John Brown and Co., Limited, were the first to take a license from Sir Henry Bessemer for the manufacture of steel on his process, and fought the up-hill fight of the introduction of Bessemer steel rails, tyres, and axles, which have since been used by nearly all the leading railways. Although a large business is still done in railway tyres and axles, the Bessemer plant has recently been dismantled to make room for extensions in the armour-plate department. The firm's armour-plate manufacture commenced in 1859; they may therefore be considered the pioneers of the armour-plate trade of this country. The manufacture commenced with wrought-iron plates rolled to 4 ins. in thickness, afterwards increased to 24 ins. In 1877 the Company started the manufacture of compound armour on a system patented by Mr. Ellis, and from that date until 1893 this class of armour was adopted in the construction of the largest battleships and cruisers, both for this country and abroad.

At the present time steel armour alone is being supplied for the protection of battleships and cruisers, and John Brown and Co., Limited, were the first to produce armour plates with faces of the excessive hardness now considered indispensable. Their patents, which have reference to the final or chilling stage in the manufacture of hard-faced plates, and are known as the Tresidder patents, have been acquired by the Harvey Company and incorporated with the Harvey and Krupp processes. The efficiency of plates treated by the new processes has enabled naval architects to reduce the thickness and weight by about one half; and to keep the supply of armour up to the estimated output of 10,000 tons per annum, the Company has for some time been making considerable additions to the plant, and important extensions are now in hand in connection with the machine shops. Electric plant has been installed, and all the new machinery will be electrically driven.

For the production of armour plates several large Siemens furnaces are constantly employed, the ingots being cast in special moulds and weigh from 30 to 50 tons.

In the case of plates not exceeding about 9 ins., the ingots are pressed into flat slabs of suitable dimensions in the forging-press department, where two large and powerful hydraulic presses, exerting a pressure of 4,000 and 10,000 tons respectively, are erected (the latter doubtless excelling any tool of its kind at present in existence), and, after pressing, are rolled to finished thicknesses in the rolling mills. For thicker plates the slabs are pressed by the larger press to the finished thickness, and rolling is dispensed with. These presses are also used for marine shaft forgings, gun forgings, &c., the Company also being engaged in the manufacture of marine forgings of the largest dimensions.

The "Purves" patent flue shop, with its interesting special machinery, was commenced in 1887, and occupies the site of a large iron forge. These flues have been received with favour in all parts of the world, and the demand has been a constantly increasing one.

The large hydraulic press for flanging marine boiler end-plates can deal with a plate 20 ft. by 10 ft. at one operation, and, except for "reverse" flanges, the flue holes and outside edge are flanged at the same time. The "serve" tube is one of the latest specialities taken up by the Company, and has led to a very important improvement in boilers for use both on sea and land. A much smaller number, or smaller-sized boilers, give, it is claimed, by the "Ellis and Eaves" suction draught combination, the required steam more economically than in the old boilers, without risk to the boilers, without smoke, and with much greater comfort to the firemen. Vessels thus fitted are giving great satisfaction.

The Company have three blast furnaces, two of which were completed and blown in during 1872, and the third in 1874. They are capable of an output of about 1,000 tons per week, and the ore is principally brought from the mines belonging to the Company.

The number of hands employed at the Atlas Works is about 4,000.

### CYCLOPS STEEL AND IRON WORKS (MESSRS. CHARLES CAMMELL AND CO., LTD.).

*Conductor:* Mr. R. M. CAVEN.

Crucible furnaces. Steel for high-class tools and instruments. Machine- and hand-cut files. Sand-blast process in operation. Spring steel. Tool steel. Bar steel. Rail mill. Rolling fishplates and billets.

Hammers:—No. 6. Reducing ingots. No. 8. Forging wagon axles. Nos. 16–17. Forging locomotive engine and tender axles.

Armour-plate rolling. Harvey furnaces charged. Planing shops. Finishing plates. Chilling armour plate.

This Company owes its origin to the late Mr. Charles Cammell, and it was established by him, in partnership with M. Thomas Johnson, in the year 1837.

In 1861 the manufacture of steel rails was commenced at the Cyclops Works. Two years later attention was turned to the production of iron armour plates. On steel being exclusively adopted for the production of heavy ordnance, Messrs. Cammell successfully entered into the business. They were also the first to introduce into England, in 1863, the system of forging steel ingots by hydraulic pressure. Quite recent experiments having proved the greater resistance of mild homogeneous steel "Harveyed" armour plates over other systems of production, Messrs. Cammell erected plant and machinery for the manufacture of this class of armour. The Cyclops Steel and Iron Works cover an area of 11 acres; the Grimesthorpe Ordnance, Steel, Tyre, and Spring Works cover an area of 22 acres; and the Derwent Iron and Steel Works, Workington, cover an area of 84¼ acres. Throughout all these works the Company employ about 10,000 men.

The following is a brief sketch of the manufacture of an armour plate. After the process of melting the charcoal pig iron and mixing of the alloys, which occupies about 14 hours, the steel is run into a huge mould to form the ingot, weighing about 56 tons, which is carried to the forging press from the casting pit along a tramway. The press shop is an exceptionally fine building, and is known in the works as the "cathedral." The press is worked by hydraulic pressure of 2½ tons to the square inch, and has two rams. The ingot already mentioned requires about 24 hours' careful heating before it can be dealt with under the press. Beneath the rams the enormous steel ingot is as plastic clay. When reduced to the requisite thickness, the slab is conveyed to the rolling mills, there to be reheated and rolled to the finished thickness of 12 ins. It is then placed in a cementation furnace for carburisation on the exterior surface. The carburisation of the face is essentially an adaptation of the old Sheffield cementation process, whereby the surface of the metal, in contact with charcoal substances and exposed to the application of great heat, absorbs a large proportion of carbon from the charcoal coating, and consequently becomes much harder than the rear surface of the plate, which has been protected by some non-carboniferous substance. The armour plate, however, having been finished as far as forging, rolling, curving, &c. is concerned, is taken to another department to render it fit for erection as part of the ship's structure. The first operation as the plate comes from the forge is to cut off the rough ends. This is done in heavy breast planing machines, in general appearance after the pattern of an ordinary plate edge planing machine. The armour plates are fixed to the structure of the ship by studs or bolts which screw partly into the plate. The holes are drilled and tapped by means of some powerful machines specially designed. The bolts for holding the plates in position are 5¼ ins. in diameter and have a special thread 3½ turns to the inch. The time occupied in making one hole and cutting the thread is about two hours. After the edges are cut the plate is run on a bogie to the fine range of furnaces near the machine shops, and heated in one of these for a period varying with the size of the plate, &c. At a suitable heat the plate is withdrawn, and the carburised face chilled with a douche of cold water forced through numberless apertures on to the surface of the plate. Finally it is again taken into the machine shops to be fitted complete.

There is a crucible steel department, a Siemens department, and a Bessemer department. In the latter, rails, angles, tees, and fishplates are rolled.

On May 21st of last year, Her Gracious Majesty the Queen witnessed the rolling of an armour plate in these works.

### RIVER DON WORKS OF MESSRS. VICKERS, SONS AND MAXIM, LTD.

*Conductor:* Mr. J. T. WOOD.

The visitors were here shown the rolling of tyres, the forging of gun parts, &c., under presses and hammers, the finishing of shafts and guns of large calibre, and the processes of armour-plate finishing.

Vickers, Sons, and Maxim, Ltd., are one of the few firms which build ships and the armaments therefor.

The first process seen was the rolling of a 50-ton, 12-inch armour plate made of nickel-steel for the "barbette" of H.M.S. "Canopus." The bloom, originally 31 ins. thick, from which it was rolled, was heated in a gas-furnace, whence it was taken by a travelling crane to the roller, 3 ft. in diameter, working over a rolling bed. After each passage, the roller was depressed about an inch by steam power. During the operation, wet brushwood was thrown upon the bloom to explode off the scale by its sudden combustion. On completion, the plate was taken to the cutting shop on a special truck drawn by a locomotive, and there the end was sheared off by powerful knives placed above and below the plate, and pressed through it by a powerful hammer. The end so removed was that portion of the bloom which contained the holes and imperfections of the casting.

The armour-plate shop (480 ft. long, with two bays of 70 ft. wide, each bay containing three 60-ton cranes) and the gun-building pit were next visited. Next, armour plates, 12 ins. in thickness, showing the effects of three 12-in. shots, fired at them from a distance of 120 yards, and a 4-in. plate showing effects of three 6-in. shots, also at 120 yards, were shown. The shots were of the Holtzer type, manufactured by Firth: and it may be here said that there is a perpetual contest being waged between armour-plate makers and gun and shot makers for supremacy, and each tests efficiency by the other.

The general machine shop, tool smithy, and gun shop, were next visited. In the last, a 10-ton breech was being hammered. Hammering is, however, being abandoned in favour of press-forging. A 4,000-ton forging press was engaged on a 14-ton gun at the same time. In this shop, the machinery was worked by electro-motors. Breeches and mechanism for opening and closing them were also shown. Also a 12-in. gun, 40 ft. long.

### Messrs. Samuel Osborn and Co., Clyde Steel and Iron Works, Sheffield.

*Conductor:* Mr. H. J. Staples.

Messrs. Osborn and Co. are contractors to the War Office and Admiralty, manufacturers of steel castings to Government and Lloyd's specification, and supply material to many of the chief English, colonial, and foreign railway companies.

The visitors were here shown the manufacture of crucible steel, railway springs, tool steel, &c.

Rutland Works, at Neepsend, was acquired in 1885 to enable the firm to meet the needs of a rapidly growing trade in steel castings. Visitors were conveyed to these works, situated on the left bank of the Don, about a mile above the Wicker Works, and cover a spacious area, the premises being designed throughout with every convenience for a modern steel foundry. At the Rutland Foundry, the Siemens-Martin furnaces and moulding and steel founding were demonstrated, and a large casting was poured. About 800 hands are employed in these works. The firm are sole makers of R. Mushet's special steel for lathe and planing machine tools, as well as for many special purposes.

At Clyde Works there is a department for the manufacture of laminated springs for locomotives and railway carriages and wagons. At another department, which has recently been considerably extended, the various articles used by agricultural machine makers are made, such as finger bars, knife bars, rake-tooth steel, plough plates, knives for reaping and mowing machines, chaff knives, turnip cutters, &c., also circular saws for wood, hot and cold iron, and other purposes. The steel foundry has a large trade in steel castings, of infinite variety, weighing up to 15 tons each. For instance, castings required by locomotive builders, such as wheel centres, crossheads, horn blocks, roof stay bars, motion plates, &c. A speciality is made of steel gearing, with straight or helical teeth (the foundry being specially well equipped for turning out machine-moulded wheels with the greatest possible accuracy), for rolling mills and driving purposes generally. Wheels for collieries, which are supplied both loose and fitted to axles, with bearings ready for use for tramways, light railways, contractors' wagons, and other purposes, form another large branch. Castings for crushing machinery used at gold mines must be mentioned—amongst others tappets, cams, heads, shoes and dies, crusher jaws and rollers. In addition to these, all sorts of rollers and sheaves, clips for rope driving, &c.

After the conclusion of the visits of the morning, luncheon was provided by the Nottingham Section, and on its invitation a large number of the members assembled in the Masonic Hall, Sheffield, at 1 p.m.

After the lunch, the afternoon was devoted to the following additional visits.

### Messrs. Walker and Hill's Silver-Plating and Cutlery Works, and Show Rooms.

By kind permission of Lieut.-Colonel Bingham.

The following is a list of the different departments seen: Sheffield show room, which contains thousands of pounds worth of stock. Here were seen the first plating-vat ever used and various other processes of interest.

*Boil Shop.*—Preparing goods for plating.—Here goods are freed from grease, &c., by means of boiling potash leys, and then scoured with wire brushes driven by steam. Plating rooms—At one end of these rooms George Walker, the founder of the firm, first commenced business. George Walker was the first man who plated any goods for use. The first vat (size 12 ins. × 8 ins. × 4 ins.) in which he plated these, is to be seen in the large show room. 26,000 quarts of silver solution are now in use in these rooms. Scratch brush and cleaning room. Spoon and fork punching and stamping shop.—The machinery in this room is capable of producing 4½ tons of spoons and forks per day.

*Table Blade Forgers' Shops.*—Making steel blades from best double shear steel by hand. This process ensures better wear and value than the cheaper "tripped" or rolled processes, so frequently used on common material. A blade-grinders' wheel, working on grindstones, glazers, and buffs. Pneumatic hammer and wheeling shops. Spinning shop, where XXX alloy teapots, dish covers, &c., are shaped under the practised eye of the workman while rapidly rotating. This separate Sheffield branch of work is particularly interesting to strangers, as teapot shapes, &c., come into existence from a flat disc of metal, controlled almost entirely by the practised eye and skill of the workman. Nickel silver ingot casting furnaces, where nickel is mixed with other ingredients at a higher temperature than molten steel; then, in order to make it white, it is again cast, and special ingredients added to make it malleable enough, or, as Sheffield silversmiths call it, "kind" enough, to be worked. Best nickel silver should be white. Common or yellow is known as German silver.

Each member was presented with a handsome watch-chain pendant in commemoration of the visit of the Society.

### The Ruskin Museum at Meersbrook Park.

Interesting collections of minerals, drawing prints, paintings, &c., presented by Mr. Ruskin.

The Mappin Art Gallery, the Sheffield Technical School, and Firth College were also thrown open to the visits of members of the Society.

### Alternative Local Visits in Nottingham.

The visitors desiring to see these works, returned from Sheffield by the 2.30 p.m. train.

### Thos. Adams and Co., Ltd., Sherwood Hill Works. Lace Bleaching, Dyeing, and Dressing.

*Conductor:* Mr. S. J. Pentecost.

Visitors to Messrs. Thos. Adams and Co.'s, and Ed. Cope and Co.'s works, which are close together, were conveyed to the works in a brake provided by the Nottingham Section.

Entering the works on the south side, the Brown Room was entered, where the cotton goods are prepared for bleaching. They consist principally of curtains, blinds, mosquito nets, cotton edgings, and laces. Those to be treated in the cisterns and dollies are tied up with coarse string; those to be bleached by the continuous process are stitched end to end in one length. The former, after boiling, &c., are treated for the removal of dirt and plumbago, which is largely used as a lubricant for the bobbins and carriages in the lace machine. The long string of lace pieces is next made to pass through the various washing, chemic, and scouring machines in a continuous length. The boiling takes place in large kiers, in which the goods are carefully packed to prevent entanglement.

After witnessing the process of dressing lace curtains, the taping machines were seen at work scalloping and braiding the edges of the lace curtains, which are then folded, pressed, and parcelled. The lace is next stiffened, dipped, and squeezed. Proceeding to the dyehouse, the dyeing of silk lace and veilings, as well as cotton laces and glove fabrics, was seen. Owing to the delicacy and lightness of the fabrics, dyeing of lace goods is done by hand in dyebecks with winches, &c., special care being required to prevent damage. The water supply is pumped from wells about 230 ft. deep, the quantity used being about 120,000 gallons per day. All the soaps used in bleaching and dyeing are manufactured on the premises. It may be interesting to know that a religious service is held for half an hour every morning in a chapel built over the entrance to the works, which is attended by nearly all the employees and is much appreciated. It was established by the Directors of the Company in 1876.

### Edward Cope and Co., Ltd.

The business was started by Mr. Wm. Cope at what was then known as Taylor's Factory, in Broad Marsh, Nottingham, in the year 1843. His first machines were converted from traverse and plain net machines, the pattern being produced by the means of bars with the aid of nogs fastened on the cards and other devices. In 1850–51 he built machines expressly for curtain work with jacquard application. In 1855 the firm Cope and Ward shortly became known as occupying a leading position in the trade as lace-curtain manufacturers, and in 1856 they purchased the factory and business of Messrs. Robinson and Sisling, at New Basford, in which the business, after various enlargements, is now carried on. To Mr. Thos. Robinson, the manager, belongs the honour of having produced the first authentically recorded lace curtain of an elaborate character, for which he was accorded a gold medal for a "machine-made lace curtain of excellent quality and graceful design," as described in the Reports of Juries published by the Royal Commission (p. 560, 25*a*). On the death of Mr. Wm. Cope, in 1868, the firm became known as "Ward and Cope."

Later on the business was purchased by Mr. Ernest Jardine, the present proprietor. He has amalgamated with the old business that of a large plant of hosiery machinery of the latest construction.

### John Jardine, Deering Street, Nottingham.

At the Deering Street Works, visitors were enabled to see lace-machine building and harness making; at Moscow Works, lace-machine building, with iron and brass casting, and printing-machine making; at the Raleigh Street Works, the delicate parts of lace machines.

### Raleigh Cycle Works, Lenton, Nottingham.

*Conductor:* L. Archbutt, Esq.

These works at Lenton, a suburb of Nottingham, were established in 1896, though the Raleigh Cycle Co. was founded in 1886. The building now covers about $6\frac{1}{2}$ acres, and the firm employs about 1,400 men. The whole process of cycle manufacture was here demonstrated.

### Messrs. Turney Brothers,
### Trent Bridge Leather Works, Nottingham.

*Conductor:* Mr. J. T. Wood.

The visitors were most kindly received, and shown through the works in parties by Sir John Turney himself, and his sons, with Mr. J. T. Wood.

These works, established in 1862, are of large extent, covering, with yards and approaches, the whole area of a considerable island surrounded by canals and the river. Sir John Turney is the managing director of the firm.

On entering the central yard there are seen three great self-feeding furnace boilers, transmitting power to 16 engines, inclusive of that driving the dynamos of a complete electric light installation. The skins, after being sorted and thoroughly inspected, undergo liming. In the pits, about 30,000 skins are treated per week, and the sheds have a capability of dealing with 90,000 skins at a time.

The next operation is performed by the "cobbers," who clear the skins of any wool or hair yet remaining, whilst the "fleshers" perform a like service in cleaning the inner of flesh; in this work the skins are placed over a wooden "beam," and worked with knives by experienced operatives. The matter that is taken off is used in the manufacture of gelatin, glue, and grease.

The skins are next split by machines. The outer, or "grain" side is, when separated, known as the "skiver," and, when tanned, makes light leathers, and the inner flesh side is converted into chamois. The knife used for splitting makes 1,500 strokes a minute. After this process the respective skins take different lines of treatment. The "skivers," or outer sides, are washed to remove the lime, and then "bated" and "drenched," great skill and care being required in the process. Sumac and larch bark are mostly used in the tanning of light skins. The sumac is imported from Palermo.

In the drying rooms over 50,000 skins are in process of drying or sorting, and many floors of several warehouses are devoted to these purposes. In some of the drying rooms a current of air accelerates the drying, the state of the atmosphere having a considerable effect on the time required. In the warehouses were seen specimens of larch-tanned "basils," which are prepared from the finest sheepskins only, for fancy leather work. Skivers are used for hatbands and purse linings. A special building is devoted to the purpose of freeing the skins from grease by means of special machinery. The dyeing factories occupy another wing of the buildings.

There is a considerable difference between the treatment in making skivers and chamois leather. For the latter, the skins, after cleansing and washing, are placed in large hydraulic presses. Under the weight of $2\frac{1}{2}$ tons to the square inch, all the animal grease exudes from the plates whereon the skins are laid. The grease is practically pure mutton suet, and is used in the manufacture of the best soaps. The next process is that of "milling." Fish-oil is the medium used, and the skins are thoroughly impregnated with the oil in large fulling mills. After the various processes, all superfluous oil is extracted by hydraulic pressure. This, which is known as "sod" oil, is used by curriers of leather, and a large trade is carried on in this substance alone. In the glacé kid department are powerful glazing machines, examples of the work of which were seen in the warehouses. The finest chamois leather is used for suède gloves. The number of hands employed is about 500.

### The Annual Dinner.

The annual dinner was held on Thursday, July 14, at 8 p.m., in the Exchange Hall. The President, Prof. F. Clowes, D.Sc., occupied the chair, and among those present were Mr. Geo. Beilby (Edinburgh), Dr. C. A. Kohn (Liverpool), Mr. Thos. Tyrer, Mr. G. E. Davis, Mr. L. Archbutt, Mr. Wm. Thorp, Dr. J. Lewkowitsch, Mr. W. F. Reid, Ald. Fraser, D.C.L. (Mayor of Nottingham), Sir John Turney (Nottingham), Mr. A. W. Black (sheriff), Lt.-Col. Bingham (Sheffield), Mr. Douglas Herman (Liverpool), and others.

The President proposed the toast of "The Queen and Royal Family," and said that, although they were not a royal society, and at present, unfortunately, possessed no royal charter, he was quite sure that would make no difference in their feelings towards Her Majesty and the Royal Family. They were quite aware that the late Prince Consort was a man who was much interested in pure science and applied science, and the Queen agreed with him in taking his very liberal estimate of scientific men and their work, while the Prince of Wales and other members of the Royal Family had been desirous of copying the example of their illustrious father. If they wanted a reason for being loyal that evening, they had only to look at those facts; and perhaps they might add that there were members of their Society who had received marks of royal distinction. He would not venture to give them a list of the numerous gentlemen in that happy position, but he need only remind them that they had one among them that evening in Sir John Turney.

The toast was duly honoured.

Mr. **Beilby**, who was most cordially received, then submitted "The Society of Chemical Industry." He thanked the members for the great privilege they had extended to him in allowing him to propose this toast, as the Society of Chemical Industry and its affairs had always been very near his heart. Moreover, he had always believed so fervently in the Society as a society, and in its possibilities, that it gave him great joy to take part in any function in which the Society was magnified. He thought that, in looking forward to what was before them, they had every reason for the very highest and brightest hopes, and if each of them took home to their various sections a strong spirit of hopefulness and a very strong belief—such a belief as he was personally very glad to confess to—in the Society and in the possibilities of making it even greater and more successful than it had ever yet been, then that meeting of theirs would not have been unavailing. In looking forward to what was before them in the coming session, one allowed oneself to wonder in what directions they might hope to move forward a little, and among the points that had struck him more than once had been the thought that the younger members of the Society were not being quite sufficiently encouraged to come forward and take that active part in the Society which might have a most beneficial effect in the importation of new beliefs and new ideas. He thought that in their sectional work, if they could feel that it was a duty upon them more and more to encourage the young men, to take notice of them, and to bring forward all those who were worthy of being brought forward, he thought that they would be doing an extremely good work, not only for the Society, but for the young men. He also agreed with the remarks that fell from the President in his address, and those which followed, drawing attention to the great value that attached to the Society as a meeting-ground. They had within their ranks the purely scientific man; they had also the academic or teaching man; and they had the practical man, all meeting withing their borders; and the result of such intercourse as occurred among the different classes of men could not but have a most beneficial effect. And it was for them to ask if they had made the fullest use of that in the past—whether it was not possible in their future meetings and gatherings for them to get even greater and greater benefit out of that possibility of the Society. In asking them to drink the toast, he coupled with it the name of the President. Those of them who had had the pleasure during the year of sitting in the Council would miss him much when he left them. They had got used to, and to expect, his kindly presence in the chair, and he was quite certain that they would feel a blank for a long time to come.

The toast was drunk with musical honours.

In reply, Dr. **Clowes** said that Mr. Beilby had valued him and his work much too highly, as it must be remembered that the work of the Society did not depend upon any president or presidents, but was done by a very capable Council, and by very capable local committees and sections, and that it was owing to the fact that all those important bodies worked together harmoniously, and had, as far as necessary, supported their president, that the year had been the successful year it had been. Speaking of his connection with Nottingham, the President remarked that he could not forget that he had spent the most active part of his life in that city, and the University College Committee had, very kindly, taken care that he should not forget the fact, by making him Emeritus Professor of the University College, a position which he very much appreciated. After an allusion to the Nottingham Section, with a brief recapitulation of the gentlemen who had been most actively connected with it, Prof. Clowes thanked the Mayor of the city (Alderman Fraser, D.C.L.) for the reception that he had given the Society in the name of the city, and commented upon the fact that since they were in Nottingham last his titles had been extended by the appellation of D.C.L., an academic title of great antiquity, conferred by the Archbishop of Canterbury and confirmed by Her Majesty the Queen, while the raising of the town to the rank of city, entitled him to be denominated not simply his Worship, but the Right Worshipful the Mayor. Turning to the Society itself, some suggestions had been made by Mr. Beilby with which he thought they would all cordially agree, and he hoped that that gentleman would fulfil the pledges he had given to them, and would see that the younger members were, during his year of office, brought more prominently to the front than during the presidency of any former gentleman. The suggestions were eminently good and practical, and all would hope to see them carried out. He might remind them that not only was the Society active when it met once a year on those occasions, but there was work always going on which those who were much concerned in the affairs of the Society knew of. They must remember that the Council was a busy body, that in its council and committee meetings it was constantly at work. Again, not only was the central council, and the committees derived from that, busy, but so too were the committees meeting in the different sections. That was not the one function of the year, as was sometimes the case in other societies; that was only a holiday or social time, when they, once a year, met together and tried to get to know one another. He had been very much struck, during the time he had presided over the Council meetings, with the support which the old members, many of them original members, had given to the Society. It would be very natural that, as gentlemen felt that they had served for a proper term of years, they should draw back and leave the work to the juniors. But that was not the case, and he could easily name five or six gentlemen who were constantly in their places at the Council meetings, helping forward the business of the Society since the time of its formation. He was sure they would all grant that that was an important element in the success of the Society. He would not attempt to give a list, because it would be incomplete, but there was one who might perhaps be named without any gentleman feeling aggrieved. He referred to Mr. Tyrer, who had served the Society in every possible capacity, and some of them would wish that his term of service might even go the cycle once more. He was a man of great experience and one who had unbounded interest in the progress of the Society, and he (the speaker) knew, as other past presidents knew, how on many occasions he had saved them much serious trouble, and even disaster. In conclusion, he hoped that the Society might progress and continue to increase as it had done in the past.

Mr. **Thomas Tyrer**, in proposing the toast of the "City and Industries of Nottingham," said that he appreciated to the fullest degree the too kind words in which the President had referred to any work of his in connection with the Society. But he thought it necessary to repeat, what was more than once recorded, *viz.*, that to the first general secretary of the Society—Mr. George E. Davis—supported by eminent colleagues on the Council, belonged the credit of outlining the representative constitution of the Society, and urging the establishment of the Journal. Without these services then rendered much that had since been done could never have been accomplished. No section of their great federation was greater than any other; each was equally represented in the Council by its chairman and secretary, and every member, wherever he might be, had absolutely equal rights. Nottingham was no mean place. It had a debt of about 4,000,000*l.*, and as seen to-day and seven years ago, it could show a good deal for the money. Unlike some vast communities, its gas and water were municipalised, and the latter had yielded a revenue in relief of rates to the extent of 12,000*l.* in 10 years. Its history was not of yesterday, as doubtless the Right Worshipful the Mayor could testify. Nottingham had been usually associated with the lace and hosiery industries. As upon other towns, periods of depression had come—fickle fortune had smiled and frowned on Nottingham. Enormous fortunes were said to have been made by men, some of whom, ill-fitted by nature or education to be stewards of so great success, sowed more freely than they reaped. Naturally, too, the townspeople followed the example. The depression referred to doubtless stimulated the economical and enterprising, and now Nottingham was no longer dependent on the historic industries, but had numerous and flourishing manufactures—machinery, brewing, finished textiles, cigars, tobacco, and last, but not least, tanning and leather. In brief, the calamity had been a blessing in disguise, and instead of ruin there was now evidence of taste in architecture, liberality in public works, and enterprise in educational provision, as evidenced in the University College of which the president was an emeritus professor and in the technical schools. It would be a grand thing for the country when the right sort of education was applied to the right sort of learners, and means taken to extract the largest amount of good out of the enormous sums spent, he feared on the "hit or miss" principle for what was miscalled "education." His feeling in common with many better judges was that the great desideratum to-day was the educated trained employer—the trained and independently minded manager, and the obedient, sober, and skilled workman. Whatever they had of the first, they had far too little of the second, and sadly too few of the latter. That day some had seen manipulation of forces in producing armour plates which roused admiration and even envy; but it was obvious that workmen were doing what machinery could do much better, to the lessening of the risk and the increase of human comfort. It was time to awake; and one could only congratulate the city and industries of Nottingham in possessing an enlightened municipality headed by the Right Worshipful Mayor, Alderman Fraser, J.P., D.C.L., and industries of the character of that conducted by Sir John Turney, who left no stone unturned, no mechanical appliance untried, and no scientific problem unsolved to promote its successful competition. Coupled with these names, he commended the toast to their acceptance.

The Mayor of Nottingham responded, and said that he thought that he could, without presumption, claim that Nottingham was a beautiful place compared with other inland cities and towns, and that where riot and disorder once reigned in the old feudal days now existed a spot of enlightenment, a spot of culture, and a spot of education, which was at once an ornament and an example to the other municipalities of the country. With regard to the Society, he assured them that the Corporation of the city gladly acceded to his request that it should be invited to their midst. They desired to welcome within their borders all societies that made for the advantage and the comfort of human life. They recognised that that Society might powerfully influence both trade and commerce, and they were therefore pleased to see them in their midst, for they realised that over and beyond what they might do for the benefit of the trade itself and for those whose capital and enterprise were embarked in it, there remained the final object for which trade was carried on—the advancement of the comfort and happiness of the whole human race.

Sir John Turney also replied, and referred to the change that had taken place not only in Nottingham during the last quarter of a century, but in its industries. He could only add to what the Mayor had said that he hoped they had enjoyed their second visit to Nottingham, and that when they had gone the round and could find no better place to go to, they would ask the Corporation of Nottingham to invite them again, in which event they might rest assured they would do their best to make the visit a pleasing and a happy one.

Dr. C. A. Kohn (Chairman of the Liverpool Section) submitted "The Local Section," and spoke in eloquent terms of the handsome manner in which the Society had been entertained, notwithstanding the manifold difficulties that stood in the way of its satisfactory accomplishment. They had been received with an enthusiasm and a well-organised hospitality which every member of every other Section would carry home with the greatest pleasure and remembrance as a type of the hospitality that ought to be given the Society when other centres were visited. In a few comments upon the success of the meeting, and upon those to whom the credit for that result was due, Dr. Kohn referred to the labours of Mr. O'Sullivan, and hoped that the brewing interest which that gentleman represented would continue to find generous treatment in the Journal. The speaker also alluded to the importance of the different Sections of the Society endeavouring to make their meetings common ground for those interested, not only in practical chemistry, but in all branches of chemical science—for those who taught and were taught the science, for those engaged in its practical application, and for those interested in theoretical investigation.

The toast was coupled with the names of Mr. O'Sullivan and Mr. J. M. C. Paton, the former of whom, in response, observed that if the members of the Society left Nottingham with the feeling that they had thoroughly enjoyed themselves in every way the local Section would be repaid for any trouble they had taken to entertain them.

Mr. Walter Reid proposed "The Guests," and dwelt upon the hospitality that had been extended to the members by those firms who had thrown open their works for inspection.

Lieut.-Col. Bingham, in reply, reflected upon the immense amount of scientific power that existed in the Society with its 3,000 members. He took it that manufacture and science, especially the science of chemistry, were so commingled that they could not well dissociate them, and while he could not, from lack of knowledge, descant upon the influence of science in its amelioration of human suffering, he could speak of the immense assistance which it gave to manufacturers, some of whose products, even including those of several of the great Sheffield steel industries, were due in the main to the practical application of scientific investigation and research.

This concluded the toast list, and the proceedings shortly afterwards closed.

### THIRD DAY.—Friday, July 15th.

### Visit and Excursion to Lathkill Dale, Haddon Hall, and Chatsworth.

Favoured on this occasion, as on all others throughout this Annual General Meeting, with weather which for magnificence can only be compared with that of the same period last year when the meetings were held in Manchester, a large party of members and ladies assembled at the Midland Station, Nottingham, at 9.30 a.m., and took train for Matlock Bath, arriving about half-past ten. Thence the party drove by the Via Gellia, Winster, and Robin Hood's Stride to Alport, for Lathkill Dale. From this beautiful spot the drive was continued to—

### Haddon Hall.

Here lunch was served, and the party was taken through the interior of this fine relic of the Elizabethan period, which in certain of its chambers and recesses bears evidence of still greater antiquity.

The party then proceeded to—

### Chatsworth House,

the seat of the Duke of Devonshire. The visitors were here divided into parties, and shown over this beautiful mansion, where many paintings by the old masters—Raphael, Paolo Veronese, Leonardo da Vinci, Hogarth, &c.—of rare value, were seen, not to speak of such objects as the splendid sculpture gallery, the table of polished malachite, and the library of scientific works. After a stroll through the grounds, with the magnificent conservatory built by Sir Jos. Paxton, and the celebrated fountains, the larger portion of the visitors went to Baslow, where a substantial tea was provided by the Section.

The model village of Edensor, adjoining the park, in the churchyard of which Lord Frederick Cavendish is buried, was also visited.

Thus ended an Annual Meeting, of the organisation, arrangements, and general conduct of which it may be truly said, that "they were equalled by few and excelled by none" of their predecessors, the credit being largely due to the excellent secretary of the Nottingham Section, Mr. J. T. Wood.

# London Section.

*Meeting held on Monday, June 20th,* 1898.

DR. R. MESSEL IN THE CHAIR.

Before proceeding to the formal business of the evening, Mr. David Howard moved a vote of thanks to Dr. Messel for his very able conduct of the business of the Section during his term of office as Chairman. The motion was seconded by Mr. Thomas Tyrer and carried with acclamation.

In returning thanks, Dr. Messel congratulated the Section on the progress it had made during the last two years, as shown by its increased numbers, and on the fact that his successor would be able to start the coming session with a number of papers already in hand or promised. He concluded by referring to the great indebtedness of the Section to its secretary, who was now also retiring, and without whose zealous work the success which they had achieved could hardly have been secured.

Mr. J. **Heron** then proceeded to lay a statement of the work of the past session before the meeting, from which it appeared that the number of members on the register when he took office was 750, and now stood at 812. During the same period the average attendances at meetings had increased from 60 to 90, and had on some occasions reached 120, and there were indications that these numbers would be increased in the future. He had every hope, therefore, that the success of the Section would continue in the able hands of his successor.

Mr. **Howard** moved that the best thanks of the Section be heartily accorded to Mr. Heron for the admirable manner in which he had fulfilled his duties as secretary.

Mr. B. E. R. **Newlands**, as Chairman of the Section during the earlier part of Mr. Heron's term of office, desired to second the motion, as he had a very high regard for the manner in which Mr. Heron had fulfilled his duties.

Mr. A. R. **Ling** supported the motion, and in doing so said that he felt it a great honour to have been nominated to succeed Mr. Heron, and would do his very best to deserve the confidence which the members had placed in him.

The motion was then put to the meeting and carried unanimously.

## ALUMINIUM AS A HEATING AND REDUCING AGENT (IN THE PRODUCTION OF CHROMIUM AND OTHER METALS).

BY DR. HANS GOLDSCHMIDT AND MR. CLAUDE VAUTIN.

(*This Journal*, 1898, 543.)

### Discussion.

Dr. S. **Rideal** congratulated the author and Dr. Goldschmidt on the success of the brilliant experiments they had seen, and asked if he was right in understanding that metallic calcium was reduced by the action of aluminium on calcium sulphate. If so, he would like to know what steps were taken for ensuring the permanency of the product, as he would have expected that, if the reduction were performed in air at the temperatures that were produced, it would have rapidly re-oxidised to the condition of lime.

Dr. **Goldschmidt** explained that he was not able to obtain pure metallic calcium. He had obtained an alloy of iron and calcium, and was so covered up during the reaction that there was a loss of only about 1 per cent.

Mr. J. B. C. **Kershaw** regarded the process as one which might have important industrial results. He need hardly remind the authors, however, that there were other methods in use for the production of metallic chromium, and that therefore the success or non-success of this particular process would depend entirely on the economical aspect of the question. In their paper they had not given any particulars as to the cost of the process generally, or of obtaining the aluminium in the powdered form; and it would be of great interest to have definite information from them upon this point of cost.

Dr. **Goldschmidt**, replying to Mr. Kershaw, said that the process was cheaper than any other now known. The cost of the chromium and ferrochromium was about 2 marks per kilo., say, a shilling a pound. The chromium in commercial ferrochromium contained about 10 per cent. of carbon. It had been mentioned that it was possible to reduce the slag back to aluminium, even if it were not sold for the purposes to which emery was now applied. Therefore, that would considerably reduce the cost chargeable to the production of the chromium.

Mr. B. **Kitto** enquired whether the author had succeded in reducing the oxide of uranium. He believed that Moissan had succeeded in getting a carbide of the metal fairly pure; still that was not uranium. Messrs. Vautin and Pickard had, about three years ago, tried some oxides of uranium with which he had supplied them, and succeeded in getting a strong uranium alloy of about 66 per cent., the rest being aluminium. Since that he had heard that metallic uranium had been obtained by the process, and would like to know if that was correct and if it had been proved by analysis to be uranium, and not an alloy. He had himself tried the ordinary text-book process of reduction from chloride, but without success. He would also like to ask which of the oxides of uranium Dr. Goldschmidt had operated upon.

Dr. O. J. **Steinhart** desired to know whether the presence of carbon in chromium acted deleteriously in the manufacture of chrome-iron or chrome-steel; for, if such was not the case, the ordinary method of working would be cheaper than that brought forward by Mr. Vautin. It was to be remembered that they had to deal with chrome-iron ore containing 40 per cent. of chromium, and costing 5*l*. per ton, whereas chromic oxide alone cost more than that, and, in addition, they had the expense of the preparation of the alumina, &c.

Dr. **Goldschmidt** said that the cost of the chromium in the chrome iron-ore was very small—only about a penny, whereas the cost of reduction by the ordinary method was very great. Moreover, in making the chromium for this purpose he avoided all cost for crucibles. That was a great saving, which enabled him to compete successfully with other processes. They had no difficulty in disposing of the pure chromium as fast as it could be made. In fact, many works were already abandoning the use of nickel in favour of chromium.

Mr. W. F. **Reid** asked if the author had tried any experiments with regard to the reduction of tungsten. He presumed not, as he did not see that metal among the specimens. With regard to the very pure chromium exhibited, he would be glad to know if the author was at liberty to state how he obtained the oxide from which it was produced.

Dr. **Goldschmidt** replied that it was obtained from the chrome-iron ore and chromates, and the process was performed easily and cheaply.

Mr. **Reid**, continuing, said he would be glad to know roughly what the process consisted in.

Dr. **Goldschmidt**, replying, said it was no secret: the chromates were reduced with coal. It was not necessary to have a very highly crystallised chromate, and therefore the cost of the oxide, when made in large quantities, was very small.

Mr. W. **Crowder** asked if any experiments had been made with vanadium, and whether that metal also was obtained in the pure state.

Dr. **Goldschmidt** said that it was obtained nearly pure. It was contaminated only with a little iron, and the operation was performed as with chromium and without much difficulty. Further replying to Mr. Reid's question, he would add that they had succeeded fairly well with the reduction of tungsten, but had not got it quite pure. The work, however, was easier than with uranium. He had made alloys of tungsten with both copper and iron, but to obtain the pure metal was very difficult, and that was a matter upon which he was still experimenting.

Mr. Bertram Blount, referring to a specimen of iron exhibited, said that that was probably the greatest curiosity in the room, as he understood that it was absolutely pure metal. If so, he would suggest that it would be exceedingly interesting to add the analysis of it to the paper. It would be very valuable as a basis for experiments in the production of alloys, starting with pure material.

Dr. Goldschmidt said that the specimen of iron was not quite pure. It was made from a pure oxide which was very hard to obtain. He could not at the moment remember the exact analysis, but he thought he might say that it contained no aluminium, only a little silicon from the aluminium used in the reduction.

Mr. E. Ackermann enquired if the author could state whether in his reaction he obtained a temperature equal in intensity to that of the electric furnace, and whether the reduction might not possibly be more economically effected by electric methods. The reduction of certain oxides by aluminium alone might be very useful where an electric furnace was not available or the quantity of metal required was relatively small. Calcium carbide, for instance, could be obtained on a small scale, both electrically and chemically. But on the large scale it could only be prepared by electrical methods, and the same with aluminium. Using an electrically obtained product on a large scale as a substitute for electricity did not appear to afford much economical advantage. Would it not be of advantage to use some other body for the purpose of the reduction, such as barium peroxide or iron filings, and to start the reaction by means of some other substance, such as liquid oxygen?

Mr. H. L. Sulman felt deep interest in the paper before the meeting, as he was one of those who was fortunate enough to witness some of the first experiments made with the method. By these means he had seen considerable ingots of pure metallic molybdenum, uranium, and vanadium produced for the first time in the world's history. On a later occasion, in view of the intense heat evolved, it was proposed to measure the temperature of the reaction, and Prof. Roberts Austen attempted this with his electric thermo-couple. The crucible was prepared as usual for a chromium reduction and heated, and in a few minutes the reaction occurred. The spot of light from the reflecting galvanometer, which up to this point had moved but slowly, then rapidly travelled round the scale, and upon removing the pile for inspection it was found that the heat had been so great that the immersed portion of the thermo-pile, with its platinum-rhodium terminals, had disappeared altogether into the fusion. He believed he was correct in saying that already, in Germany, large quantities of metallic chromium were being produced by this method for the manufacture of the chrome-steel for which Messrs. Krupp and Co. were famous. It was interesting to learn that the slag produced was applicable to the same uses as corundum. Altogether he regarded the process as one of the most elegant and complete reactions he was acquainted with.

Mr. Claude Vautin having been invited to reply, said that Dr. Goldschmidt had so ably replied to questions that had been put that he had but little to say. Referring to Mr. Ackerman's remarks, however, he might observe that the temperature of burning iron into oxide was several thousand calories less than that generated by the burning of an equal number of molecules of aluminium. In fact, results were obtained by the combustion of aluminium that were not possible in the electric furnace. Dr. Goldschmidt had made some experiments for determining the temperature reached, and estimated it up to 2,900° C. The process was already in practical operation, and the demand for chromium was so great that it was sufficient to use up the present production of aluminium for the purpose of reducing the chromic oxide. He understood the demand was attributable to the greater certainty of composition as compared with the use of ferrochromium and the freedom from carbon. He quite agreed with Mr. Blount that the specimen of iron shown was one of great chemical curiosity. He understood that although it contained a little silicon there was no carbon present.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Filter Presses, Impts. in.* J. Hill, Fenton, Stoke-on-Trent. Eng. Pat. 11,872, May 13, 1897.

The trays or sections consist of a parallel plate fitted with a wire mesh on each side, or with wire threaded to form a mesh, upon which the cloths are placed so as to allow the filtrate to escape.—E. G. C.

*Filtering Apparatus, New or Improved.* J. Mittwich. Bingen-on-the-Rhine. Eng. Pat. 8238, April 6, 1898.

A continuous filtering apparatus for wine, beer, syrup, and the like, which consists of a number of vertical filter chambers, containing filtering material between perforated plates, and provided with suitable inlet and outlet pipes. The chambers are connected by interchangeable pipes, so that any one of the series may be disconnected for cleaning and repairs. Each chamber is also provided with suitable air- and inspection taps.—E. G. C.

*Drying or other Treatment of Tea Leaf, Tobacco Leaf, Coffee, Grain, Malt, Fruits, and other Substances by Means of a Current of Hot or Dry Air, or Mixed Gas, or Gases and Air; Impts. in Apparatus for the.* S. C. Davidson, Belfast. Eng. Pat. 15,106, June 23, 1897.

The substance to be treated is caused to traverse a drying chamber upon an endless wire band, moved by any suitable agency, and is there exposed to currents of hot air drawn in from one end by a fan placed near the centre, and forced through the remaining length of the chamber by the same agency. Inclined perforated plates are interposed at suitable points, to cause the hot air to pass through the openwork band and the material carried upon it, while the perforations in these plates are so disposed as to equalise the drying effect throughout the chamber. Suitable means are provided for feeding the material upon the band at the inlet and removing it at the outlet.—E. G. C.

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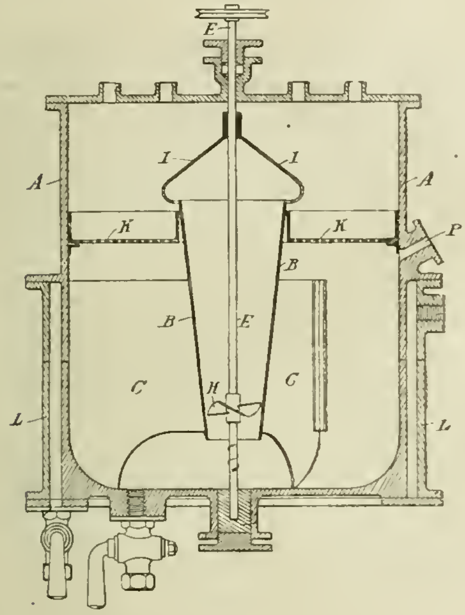
* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Air or other Gases or Liquids or Solids; applicable also for Mixing, Cooling, Heating, and other analogous Operations; Impts. in and Apparatus for Use in Subjecting to the Action of Air, Gases, or Liquids.* Forbes, Abbott, and Leonard, Ltd., and L. Gaster, London. Eng. Pat. 17,475, July 24, 1897.

A mixing, heating, or cooling apparatus for use in the manufacture of aniline, vinegar, dyes, and oils, and for other like purposes, is shown in sectional elevation in the Fig., and consists of a closed vessel A, having an open-ended cone B, secured therein by radial wings C. A screw propeller H, mounted upon a vertical shaft E, is rotated within the cone so as to cause an upward current of the fluid in the vessel, and above, there is a perforated revolving cap I, through which the fluid is forced in a series of jets, so that it may intimately mix with any solid matter placed upon a perforated tray K.



Gas, under pressure, may be admitted through an inlet P, and the mixture may be heated or cooled by a jacket L, while rotary motion of the fluid, due to the propeller, is arrested by the wings C.

In modified arrangements there are several cones placed one above the other in the chamber, and enveloping screw propellers keyed to the central shaft. In a further modification, the cones are placed side by side, and the blades of the propellers may be arranged to cause currents in opposite directions.—E. G. C.

*Continuous Extraction, Process and Apparatus for.* E. Schliemann and E. von Boyen, Hamburg, Germany. Eng. Pat. 6431, March 16, 1898.

An apparatus for treating such materials as cerasin (ceresin), petroleum residues, bones, and the like, with solvents such as naphtha, carbon bisulphide, alcohol, and ether, consisting of a number of tanks arranged column-wise for holding the material to be treated. The solvent is allowed to traverse the tanks in an order corresponding to the degree of exhaustion of the material contained therein, fresh solvent being always supplied to the tank containing the most exhausted material. The chambers are connected by piping controlled by valves, so that the path of the solvent may be adjusted, and are also connected by branches to a pipe supplying steam for removing the last trace of solvent and extract.—E. G. C.

## II.—FUEL, GAS, AND LIGHT.

*Petroleum Briquettes in Germany.* U.S. Cons. Reps., June 1898, 254.

Joseph Kohlendorfer, of Landshut, Bavaria, has invented a process for making petroleum briquettes, by which petroleum refuse is worked up to assume the form of a solid substance, easily handled, and constituting a cheap and convenient fuel.

About 10 per cent. of soda lye, with 10 per cent. of any other kind of fatty matter, is heated in a boiler, and enough petroleum refuse is added to make up 100 parts. This mixture is then heated, under constant stirring, for about an hour, the time varying with the mode and intensity of heating. Attention should be paid to prevent the temperature reaching the boiling point of petroleum, as in this case a large quantity of froth would form. In this incipient state of saponification, the mass acquires the property of taking up large quantities of fluid rock oil, and if this incorporation proceeds too slowly, or remains incomplete, it may be hastened by adding a little soda lye. The mixture is run into moulds and allowed to cool, when it may be cut into pieces of any desired form.

Coal dust, sawdust, or other refuse may be added during the process of manufacture, according to the use for which the briquettes are destined; and, if it should be desired to obtain a product of less consistency, the grease may be entirely or partially replaced by resin or resin acid, the product in either case having more than 80 per cent. of petroleum, more than 90 per cent. of combustible substances, and less than 5 per cent. of incombustible residue.

*Peat Bogs, Utilisation of, as Electrical-Power Stations.* Frank. Stahl und Eisen, 1897, **17**, 983; through Proc. Inst. Civil Eng. 1898.

It is stated that a square mile of bog, averaging 10 ft. in thickness, contains about 640,000 tons of dry peat, equivalent in heating power to 313,000 tons of coal. The author suggests the erection, in the neighbourhood of the great peat mosses of the North German plain, of an electric power station of 10,000 h.p., which would consume 200,000 tons of peat yearly, or the yield of about 200 acres. The electric energy could be utilised for power purposes or more particularly for the manufacture of acetylene, the necessary materials being both abundant and cheap in the district It is stated that with 10,000 h.p. a quantity of acetylene equivalent in illuminating value to 15,840 galls. of petroleum could be produced daily, representing an annual saving equal to the cost of about 20,000 tons of imported petroleum. —A. S.

*Combustible Vapours, Limits of Inflammability of.* Le Chatelier and Boudouard. Comptes Rend. **126**, [21], 1510—1513.

Volatile liquids (ether, benzene, &c.), were introduced in known amounts into a 2-litre flask of air, and allowed to evaporate, evaporation and admixture being aided by the use of an agitator in the flask; successive amounts (10 per cent. at a time) were added, till the mixture would inflame when tested with a light. In the case of less volatile liquids, the air of a 500-c.c. flask was saturated at varying temperatures, and the temperature gradually raised till inflammation took place on applying a light. The amount of vapour present was then determined by passing a known volume of air through the liquid (spread over pumice in a U tube) at this temperature, and determining the loss of weight of the liquid. In one or other of these ways the composition of that mixture was determined which contained the smallest proportion of the combustible vapour and was yet inflammable. A table of results is given, from which it appears that, with the exception of hydrogen and carbon bisulphide, the heat of combustion of a molecular volume of each of these mixtures of minimum inflammability, lies between 9 and 13 calories, and in most cases between 12 and 13 calories. The amount of oxygen needed for the combustion of these mixtures is also nearly constant, and approaches 11½ per cent. of the total volume.—J. T. D.

*Carbon Monoxide, Limits of Inflammability of.* Le Chatelier and Boudouard. Comptes Rend. **126**, [19], 1344—1347.

The carbon monoxide used was prepared from sodium formate and sulphuric acid, and contained 7 per cent. of air. The limits of inflammability, in tubes 250 mm. long and 40 mm. diameter, the open end being held in the air to a flame 1 cm. high, were 15·9 and 74·5 per cent. As the diameter of the tube was decreased, the inflammability became less, till at 2·3 mm. diameter, no mixture would inflame; the proportion of carbon monoxide and the limiting diameter of tube were connected as follows :—

| | | | | |
|---|---|---|---|---|
| Per cent. of CO ............... | 16·1 | 17·0 | 19·1 | 26·5 |
| Diameter of tube......... mm. | 13·1 | 7·0 | 5·7 | 3·0 |

| | | | | | |
|---|---|---|---|---|---|
| Per cent. of CO ............... | 38·0 | 47·5 | 57·0 | 61·7 | 71·3 |
| Diameter of tube ........ mm. | 3·0 | 2·3 | 3·0 | 5·7 | 11·0 |

Diminution of pressure also diminishes the inflammability of mixtures of carbon monoxide and air, no mixture being inflammable at a lower pressure than 80 mm. of mercury, whilst a mixture of coal-gas and air containing 15 per cent. of gas was still highly inflammable below 64 mm.

Rise of temperature increases the inflammability. When mixtures of gas and air were introduced into hot vessels, and a light applied after a few seconds, it was found that mixtures containing respectively 11·2, 9·3, and 7·4 per cent. of the monoxide could be inflamed at temperatures of 400°, 490°, and 650° C. respectively. Mixtures of carbon monoxide with acetylene and with hydrogen, when mixed with air, were found to obey very closely the authors' law for mixtures of methane and coal gas—

$$\frac{n}{N} + \frac{n'}{N'} = 1,$$

where $n$, $n'$, represent the percentages of the two gases in the mixture, and N, N', the limiting percentages for inflammability of each gas separately. In the case of mixtures of carbon monoxide and hydrogen, deviations from the law occur, perhaps due to the influence of the water formed, which the authors are investigating.—J. T. D.

*Carbon Monoxide in Blood: the Amount normally contained.* M. Nicloux. Comptes Rend. **126**, [21], 1526.

*See under* XXIV., *page* 701.

*Carbon Monoxide, Determination of Traces of, in Air, in the Presence of Hydrocarbons.* A. Gautier. Comptes Rend. **126**, [19], 1299.

*See under* XXIII., *page* 694.

*Phosphorescent Mixtures formed by Strontium Sulphide.* J. R. Mourelo. Comptes Rend. **126**, [21], 1508—1510.

Fifty grms. of barium carbonate, 50 grms. of strontium carbonate, and 30 grms. of sulphur, heated to cherry-red for two hours, gave a greenish mass, with a greenish-yellow phosphorescence of feeble intensity. A similar mixture, containing calcium carbonate instead of barium carbonate, gave a dirty white mass with a feeble violet green phosphorescence; while a mixture of the carbonates of calcium and barium with sulphur in the same proportions, gave a substance with a greenish phosphorescence. In all these cases, the addition to the mass before heating of sodium carbonate (2 grms.) and chloride (0·03 grm.) and a trace of bismuth subnitrate increases the intensity of the phosphorescence very considerably. On heating 100 grms. of barium sulphate with small quantities of the carbonates of barium and calcium, calcium and strontium, strontium and barium respectively, and a little sulphur in each case, to a cherry-red heat for two hours, mixtures are obtained containing two phosphorescent sulphides disseminated through the mass. Phosphorescence is not exhibited with less than 2½ per cent. of each of these substances, and increases in intensity as the proportions of the sulphides increase from that minimum. In each case, too, a more prolonged exposure is needed than when a mixture of two active substances is concerned. The intensity of phosphorescence is greatly increased by the addition of traces of sodium carbonate and chloride with bismuth subnitrate. By varying the proportions of the active substances relatively to one another, varying shades of colour can be obtained, from yellow to violet or green, the latter colour predominating usually, as the phosphorescence of strontium sulphide is more marked than that of the other two.

—J. T. D.

*Acetylene, Preliminary Note on the Action of Light on.* William A. Bone and John Wilson. Proc. Chem. Soc. 1898, [197], 155—156.

Some time ago the authors noticed that acetylene undergoes a well-marked change on exposure to bright sunlight. Tubes of about 100 c.c. capacity drawn out at each end and terminated by capillary glass taps were filled at atmospheric pressure with pure acetylene prepared from copper acetylide, and dried by passing through a concentrated solution of potassium hydroxide. Some of these tubes were exposed on the roof of the laboratory throughout June and July of last year, when the weather was particularly fine. After two or three days' exposure, a faint brown deposit was observable on the inside of the bulbs. This gradually increased in extent and thickness until, at the end of a fortnight, the tubes were entirely covered with a dark brown, greasy deposit. The change was evidently due to the action of sunlight, for if a portion of the tube were screened from the light, no deposit was formed over the area so protected, and, further, after the tube had become coated with the opaque deposit, no further action was noticeable even after prolonged exposure. Acetylene was decomposed to a less extent when exposed in tubes during August and September, 1897, than during the previous two months. The decomposition is independent of the presence of air, because acetylene mixed with its own volume of oxygen or nitrogen was exposed to sunlight for a very long period without any appreciable change occurring.

A slight contraction in volume was observed when tubes exposed during last summer were opened over mercury. A measured quantity of the gas sent into a Hempel pipette containing a freshly prepared ammoniacal solution of cuprous chloride was quickly reduced to about 2 per cent. of its original volume. The residual gas, after treatment with dilute sulphuric acid, appeared to contain a fairly dense hydrocarbon absorbed by fuming sulphuric acid, mixed possibly with a small quantity of hydrogen; no saturated hydrocarbon could be detected.

## PATENTS.

*Composition, Mixture, or Cement; An Improved, for Lining or Re-lining and Jointing, Mending, or Renewing Gas-Retorts, Furnaces, and the like.* A. W. Crowther, Huddersfield, Eng. Pat. 13,000, May 27, 1897.

*See under* IX., *page* 669.

*Gas for Lighting, Heating, and Motive-Power Purposes; Impts. in the Process of Obtaining, and in Apparatus therefor.* E. Soxhlet, F. van den Berghe, and Borremans frères, Hal, Belgium. Eng. Pat. 13,905 (under Internat. Convention), Jan. 22, 1897.

The air or other gas to be carburetted with acetylene or other carbide gas, is forced by a blower into a bell receiver, which, when it has reached a predetermined height, sets in motion, by means of a projecting finger, the chain gearing actuating an endless screw delivering the supply of carbide to the generator. The gas thus produced is conducted into the bell receiver and mixed with the contents already therein.

To enable the supply of gas to the burner to be regulated, and to prevent lighting back or back pressure, a tapering roll of metallic gauze with external helical ribs is inserted in the pipe, close to the burner.—C. S.

*Gas for Illuminating and other Purposes, Impts. in the Manufacture of.* H. H. Lake, London. From A. Piatti and Co., Rome. Eng. Pat. 14,505, June 15, 1897.

The apparatus is intended for the production of gas from straw and other vegetable substances, and for carburetting the said gas with acetylene (10 to 50 per cent.) or other hydrocarbons, &c.

It consists of a series of generators wherein the straw, &c., is carbonised by the aid of external heat, sufficient air being admitted to effect a flameless combustion resulting in the formation of "carbonic acid," methane, and acetylene. The gas is conducted to an accumulator, and thence through drying and purifying vessels to the gasometers, where it is mixed with acetylene delivered from a separate generator. —C. S.

*Carburetting Air, Impts. in Apparatus for.* E. Bouchaud-Praceiq, Angouleme, France. Eng. Pat. 8485, April 2, 1897.

The carburetting vessel is fitted with a perforated false bottom, and filled with blocks of porous material—preferably wood—saturated with the carburetting liquid, and arranged so as to leave interstitial spaces between each pair. The air is introduced at the top, and in its descent becomes charged with the carburetting vapour, the mixture then escaping through an uptake pipe, the inlet of which is at or near the false bottom of the vessel.

The carburetted air being heavy, the vessel is placed in an upper storey, and connected with the gas service pipes leading to the burners in the rooms underneath.—C. S.

*Coke Ovens, Impts. in, and in Machinery and Appliances in connection therewith.* W. H. Chambers, Conisborough, Yorkshire. Eng. Pat. 13,481, June 1, 1897.

In these ovens arrangements are made for equalising the applied heat, so that when any one oven is being drawn, or the gases are being delivered to the by-product recovery plant, the hot gases from the other flues are employed in maintaining the temperature, the result being that the coking of the fresh charge of coal begins immediately, thus expediting the operation and increasing the time capacity of the ovens.

Provision is also made for rapidly discharging the coke into trucks by means of a double ram actuated by a steam or other motor, and for the simultaneous raising and lowering of the end doors of each oven by a winding drum worked by the ram motor.—C. S.

*Hydrogen Gas, the Generation and Utilisation of, and Electricity for Motive Power, Lighting, and Heating Purposes; Impts. in.* L. H. Wattles, Providence, Rhode Island, U.S.A. Eng. Pat. 14,397, June 14, 1897.

It is proposed to utilise the hydrogen gas given off from electric batteries, zinc and sulphuric acid, or other "generators," in gas engines; the combination of gas generator and engine is claimed for lighting and power purposes, the mechanism being indicated by diagrammatic drawings. —J. C. R.

*Oil- or Spirit-vaporising Apparatus for Heating or Similar Purposes, Impts. in.* T. Watkins, Oxford. Eng. Pat. 14,535, June 16, 1897.

The generating chamber consists of an inverted bell of metal, in the walls of which are a series of zig-zag passages, through which the oil, &c. is forced, and becomes vaporised in its passage by the heat applied to the external surface. The gas thus formed, is delivered to a series of burners around the lower part of the bell, where it escapes into an annular mixing chamber admitting air through suitable perforations so as to produce a Bunsen flame, which envelops and heats the outer surface of the bell. The mouth of the latter being enlarged and overhanging, increases the size of the flame. The main pipe leading to the burners is fitted with a removable filtering appliance of gauze, &c., to free the gas from solid particles; and the burners are cleaned, and the flame of each regulated, by means of internal wires or pins, each of which is adjusted by a tap with a cranked spindle.—C. S.

*Waste Gases escaping from a Boiler Furnace or other Furnace, An Apparatus for Indicating the Proportion of Air contained in the.* C. Day, Bradford. Eng. Pat. 16,192. July 8, 1897.

A ball or float, of large capacity, is suspended from one arm of a steelyard, the other end of which is suitably weighted, so that when the float is surrounded by air, the index points to zero. Suitable openings are provided to allow furnace gases to flow through the casing, which latter closely surrounds the instrument to render its cubic capacity small in comparison with that of the float. The furnace gases buoy up the float and cause the index to move over a scale suitably graduated.—E. G. C.

*Glow Light, Electrical.* W. Nernst, Göttingen, Germany. Eng. Pat. 19,424, Aug. 23, 1897.

The improved glow lamp consists of a cylinder of some substance, such as magnesia, which is practically a non-conductor when cold, but becomes conductive when heated to a high temperature. The cylinder is provided with electrical connections, and means, such as a Bunsen burner, are used to raise it to the required temperature so as to make it conducting, when the current alone, or in conjunction with the gas flame, renders it incandescent.—G. H. R.

*Incandescent Electric Lamps [Carbides, &c.], and in Processes for Manufacturing them, Impts. in.* W. L. Voelker, London. Eng. Pat. 5863, March 10, 1898.

Carbides of the following metals are used for manufacturing filaments for use in incandescent electric lighting:—Uranium, thorium, ytterbium, erbium, cerium, calcium, and magnesium. "The oxides of the metals used should have a low specific heat, as this will determine the efficiency of the finished lamp." The claims are:—

I. "A cone, body, or filament for producing light by incandescing in a vacuum, such filament being formed of a metal or metals combined with carbon in the proportion of their chemically combining weights."

II. "A body, formed at a high temperature, consisting of one or more metals combined with carbon, and producing light by incandescence when subjected to the action of electricity in an exhausted glass chamber."—J. C. R.

*Furnaces, Refuse-consuming; Impts. in, and in Means for Utilising the Heat therefrom.* T. R. Berkley, London. Eng. Pat. 19,718, Aug. 26, 1897.

The inventor arranges the furnaces—preferably in pairs—on either side of the boiler, the products of combustion being led into combustion chambers, and thence into a central flue running longitudinally under the boiler. Suitable dampers for regulating the flow of the gases and for cutting off any of the furnaces at will are provided, and the furnaces are fitted with air-blast apparatus for forcing the draught. A subsidiary boiler (or boilers) with corresponding furnaces, &c., forms part of the general scheme, in order to enable steam to be got up quickly when the main furnaces are damped down.—C. S.

*Acetylene Gas, A New or Improved Apparatus for the Manufacture of.* A. Preston, Atherton, Lancs. Eng. Pat. 12,263, May 18, 1897.

The bell of a gasholder at a certain point in the course of its descent acts on a stem which operates a ball valve on a pipe leading to a chamber containing carbide. The valve, when opened owing to the descent of the bell, allows water to pass to the carbide, and the generated gas passes through a purifier, and thence through a pipe provided with a non-return valve to the gasholder.—J. A. B.

*Acetylene Gas, Impts. in and appertaining to Apparatus for the Manufacture of.* T. R. Fowler, Seacombe, County of Chester, formerly of Liverpool. Eng. Pat. 14,742, June 18, 1897.

The bell of a gasholder, when it falls to a certain point, actuates, by means of rods, a valve which allows water to pass from the tank to the annular space of one of two or more double-walled generators, in the inner chambers of which is carbide. The water passes through a U-shaped pipe from the annular space to the carbide chamber, and the acetylene generated, escapes through a coil to the gasholder.

Cocks are attached to the generators, and interlocked so as to prevent escape of gas when a lid is removed for recharging. Drip pipes for the escape of condensed water are provided.
—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Making.* J. and B. Aykroyd, both of Cullingworth, Yorks. Eng. Pat. 16,241, July 10, 1897.

A BELL gas-holder floats in an upper water vessel, and receives gas from a lower and closed water-vessel, in which water received from the upper vessel is maintained by means of an overflow at a constant level. Generators are fed with water from the upper vessel when the bell has fallen to such an extent that it depresses a lever which operates a valve on the water-supply pipe. The gas passes from the generators to the upper part of the lower water-vessel. Safety devices are described.—J. A. B.

*Gas-producing Materials, Impts. in.* J. Zimmerman, Chicago, U.S.A. Eng. Pat. 25,750, Nov. 5, 1897.

CALCIUM carbide or similiar material is crushed and mixed with a solution of shellac and alcohol or similar binding agent, and pressed into cakes or sticks. The presence of the binding agent is said to retard the action of water on the carbide.—J. A. B.

*Acetylene Gas, An Improved Purifier and Enricher for.* H. R. Bean, Plaistow, Essex, and H. Ringwood, Poplar, Middlesex. Eng. Pat. 14,700, June 17, 1897.

IN order to facilitate the perfect combustion of acetylene through ordinary gas burners, the acetylene is passed beneath the perforated bottom of a vessel containing a fibrous material saturated with methylated spirit or alcohol. It emerges at the top of the vessel saturated with the vapour of spirit, and cleansed of dust or foreign matter.—J. A. B.

*Acetylene Gas, Improved Apparatus for the Generation of, and the like Purposes.* J. W. Scarth, Pudsey, Yorks. Eng. Pat. 15,125, June 24, 1897.

A GASHOLDER bell, the displacement of which increases as it descends in the water cylinder in which it is placed, receives the gas from an adjacent generator which contains carbide, which is supplied with water from the gasholder whenever the level of water therein rises owing to the descent of the bell, above the top of a supply pipe. The gas passes from the bell down a condensing coil to the place of consumption.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Automatic Production of.* A. H. Barthez, Algiers. Eng. Pat. 25,870, Nov 6, 1897.

THE feed pipe for carbide mentioned in the specification of Eng. Pat. 9294 of 1897 (this Journal, 1898, 36) is extended below the surface of the water in the generator, and beneath its orifice is a distributing cone and a grid. By this arrangement the escape of acetylene to the distributing wheel is avoided.—J. A. B.

*Acetylene Gas, Novel Apparatus for the Production of.* V. Sockeel, A. Lanby, C. Drisse, and A. Sneck, all of Calais. Eng. Pat. 28,139, Dec. 2, 1897.

WITHIN a bell, and attached to its crown, is a carbide chamber with perforated bottom, by which it communicates with a lower sludge chamber. A pipe passes up into the carbide chamber and is there perforated, so that when the bell descends in its tank, water passes therefrom to the carbide, and causes evolution of gas and rise of the bell. The gas then passes into a gasholder.—J. A. B.

*Acetylene, Impts. in and relating to Apparatus for Generating and Purifying.* B. Richard, Lyons. Eng. Pat. 28,798, Dec. 6, 1897.

WATER from a vessel passes to the base of a chamber in which carbide cartridges are suspended, and the generated gas passes through a purifier and drier to a receiver. Safety devices are described.—J. A. B.

*Acetylene Gas Apparatus, Impts. in.* E. Edwards, Chancery Lane, Middlesex. From L. Steiner, Bucharest, Roumania. Eng. Pat. 7070, March 23, 1898.

ATTACHED to the crown of a bell is a carbide tank, in which the carbide is arranged at different levels, so that it is attacked successively by the water in the tank. A pipe rising above the bell serves as a safety-valve. The apparatus may be placed in the lower hollow part of a street lamp.—J. A. B.

*Calcium Carbide, Process for the Production of, in Blast Furnaces.* F. J. Bergmann, Neheim-on-the-Ruhr, Germany. Eng. Pat. 29,381, Dec. 11, 1897.

THE lime and coke are heated in a water-jacketed furnace enclosed between or encased in outer furnaces and communicating with the same,—in which coke or liquid hydrocarbon is burnt with oxygen. The high temperature thus obtained produces calcium carbide and dispenses with the electric furnace. The oxygen is blown in from a gasometer, which is kept supplied by two furnaces in which manganese dioxide is alternately heated to yield oxygen and exposed to air at a lower temperature to regenerate.
—A. W.

*Calcium Carbide, Improved Method and Apparatus for Producing.* [*Coke and Lime Blocks.*] W. P. Thompson, London. From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuehel, Holstein, Iowa, U.S.A. Eng. Pat. 7574, March 29, 1898.

THE furnace is loosely lined with granulated calcium carbide, and means are provided for striking an electric arc within the furnace, between electrodes which may be arranged in any position from the horizontal to the vertical, and may be either tubular or solid. The coke and lime, which have been previously pulverised and compressed into blocks or sticks, and connected to the metal hooks, are fed into the arc, either through the electrodes, or passed between them at right angles to the arc; and in either case there is no incrustation of imperfectly reduced lime or carbon on the finished product, on the stoppage of the current.—G. H. R.

*Calcium Carbide, Impts. in Electric Furnaces for Manufacturing.* [*Hand-Feeding.*] W. P. Thompson, London. From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuehel, Holstein, Iowa, U.S.A. Eng. Pat. 7575, March 29, 1898.

THE furnace consists of a base, an electrode mounted on it, a metal casing divided into two parts, a latch at the opposite side thereof from the hinges, a hook on each part, and slides hinged to the top of the casing to cover openings therein. The upper electrode, which is movable, is suspended within the casing, and has one or more vertical openings extending through it, and is surrounded at its upper end by a band connected by a cable to the source of electric supply. Above the band is a metal collar connecting the electrode with an extension of non-conducting material and provided with corresponding vertical openings. By means of ropes attached to hooks at opposite sides of the collar, the casing and electrode are connected to one another and to a crane, so that when the electrode has been raised to its full extent, the casing is lifted, allowing the charge to be withdrawn. The block of insulating material, with openings in it, allows of sticks of compressed carbon and lime being fed into the arc by hand at the proper speed to produce the best results.—G. H. R.

*Vaporising Burners, Impts. in.* H. H. Lake, London. From A. af Forselles, Helsingfors, Finland. Eng. Pat. 6764, March 19, 1898.

THE apparatus consists of a feed tube closed at its upper extremity and projecting about half an inch above the surrounding burner tube, which is covered by a gauze top and turned up at the lower end to form an inlet, narrower than the rest of the tube. The oil vaporised by heat in the feed-tube is discharged—through a capillary aperture in the lower end of a small bent tube issuing from the upper end of the feed-tube—in the form of a conical jet into the mouth of the U-shaped burner tube, and thereby carries along into the latter sufficient air to ensure complete combustion.

Back pressure in the feed-tube is prevented by the provision of metallic gauze, baffles, or other suitable material attached to a rod and inserted from below.—C. S.

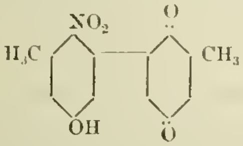
## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Cedriret* [*from Wood-Tar*]. R. Nietzki and R. Bernard. Ber. **31**, 1334—1338.

Hofmann in 1878 showed that a quinone-like substance, termed coerulignone by Liebermann, was in fact a tetramethoxydiphenylquinone, the formation of which was due to the oxidation of pyrogallol dimethyl ether. He also identified it with a substance obtained 40 years earlier by Reichenbach, who had named it "cedriret." Nietzki, in the same year as Hofmann, obtained a similar substance by oxidising hydrotoluquinone dimethyl ether, to which he gave the formula—

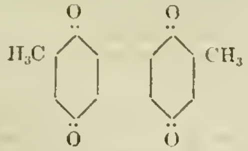
$$\begin{array}{c} CH_3O.C_7H_5.O \\ \cdot \qquad \cdot \\ CH_3O.C_7H_5.O, \end{array}$$

and terms ditolylcedriret and with a view of obtaining some light on the formula of cedriret, the authors have further examined Nietzki's product. On treatment with hydroxylamine hydrochloride in alcoholic solution, an oxime, $C_{16}H_{17}NO_4$, is obtained, which gives an acetyl derivative, $C_{18}H_{19}NO_5$. The oxime, when warmed with six times its weight of nitric acid (sp. gr. 1·3), dissolves, evolving red fumes, and, on cooling, a compound separates out which crystallises from alcohol in red needles, is simultaneously a nitro body and a quinone, and has the formula—

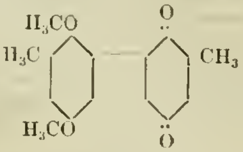


On heating with acetic anhydride it yields an acetyl derivative which crystallises in light yellow needles melting at 143° C.

On oxidising hydrotoluquinone, K. Brunner obtained two substances, one of which he termed ditolyldiquinone—



yellow crystals melting at 163° C.; the other, having much similarity with ditolylcedriret, is a quinhydrone compound of the above with its hydro derivative. By boiling ditolylcedriret with dilute nitric acid it is converted into Brunner's ditolyldiquinone, showing all the reactions of this substance, and when reduced with ammonium sulphide, it forms tetrahydroxyditolyl (m.p. 203° C.). This throws a new light upon the oxidation products of the hydrotoluquinone ether, and it would appear from this that the cedrirets have an unsymmetrical constitution, the cedriret from dimethylhydrotoluquinone having the formula—



This on further oxidation splits off two methyl groups, forming Brunner's ditolyldiquinone. This view does not altogether agree with the formation of the cedrirets of Liebermann and Hofmann.—T. A. L.

### PATENT.

*Continuous Extraction, Process and Apparatus for.* E. Schliemann and E. von Boyen, Hamburg, Germany. Eng. Pat. 6431, March 16, 1898.

*See under I., page* 651.

## IV.—COLOURING MATTERS AND DYES.

*Safranine, Constitution of.* G. F. Jaubert. Comptes Rend. **126**, [21], 1515—1518.

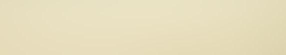
By heating together for two hours with a reflux condenser, 5 grms. each of picryl chloride, *m*-phenylenediamine hydrochloride, and sodium acetate with 50 c.c. of alcohol, the author obtains red crystals of trinitro-*m*-amidodiphenylamine, which, when oxidised with *p*-phenylenediamine, gives Trinitrophenosafranine, a substance with considerable tinctorial power, which dyes cotton mordanted with tannin or tartar emetic, ruby-red. As the trinitro-*m*-amidodiphenylamine is substituted in the two ortho-positions with regard to the nitrogen, it is impossible to formulate the trinitrophenosafranine (this Journal, 1896, 608) by the unsymmetrical formula, and the formation of this substance therefore gives additional confirmation to the symmetrical formula for the Safranines (see also this Journal, 1895, 564 and 858).—J. T. D.

*Indigo Oxime.* J. Thiele and R. H. Pickard. Ber. 1898, **31**, [8], 1252—1253.

Indigo oxime is prepared by warming very finely divided indigo (prepared by oxidation from the zinc-dust vat or synthetically from *o*-nitrobenzaldehyde) with caustic soda on the water-bath, expelling dissolved air by hydrogen, and dropping in with continual shaking, twice as much hydroxylamine hydrochloride. The greater part of the indigo dissolves; after warming for half an hour the liquid is filtered directly into cold dilute hydrochloric acid, when the oxime separates as a voluminous brownish-violet precipitate; this is washed with boiling water, in which it is very slightly soluble. The oxime crystallises from alcohol in brownish-violet needles of coppery lustre, which melt at 205° with decomposition. Indigo oxime is somewhat soluble in alcohol, ether, and acetone; insoluble in benzene and petroleum ether. Hot hydrochloric acid separates no indigo; dilute soda readily dissolves the oxime to a wine-red solution, which rapidly oxidises in the air. In an alkaline solution, zinc dust produces a blue precipitate which soon dissolves. In the air the solution so obtained, gives a blue amorphous precipitate, soluble in alcohol and acetone, and in small quantity in dilute acids, to a blue solution; its solution in strong sulphuric acid is green, and becomes blue on warming. Alkaline hydroxylamine readily reconverts the body into indigo oxime. Probably this substance is indigo imide; it readily evolves ammonia, and could not be obtained pure. No indigo dioxime could be prepared.
—A. C. W.

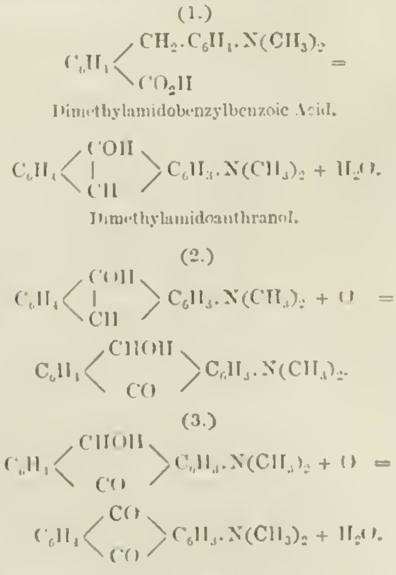
*3-Dialkyl-amido-anthraquinones, Preparation and Properties of.* A. Haller and A. Guyot. Comptes Rend. **126**, 1544—1546.

In their previous communication (Comptes Rend. **126**, 1249) the authors showed that substituted o-benzoylbenzoic acids, when reduced, are first converted into substituted monophenylphthalides and then into benzylbenzoic compounds. They have now condensed these same acids into anthraquinone compounds, using the method employed in analogous condensations of the non-substituted benzylbenzoic acid. In order to condense dimethylamidobenzoylbenzoic acid, it is heated with 10 times its weight of sulphuric acid to 175°—180° C., sulphurous acid being liberated. On pouring the melt into water, a red flocculent precipitate is obtained, which may be crystallised from benzene in red needles melting at 181° C., and having the formula—



$$C_6H_4\left\langle\begin{array}{c} CO \\ CO \end{array}\right\rangle C_6H_3.N(CH_3)_2$$

This substance, dimethylamido-anthraquinone, is obtained to the amount of 35—40 per cent. of the theoretical quantity, whilst at the same time, ill-defined oxidation products are formed, which are being investigated. In order to increase their yield, the authors have modified the above process, taking, not dimethylamidobenzoylbenzoic acid, but its reduction product, dimethylamidobenzylbenzoic acid. When this acid is heated with 12 times its weight of sulphuric acid at 80° C. for half an hour, a reddish-brown solution is obtained, which becomes a clear yellow on dilution with water. Neutralisation with sodium carbonate gives a precipitate which, when crystallised from benzene, yields diamidoanthranol in the form of yellow needles, easily oxidised by the air even in benzene solution, and more easily in alkaline solution, with the production of dimethylamidoanthraquinone. For the preparation of this substituted anthraquinone, the sulphate of dimethylamidoanthranol obtained as above, is treated with a solution of ferric chloride (sp. gr. 1·30), when the liquid turns violet, probably due to the transient formation of dimethylamido-oxanthranol, afterwards it becomes nearly colourless and deposits a voluminous red magma of matted needle-shaped crystals. On boiling the liquid for an hour, these become denser, and may then be collected and dried. They consist of pure dimethylamidoanthraquinone, the yield being 80—85 per cent. of the theoretical. The series of changes may be expressed thus:—



(1.)

$$C_6H_4\left\langle\begin{array}{l}CH_2.C_6H_4.N(CH_3)_2\\ CO_2H\end{array}\right. =$$

Dimethylamidobenzylbenzoic Acid.

$$C_6H_4\left\langle\begin{array}{c}COH\\ |\\ CH\end{array}\right\rangle C_6H_3.N(CH_3)_2 + H_2O.$$

Dimethylamidoanthranol.

(2.)

$$C_6H_4\left\langle\begin{array}{c}COH\\ |\\ CH\end{array}\right\rangle C_6H_3.N(CH_3)_2 + O =$$

$$C_6H_4\left\langle\begin{array}{c}CHOH\\ \\ CO\end{array}\right\rangle C_6H_3.N(CH_3)_2.$$

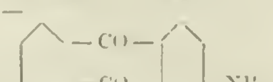
(3.)

$$C_6H_4\left\langle\begin{array}{c}CHOH\\ \\ CO\end{array}\right\rangle C_6H_3.N(CH_3)_2 + O =$$

$$C_6H_4\left\langle\begin{array}{c}CO\\ \\ CO\end{array}\right\rangle C_6H_3.N(CH_3)_2 + H_2O.$$

Dimethylamidoanthraquinone.

The homologue, diethylamidobenzoylbenzoic acid behaves in a similar manner. The diethylamidoanthraquinone obtained by one of the above methods crystallises from boiling toluene in red leaflets melting at 162° C. The constitution of these new anthraquinone derivatives must be as follows:—

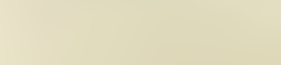


A number of nitro-, amido-, sulpho-, and hydroxy-compounds may easily be obtained from them, which are under investigation, some of them being capable of technical application.—T. A. L.

### *Ethenyltriamidonaphthalene and its Acetyl Derivative.*

O. Markfeldt. Ber. **31**, [8], 1174—1178.

Meldola and Streatfeild (J. Chem. Soc. 1887, 691) described ethenyltriamidonaphthalene—



$$H_2N.C_{10}H_5\left\langle\begin{array}{c}NH\\ \\ N\end{array}\right\rangle C.CH_3$$

but were not able to prepare its acetyl derivative. The author proceeds as follows:—100 grms. of dinitro-acetnaphthalide in 600 c.c. of alcohol are mixed with 200 grms. of iron filings, heated on the water-bath under an inverted condenser, and 500 grms. of hydrochloric acid (sp. gr. 1·19) are allowed to drop in slowly, the whole being then boiled for 1—1½ hours. About 300—400 c.c. of water are added, and, on cooling, the hydrochloride crystallises out in needles, and is purified by dissolving in water and precipitating by the addition of an equal volume of hydrochloric acid. The primary azo colours obtained from the diazo compound of this substance with the usual components have a distinct affinity for vegetable fibres, but on account of their instability to light, washing, and acids, have little technical value. On the other hand, the secondary disazo dyestuffs obtained by employing a further diazotisable intermediate amido compound are valuable products comparable with the diamine colours. Those dyestuffs which contain no sulphonic acid group possess further the valuable property of dyeing unmordanted cotton from an acid bath, and this property is also shared by those derivatives which contain a carboxyl group. Thus the dyestuff from ethenyltriamidonaphthalene + phenyloxamic acid + *m*-phenylene diamine, dyes cotton from a bath containing acetic acid. The presence of a nitro group does not affect this property. The preparation of the acetyl derivative of ethenyltriamidonaphthalene was effected by boiling 100 grms. of the hydrochloride with 80 grms. of fused sodium acetate and 400 grms. of acetic anhydride. The acetate of the acetyl derivative separates out on cooling, and can be recrystallised from acetic acid. It melts above 280° C. The free acetyl derivative forms silky white needles insoluble in ether, benzene, petroleum ether, and chloroform, very easily soluble in alcohol and sparingly soluble in hot water. It is much more stable than the free base, which, on account of its solubility and extreme oxidisability, has not been obtained in a free state. Various salts of the acetyl derivative have been prepared.—T. A. L.

### *Fluorescence Phenomena in the Coumarin Series.*

H. Kunz-Krause. Ber. **31**, [8], 1189.

*See under* XXIV., *page* 701.

### *Indian Dyestuff "Waras," Constituents of the.*

A. G. Perkin. Proc. Chem. Soc. 1897, [197], 162.

Waras is a purplish powder which covers the seed pods of *Flemingia congesta*, an erect, woody shrub of Africa and India. In its general properties and microscopic appearance it closely resembles Kamala (*Mallotus Phillipinensis*) previously investigated (this Journal, 1893, 672; Trans. Chem. Soc. 1894, **65**, 232).

*Flemingin*, $C_{12}H_{12}O_3$, the principal crystalline constituent, was obtained as an orange-red powder consisting of small prismatic needles melting at 171°—172°. In appearance and numerous properties, it resembles the Rottlerin of Kamala, but is distinguished from this by its solubility in alcohol and by the browner tint of its alkaline solutions. In an alkaline bath it dyes silk a golden yellow, and is a stronger dyestuff than Rottlerin.

*Homoflemingin*, present only in minute quantity, forms glistening yellow needles, melts at 164°—166°, and possesses properties resembling those of Flemingin.

The *resin of high melting point*, $C_{12}H_{12}O_3$, forms a brick-red powder, soluble in alkali, with a deep brown tint, and yields acetic and salicylic acids on fusion with alkali. It dyes silk in shades which are redder than those produced by Flemingin.

The *resin of low melting point*, $C_{13}H_{14}O_3$, is a deep orange-brown transparent mass which melts below 100°, is soluble in alkali with an orange-brown colour, and closely resembles the resin of low melting point of Kamala.

It is concluded that the above substances are closely related to, though not identical with, those present in kamala.

Waras dyes silk a golden yellow shade, and is a much stronger dyestuff than Kamala.

## PATENTS.

*Colouring or Carbinol Matters [Malachite Green], Impts. in the Manufacture of.* M. Otto, Neuilly, Paris, France. Eng. Pat. 14,917, June 19, 1897.

The patentee makes use of ozone as an oxidising agent for converting leuco bases into the corresponding dyestuffs. For instance, by passing the theoretical quantity of ozonised air at 100° C. into a solution of tetramethyldiamidotriphenylmethane hydrochloride, the solution turns a cantharides green colour, and the dyestuff may then be separated in the usual manner by precipitation with salt or zinc chloride. In the case of Malachite Green, in place of the leuco base, a mixture may be employed consisting of 50 kilos. of dimethylaniline, 22 kilos. of benzaldehyde, and 13·4 kilos. of sulphuric acid (66° B.), the product formed being separated as above.—T. A. L.

*Black Colouring Matters of the Anthraquinone Series, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Anilin- u. Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 14,918, June 19, 1897.

This is an extension of Eng. Pat. 15,242 of 1895 (this Journal, 1896, 587) and refers to the action of sulphur and sodium sulphide on the following anthraquinone derivatives: Alizarin, anthrapurpurin, flavopurpurin, hexahydroxyanthraquinone, anthraquinone sulphonic acid, anthraquinone β-disulphonic acid, anthraquinone α-disulphonic acid, β-nitro-alizarin, and β-dibromo-anthraquinone. Anthraquinone itself does not give any useful result. About 40 kilos. of sodium sulphide (containing 65 per cent. of $Na_2S$), 15 kilos. of flowers of sulphur, and 10 kilos. of one of the above substances are heated together in an iron vessel provided with an agitator until successive tests show no change. The melt is then cooled and powdered, and can be used directly for dyeing. The dyestuffs obtained are soluble in water, and are precipitated by dilute acids. They are practically insoluble in alcohol and acetone, and dye cotton black from a cold bath.—T. A. L.

*Yellow Colouring Matters from Benzoin, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Fabrik, Ludwigshafen, Germany. Eng. Pat. 14,919, June 19, 1897.

Dyestuffs for chrome-mordanted wool giving yellow shades fast to fulling are obtained, according to this invention, by condensing benzoin with aromatic hydroxycarboxylic acids, such as gallic acid or β-dihydroxybenzoic acid, in sulphuric acid solution. A solution of 100 kilos. of gallic acid in 1,600 kilos. of sulphuric acid (66° B.) is cooled to 0°—5° C., and 126 kilos. of benzoin are stirred in, the stirring being continued for 24 hours. The melt is then poured into 8,000 litres of water, and the precipitated dyestuff is filtered off and washed. It forms a yellow paste similar to alizarin, is insoluble in water, but dissolves in sodium carbonate to a yellowish-brown solution, and in caustic soda to a cherry-red. The solution in concentrated sulphuric acid is yellow, with a green fluorescence. It dissolves with difficulty in benzene and petroleum ether, but is readily soluble in alcohol, glacial acetic acid, and nitrobenzene. The acetyl compound, which crystallises in yellow needles, melts at 237° C. The condensation product from dihydroxybenzoic acid and benzoin gives greener shades, and dissolves in alkalis to yellowish-red solutions, whilst the sulphuric acid solution is a Rhodamine Red shade and has a brownish fluorescence.—T. A. L.

*Green Mordant-dyeing Colouring Matters from Halogen-substituted Fluoresceïnes, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Fabrik, Ludwigshafen, Germany. Eng. Pat. 14,921, June 19, 1897.

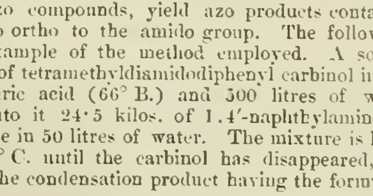
When eosine is heated with sulphuric acid, traces of bromcœrulein are obtained. If, however, boric acid be added to the melt, a large yield of a useful dyestuff is produced, which gives fast green shades on chrome-mordanted wool. A similar dyestuff is obtained if dibromofluoresceïn be employed instead of eosine (tetrabromofluoresceïn). For example, about 10 kilos. of dibromofluoresceïn and 10 kilos. of boric acid are stirred into 100 kilos. of sulphuric acid (66° B.), and heated with agitation at 240°—250° C. until the dibromofluoresceïn has disappeared. The melt is then cooled, poured into water, the dyestuff filtered off, and made into a paste. It dissolves in sodium carbonate or caustic soda to a bluish-green solution, the colour in sulphuric acid being brownish-red. The dyestuff is insoluble in water, and dissolves with difficulty in alcohol, forming a violet solution. It dyes green shades on chrome-mordanted wool.—T. A. L.

*Diazo Salts in a Durable Form, Producing.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 15,353, June 26, 1897.

Paranitraniline is diazotised with nitrous acid in sulphuric acid solution, and a quantity of a normal alkali sulphate is added equivalent to the sulphuric acid, so that a dry mixture of the sulphate of the diazo compound and an alkaline bisulphate results. Nitrous acid is passed into 80 kilos. of sulphuric acid until 10 kilos. have been absorbed, when 30 kilos. of *p*-nitraniline are stirred in, in the cold. To this solution 120 kilos. of calcined sodium sulphate are added, and after a short time the paste becomes solid, when it may be broken into lumps or powdered. It form a perfectly clear solution in water, and is not affected by percussion or heat.—T. A. L.

*Dyestuffs [Blue], The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 15,478, June 29, 1897.

This is an extension of Eng. Pat. 3452 of 1897 (this Journal, 1898, 42), and consists in applying the process there described to sulphonic acids of the diphenylnaphthylmethane series, containing an amido group ortho to the methane carbon. These products are obtained by condensing alkylated diamidodiphenyl carbinols with those α-naphthylamine sulphonic acids which, when combined with diazo compounds, yield azo products containing the azo group ortho to the amido group. The following is a typical example of the method employed. A solution of 27 kilos. of tetramethyldiamidodiphenyl carbinol in 20 kilos. of sulphuric acid (66° B.) and 500 litres of water has stirred into it 24·5 kilos. of 1.4′-naphthylamine sodium sulphonate in 50 litres of water. The mixture is heated to about 80° C. until the carbinol has disappeared, and, on cooling, the condensation product having the formula—


$$[(H_3C)_2N.C_6H_4]_2:\overset{H}{C}—$$

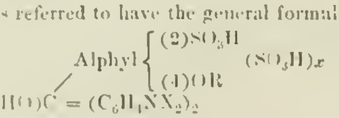
crystallises out. A quantity of this substance corresponding to 7 kilos. of sodium nitrite is dissolved in 400 litres of water and 100 kilos. of sulphuric acid, and diazotised in the cold by means of an aqueous solution of 7 kilos. of sodium nitrite. The diazo solution has then passed into it 20 kilos. of sulphur dioxide, and subsequently 15 kilos. of copper powder are added, care being taken that the temperature does not rise above 0° C. When nitrogen is no longer evolved, 200 kilos. of finely powdered salt are added; the sulphinic sulphonic acid is filtered off and washed with brine. It is separated from the copper by extraction with sodium carbonate, and the free sulphinic sulphonic acid is precipitated from this solution by adding a mineral acid. 52·4 kilos. of this acid are dissolved in 500 litres of water containing 8 kilos. of sodium hydroxide, and a sodium hypochlorite solution containing 7·1 kilos. of available chlorine is allowed to flow in slowly, when the sulphinic group is converted into a sulphonic group. After neutralising, the solution is mixed with 100 kilos. of 50 per cent. acetic acid, 10 kilos. of sulphuric acid (66° B.), and 160 kilos. of a lead peroxide paste containing 15 per cent. of $PbO_2$, the temperature being kept at about 30° C. The lead sulphate is then filtered off and the dyestuff is salted out. It forms brownish crystals, dyeing clear greenish-blue level shades on wool from an acid bath, fast to alkalis. Other naphthylamine sulphonic acids suitable for this process are the 1.4-naphthylamine sulphonic acid, the 1.4′.2′ and the 1.4.2′-naphthylamine disulphonic acids.—T. A. L.

*Ortho- and Para-nitrobenzylaniline Sulphonic Acids and their Homologues, Manufacture of.* O. Imray, London. From The Farbwerke Hoechst, Hoechst a M., Germany. Eng. Pat. 15,890, July 3, 1897.

According to Eng. Pat. 22,041 of 1896 (this Journal, 1897, 795), the crude product obtained by chlorinating *o*- or *p*-nitrotoluene is treated with aniline or one of its homologues, whereby a separation into nitrobenzylaniline, nitrotoluene, and aniline hydrochloride is effected. The same result can be arrived at more simply, according to the present specification, by employing an aniline sulphonic acid or a homologue thereof in place of aniline. The following example illustrates the method employed:—34·2 kilos. of the crude product of chlorination of *o*- or *p*-nitrotoluene (corresponding to 17·1 kilos. of nitrobenzyl chloride), together with 23·3 kilos. of sodium sulphanilate (83·5 per cent.), and 6 kilos. of calcined soda in 100 litres of water are boiled for several hours in a vessel provided with an agitator under an inverted condenser. On cooling, the aqueous solution containing the nitrobenzyl-sulphanilic acid is separated from the nitrotoluene, which is used for another chlorination. The free nitrobenzylsulphanilic acid is precipitated by adding a dilute mineral acid to the aqueous solution, and is filter-pressed and dried. Other acids suitable for the purpose are metanilic acid: aniline *o*-sulphonic acid; 1.2.4-, 1.2.5-, 1.4.2-, and 1.4.3-toluidine sulphonic acid; 1.3.4.6-, 1.4.2.6-, and 1.4.2.5-xylidine sulphonic acid, or naphthionic acid.—T. A. L.

*Dyestuffs [Green, Blue] of the Triphenylmethane Series Fast to Alkali, Manufacture of.* O. Imray, London. From The Farbwerke Hoechst, Hoechst a M., Germany. Eng. Pat. 16,483, July 12, 1897.

The dyestuffs referred to have the general formula—



$$\mathrm{(HO)C}=(\mathrm{C_6H_4NX_2})_2,\ \mathrm{Alphyl}\begin{cases}(2)\mathrm{SO_3H}\\(4)\mathrm{OR}\end{cases}(\mathrm{SO_3H})x$$

and are obtained by condensing tetra-alkyl-diamido-diphenyl carbinols with *m*-alkyloxysulphonic acids, such as *m*-methoxybenzene sulphonic acid, 1.3-methoxynaphthalene sulphonic acid, 1.3.3′- and 1.3.1′-methoxynaphthalene disulphonic acid, and oxidising the leuco compounds thus formed. If the leuco compound only contains one sulphonic acid group a further sulphonation is necessary, which may precede or succeed the oxidation. The sulphonic acid group enters the benzene or naphthalene nucleus, or the hydrol, if this contains benzyl radicles which are easily sulphonated. 7·5 kilos. of tetramethyl-*p*-diamidodiphenyl carbinol, 10 kilos. of 1.3.3′-methoxynaphthalene sodium disulphonate, and 60 kilos. of concentrated sulphuric acid are digested in the cold for a few hours. The melt is then poured on to ice, limed, and the solution of the calcium salt is evaporated. This salt is oxidised by dissolving it in 30 times its weight of water and adding the calculated quantity of dilute sulphuric acid and lead peroxide. After filtering, the solution of the dyestuff is evaporated and forms a brilliant copper-coloured mass soluble in water to a greenish-blue solution and dyeing wool the same shade from an acid bath fast to alkalis.—T. A. L.

*Colouring Matters [Violet] having Acid Action, Manufacture of.* M. Prud'homme, Paris, France. Eng. Pat. 16,713, July 14, 1897.

Basic dyestuffs, such as Magenta, Safranine, Amido-indulines, Thionine, or Lauth's Violet, Eurhodines, Phosphines, Acridines, Hofmann Violets, Greens derived from *m*-amidobenzaldehyde, phthaleïnes containing a primary or secondary amido group, or, in general, dyestuffs containing primary or secondary amido groups, when treated with formaldehyde and sodium bisulphite, are converted into dyestuffs having acid functions. For example, 1 kilo of safranine hydrochloride dissolved in 30 litres of water is mixed with 15 kilos. of 10 per cent. sulphuric acid, 3 kilos. of sodium sulphite (30° B.), and 1 kilo. of a 40 per cent. solution of formaldehyde, the whole being heated on the water-bath for two hours, after which the dyestuff is salted out. It dyes a more violet shade than the original, and possesses all the characteristics of a sulphonic acid dyestuff, is not precipitated by alkalis, has no affinity for tannined cotton, but dyes wool from an acid bath. The dyestuff obtained from magenta is very easily soluble in water, but may be completely precipitated by adding a primary amine, forming a very blue lake, which can be easily decomposed by concentrated alkaline lyes, which liberate the amine, regenerating the dyestuff.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Net Lace, The Finishing of.* E. Ackermann. Textile Colorist, 1898, **20**, 133—134.

In finishing net lace, what is wanted is to give firmness to the thread and rigidity to the tissues.

To obtain these qualities, the lace is stretched on frames, and, by means of a brush, worked manually or by machinery, coated with various compositions, and brushed till dry: the workshop is kept at a temperature of about 40° C. Several compositions are given, and the following may be taken as an example:—

For black silk lace, 5 kilos. of gum tragacanth are steeped in 100 litres of water for twelve hours.

A warm mixture of 2 kilos. of wax, 1 kilo. of spermaceti, and 10 lb. of ammonia, is then made. The two are mixed together and 5 kilos. of sugar are added. The whole is then made up to 500 litres. After passing through the gum solution, the lace may, if desired, be treated with a solution of paraffin in benzene.

Vegetable fibres resist the action of either hot or cold alkaline solutions, but will not stand the action of mineral acids without alteration.

Animal fibres contain a large proportion of sulphur, and should not, on this account, be treated with metallic salts, such as lead acetate. They will not stand the action of strong acids or alkalis.—C. M.

*Wool Scouring, Loss from.* E. F. Ladd, Gov. Agric. Exp. Stat., N. Dakota. Bull. **32**, 271.

The loss on scouring three samples of wool amounted to 60·01, 62·64, and 61·77 per cent. respectively. These were fair samples from the sheep bred upon the prairies in the sheep belt of the western part of the States. Data which have come under the observation of the author show that the usual loss of North Dakota wool is from 55 to 70 per cent.—W. P. S.

*Silk Lustre on Cotton, Production of.* A. Buntrock. Zeits. f. angew. Chem. 1898, 479—482.

Considerable interest has been aroused within the last few years in the textile industry by the publication of Thomas and Prevost's method for the production of a silk lustre on cotton by mercerising (*i.e.*, treatment with soda lye) under tension. The patent has been refused in England, and the paper contains a historical survey of the methods previously patented and bearing on the subject. In Oct. 1850, J. Mercer patented a method for increasing the strength, tenacity, and affinity for dyestuffs of cotton, by immersing it, after damping, in soda or potash lye of 34°—38° B. at 16° C. The cotton is then wrung out, hydro-extracted, washed, passed through dilute sulphuric acid, and again washed. Mercer states that cotton materials so treated decrease in length and breadth, becoming thicker and denser, the effect being similar to that obtained by milling wool. Sulphuric acid of 50°—51° B. at 16° C., or a zinc chloride solution (61° B.), may be employed in place of the soda lye. According to Mercer, the lower the temperature, the greater the action of the soda lye on the cotton. In Dec. 1887, Lightoller and Laughan patented a process (Eng. Pat. 5713 of 1887) for increasing the tenacity of cotton fibres by treatment with sulphuric acid or zinc chloride solution in a suitable apparatus. Two years later (Eng. Pat. 20,314 of 1889) H. A. Lowe took out a patent for increasing the lustre, toughness, and colour-affinity of the cotton fibre by treatment with soda or potash lye, with or without the addition of zinc oxide. In a later specification (Eng. Pat. 1452 of

1890) this process was modified by preventing the fibre from shrinking either during immersion in the soda lye and until it has passed the washing process, or else stretching the material before finally drying it. Thomas and Prevost, obtained a German patent in 1896 (*viz.* No. 85,564) on their application in 1895. They point out that vegetable fibres are altered by the action of strong alkaline lyes or acids, acquiring a greater affinity for dyestuffs and mordants. The strong contraction which takes place simultaneously, can be prevented by mercerising under tension and maintaining this during washing until the contraction in the fibre has ceased. The alkaline lye is preferably caustic soda of 15°—32° B., whilst, when employing acid, a sulphuric acid of 49°·5—55°·5 B. may be used, but this must be applied for a shorter time and the fibre at once well washed. In either case the reaction soon sets in, especially if the cotton be well freed from oil and treated in a somewhat damp condition. A parchment-like appearance of the material or fibre indicates the completion of the reaction. In an addition patent, No. 97,664, Sept. 4, 1896, the patentees state that the stretching to the original dimensions may be subsequent to the immersion in the mercerising bath. It is to be observed that Thomas and Prevost, in their German patent, do not mention the silky lustre which the fibre acquires after treatment, whilst Lowe, in his lapsed English patent, states that the treated fibre has a more silky appearance than the original. In all other essential points the two patents are very similar, although Lowe, it is true, does not allude in his last patent to the action of sulphuric acid; but this is not a practical method. It must be assumed that Thomas and Prevost discovered their process independently of Lowe, and to them is due the credit of having applied it technically. Their patent, so far as it relates to the treatment of cotton with caustic alkalis under tension, has been disallowed in England on the ground of the existence of Lowe's patent, but it is upheld in Germany. (See also Eng. Pat. 9517, 1897, this Journal, 1898, 573.)—T. A. L.

*Asbestos from Different Sources, Composition and Properties of.* P. Kersting. Chem. Ind. **21**, [9], 171—174.

The purest and most expensive asbestos is found in North Italy in light grey or light brown and occasionally in pure white masses consisting of very soft fibres of extraordinary length, which can be spun without previous treatment with acid. The mines, of which there are about 180, belong to the United Asbestos Co., Ltd., of London.

Canadian asbestos occurs in white masses which have the appearance of stones covered with a fibrous material. The fibres are shorter than the Italian, and are sometimes of a woolly nature. Before spinning, they require mechanical treatment and washing with hydrochloric acid and water. Asbestos of a quality equal to that of the Italian has recently been obtained from mines in the province of Quebec, of which the most important belong to the Boston Asbestos Packing Co. The yearly output of Canadian asbestos has increased from 300 tons, of a value equal to 19,500 dols., in 1879, to 7,630 tons, of a value equal to 420,825 dols., in 1894.

Asbestos occurs also extremely abundantly in Corsica, but owing to clayey impurities, it is very brittle and unsuitable for spinning. It is practically the only kind worked in Italy, owing to the English monopoly of the Italian mines, and fetches so low a price that it is even used instead of straw for packing.

South African asbestos has become of importance during the last few years, and an English company with a branch in Paris has been formed to work it. It is found in very delicate, silky, bluish-grey to blue masses, with threads of darker colour interspersed. The fibres are of extraordinary length, and can be spun well.

In the following analyses, No. 1 is a Canadian asbestos which consisted of long, silvery-grey, silky fibres, easily split into single threads. It contained hard, stony portions formed from the same material as the asbestos itself; these are not included in the analysis. Its price was 4 marks per kilo. No. 2 is also a Canadian asbestos, which consisted of somewhat shorter, dirty grey, woolly fibres. The stony portions were removed before analysing. Its price was 2 marks per kilo. No. 3 is a South African asbestos, which formed a light, bulky, bluish-grey, silky mass, containing darker threads, but no impurities. Fusion mixture was used for the determination of silica and bases, and sulphuric and hydrofluoric acids for the determination of the alkalis:—

| — | 1. | 2. | 3. |
|---|---|---|---|
| $SiO_2$ | 39·04 | 41·56 | 43·61 |
| $Al_2O_3$ | Traces | 3·42 | 0·21 |
| $Fe_2O_3$ | 0·25 | 0·41 | 16·57 |
| FeO | 2·10 | 6·67 | 12·15 |
| CaO | 0·51 | 0·83 | 0·89 |
| MgO | 42·57 | 33·88 | 7·02 |
| $K_2O$ | 0·09 | 0·11 | 3·06 |
| $Na_2O$ | 0·22 | 0·30 | 2·14 |
| $P_2O_5$ | .. | .. | 0·06 |
| $SO_3$ | 0·10 | 0·08 | 0·06 |
| $H_2O$ | 15·20 | 13·15 | 14·30 |

From the high percentages of water, it is evident that all three samples consist of serpentine-asbestos, and this is confirmed by their behaviour when heated: at 300° C., or by standing in a vacuum over sulphuric acid, little water is lost. When heated at 120° C. for several days, in accordance with Lindner's suggestion for the determination of hygroscopic water contained in serpentine, they lost 0·87, 1·79, and 1·25 per cent. respectively. Assuming, then, that a portion of the ferrous oxide has been converted into ferric oxide in the process of transformation from serpentine, the following formulæ may be calculated:—For Nos. 1 and 2, $MgSiO_3$, $Mg_2SiO_4 + 2H_2O$; for No. 3, $(MgFe)SiO_3 + H_2O$(Fe:Mg = 3·1). None of the samples could be melted by the blowpipe. When heated at 1,000° C. in the muffle, No. 1 became a little more brittle, No. 2 became of a reddish tint and very brittle, and No. 3 broke down to a hard red powder containing small crystalline needles. The residues, after ignition, when moistened with water, did not show an alkaline reaction to litmus.

The power of resisting the action of acids was determined by allowing 10 grms of the asbestos to remain with 100 c.c. of hydrochloric acid of sp. gr. 1·124 for 48 hours, and then weighing the residue after washing, and drying at 120°. By this treatment No. 1 lost 41·24; No. 2, 37·48; and No. 3, 12·62 per cent. of its weight. The Canadian samples became whiter, whilst the South African appeared unchanged. A rope of $\frac{3}{4}$ in. diameter made from the last, and exposed to a gas flame, supported a weight of 100 kilos. for 22 hours. The author regards the South African asbestos as superior to the Canadian, except as regards its resistance to heat. This exception would prevent its use in places where the temperature ever rose to more than 200°.—E. W. W.

## PATENTS.

*Textile Vegetable Fibres, Impts. relating to the Treatment of.* C. W. Radeke, Loughborough. Eng. Pat. 15,642, July 1, 1897.

Vegetable fibres, especially china grass, &c., are heated to a temperature a few degrees below the boiling point, in a bath of about equal parts of urine and water, with the addition of about 4 oz. to the gallon of sodium chloride. After about an hour's digestion the liquid is run off, and a hot solution of tartaric acid ($\frac{1}{2}$ oz. to 1 oz. to the gallon of water) is poured on the fibres. After an hour or two the fibres are removed, and in their moist state exposed to air at from 30° to 50° C. for a stated time, and are then treated in a hot bath containing from $2\frac{1}{2}$ to 5 parts of soap to 100 parts of water; after two or three hours a small proportion of ammonium carbonate is added to the bath, and the fibres are agitated until freed from gummy matter. Lastly, the fibres are rinsed successively in hot and cold water, and dried. In the first bath used, urine may be replaced by a stated proportion of ammonium carbonate, phosphate, or chloride.—E. S.

*Nitrocellulose, Impts. in Dissolving, and in the Treatment therewith of Vegetable Textile Material, such as Cotton Yarn, for imparting to it a Silk-like Gloss.* J. Wetter, London. From Heberlein and Co., Wattwyl, Switzerland. Eng. Pat. 17,302, July 22, 1897.

Nitrocellulose is steeped, for at least an hour, in alcohol of 95 per cent., whereby it becomes soluble in solutions of many inorganic salts. Preferably, the treated nitrocellulose is dissolved in caustic soda solution of from 5° to 10° B. to the extent of from 3 to 5 per cent. Yarn, in hanks of any desired length, may be passed through this solution and subsequently dyed without losing the gloss thus acquired. Reference is made to Eng. Pat. 13,198, 1896 (this Journal, 1896, 650).—E. S.

*Size, the Continuous Treatment of, by Boiling: An Apparatus for the.* A. Stephan, 30, Breitenbach, Muster, Alsatia. Eng. Pat. 5164, March 2, 1898.

This invention relates to an apparatus for the continuous preparation of size, in which the manufacture of the latter is not effected by boiling direct with steam mixed with the size, as is done at present, so that the weakening of the size by dilution is avoided. The apparatus further serves to render the preparation of the size one continuous operation, the raw size being continuously pumped into a closed boiler having double walls. The boiler is heated by steam, which is introduced between the walls, so that the size is brought to the required temperature under pressure, in order to be afterwards continuously fed into the size trough. This apparatus allows of the quantity of the material to be fed into the size trough being accurately measured, so that the same is adequate to the quantity of yarn or other material to be treated.—J. C. P.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Dyeing, Theory of.* R. Gnehm and E. Rötheli. Zeits. f. angew. Chem. 1898, 482—488.

The authors criticise the various dyeing theories, which may be shortly stated as follows. According to the views of different investigators, the process is either purely chemical (salt formation) or entirely mechanical, or is intermediate between these two, *i.e.*, is a function of the separate fibres and not of the whole material. Others, again, consider that a kind of solid solution takes place; and finally there are others who maintain that dyeing is not a homogeneous process at all. Knecht and Vignon are the principal upholders of the chemical theory, the latter from thermo-chemical investigations and the former from his observations that acid dyestuffs are taken up by wool in molecular quantities, and further, that wool itself, on boiling, yields lanuginic acid, which forms insoluble lakes with all substantive dyestuffs. Reisse, who studied the behaviour of dyestuffs towards mixtures of wool and silk, came to the same conclusion. The principal advocate of the mechanical theory is Georgevics, who considers dyeing as an analogous phenomenon to the condensation of gases on solid bodies (carbon, glass, &c.), which du Bois-Reymond has termed adsorption in contradistinction to absorption, which is applied in the case of liquids. He bases his views on the assumption that no molecular proportion exists between dyestuff and fibre, and that the products exhibit the original properties of the components; this is also specially pointed out by Hwass and von Perger. Two important facts in support of the mechanical theory are, according to Georgevics, the dyeing of indifferent substances and the property possessed by many dyestuffs of subliming from the fibre. He considers as conclusive evidence the fact that in the case of two dyestuffs, Indigo Carmine and Methylene Blue, the coefficient of division between fibre and dyebath is a constant. Weber holds that different fibres behave differently. Thus, substantive dyestuffs fix themselves as lakes on wool and silk, whilst Aniline Black dyeings and azo colours formed on the fibre, &c. are purely mechanical precipitates, like pigments. (See also Prud'homme, page 663.) But substantive cotton dyestuffs are solutions of the dyestuff in the cell sap, their relative stability to washing being due to the smaller diffusion coefficient of the dyestuff. Witt considers dyeing as a case of solid solution, and compares the process to that employed in extracting aqueous solutions with benzene, ether, &c. Finally, Gnehm maintains that dyeing, even for one and the same fibre and the same dyestuff, is not to be considered as a homogeneous process.

With the object of gaining further information with regard to these different views, the authors have repeated several of the experiments and performed new ones. In the first place, glass beads were immersed in hot aqueous solutions of Magenta, Magenta and ammonia, and rosaniline base, and left for 11 weeks, when they all showed the same appearance. On washing with alcohol, however, the beads dyed in the two latter solutions soon lost all their colour, whilst those from the Magenta bath retained their colour even after continued extraction. This coincides with Gnehm's earlier experiments. The results in the case of silk were not so concordant, and it was observed that the presence of moisture in the silk had some effect. In any case it was never possible to completely exhaust the silk; and an experiment comparing the effect of a concentrated solution for a short time with that of a very dilute solution for a long time showed that on extraction the latter was least altered. From this it would appear that Magenta dyes partly chemically and partly mechanically, the latter preponderating; but since different results are obtained by the use of different solvents, some unknown factor must take part in the dyeing or extraction; and from these experiments it can only be deduced that dyeings with colour salt or base do not always behave similarly, that the colour is not entirely extracted, and that the amount of the colour not extracted is dependent upon the duration of the dyeing process. Georgevics' statement with regard to the stability of the carbinol base of Magenta, on investigation, led to the result that it is tolerably stable, and requires somewhat strong acids to convert it into the ammonium derivative. The silk in this case acts as an acid. As already mentioned, the fact that only part of the dyestuff can be extracted, is evidence in favour of the chemical theory of dyeing, and this received further confirmation in a series of alkylation experiments to determine whether the dyed pattern behaved like a salt or base. A skein of silk dyed with Magenta was allowed to stand in the cold in methyl iodide and methyl alcohol, side by side with rosaniline base, rosaniline hydrochloride, stearate, and amido-stearate; also in presence of methyl iodide and alcohol. All these tests remained unaltered, with the exception of the free base, which turned deep blue, but on warming to 35°—40° C. for about 20 hours, the salts, as well as the skein, were alkylated. This appears to point to the formation of a salt in the process of dyeing, although all dyestuffs are not necessarily taken up in the same manner. It follows, from the foregoing, that, in the case of animal fibre, the dyeing process is probably a chemical one, but in no way could this be assumed with regard to cotton fibres. According to Weber the Benzidine dyestuffs attach themselves as salts to the fibre, and, in order to test this, 100 grms. of cotton were immersed in boiling baths containing 1 grm. of the purified barium salt of Benzopurpurin 4 B and Benzazurin 3 G until the baths were exhausted. After well washing the fibres and drying, the barium was estimated by means of sulphuric acid, and the barium sulphate obtained, weighed. It corresponded in amount to that originally taken, so that Weber's supposition is correct. It was also observed that, owing to the small coefficient of diffusion of the barium salts, they only dyed slowly; but, on the other hand, they did not bleed at all. On examining the dyed fibres by means of Röntgen rays it was found that Magenta, Crystal Violet, and Malachite Green (oxalate) on wool and silk are quite transparent; cotton dyed with the sodium salt of Congo Red partially absorbs the rays; whilst the dyeings with the barium salt are opaque to a high degree, which is confirmatory evidence that the benzidine colour salts dye intact. Microscopical examination of transverse sections of the dyed fibre showed that wool dyed with Malachite Green or Crystal Violet was impregnated throughout with the colour, whilst cotton dyed with Congo and Benzazurin is deepest at the centre. Magenta, on silk, appears homogeneous, and the same is true of a silk fibre dipped in an iron salt. When this is treated with ferrocyanide, only the outer layers become stained, and when treated with catechu the ring is only somewhat intensified. It would appear that part of the colour first formed acts as a filter, and, after saturation of the external layers, a further infiltration takes place. In order to test the statements of Georgevics and Knecht that colour salts are decomposed on dyeing, the authors employed dyestuffs combined with acids or bases having high molecular weights. For example, basic dyestuffs were combined with palmitic or stearic acids and acid dyestuffs with organic bases. Silk dyed with these salts was dissolved in hydrochloric acid, when any fatty acid would manifest itself or could be extracted with ether. It was, however, found that if the fibre, after drying, had been passed through benzene to remove any acid adhering mechanically, no fatty acid remained on the fibre. This indirect proof, which agreed with Knecht and Georgevics' observations, was further confirmed by an investigation of the benzidine salt of Naphthol Yellow S. In the case of wool, owing to much decomposition product in the bath, the number obtained was somewhat high, but with silk very close numbers for the benzidine remaining in the bath were obtained. The existence of dyestuff salts with relatively weak acids of high molecular weight is a further proof of the chemical theory of dyeing, and at the conclusion of the paper several of these salts are described. The general method employed for their preparation was to add the finely powdered base (precipitated cold by ammonia) to the molten acid, the salt, after cooling, being purified by means of alcohol. The salts form metallic powders, but do not possess any crystalline appearance. In an impure state these substances have long been known. They are probably contained in the colours used for the dry dyeing of velvet, &c. The material is worked in a benzene solution of the colour until the desired effect is produced, and then passed through a benzene bath apparently to remove adherent fatty acid. The following table gives the melting points and solubilities of the palmitates prepared ; *e* signifies easily and *s* sparingly soluble :—

| Palmitate of | Melting Point. | Colour and Solubility in | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Alcohol. | Chloroform. | Ether. | Acetone. | Benzene. | Carbon Bisulphide. | Petroleum Ether. |
| | ° C. | | | | | | | |
| Auramine, $C_{33}H_{51}N_3O_2$........ | 57 | Yellow, *e* | Yellow, *e* | Yellow, *s* | .. | Yellow, *e* | Yellow, *e* | .. |
| New Magenta ................ | 79 | *e* | *e* | .. | *e* | Insoluble | .. | Insoluble |
| Pararosaniline................ | 85 | *e* | *e* | .. | *e* | Insoluble | Insoluble | Insoluble |
| Night Blue .................. | 88 | Blue | Blue | Deep red | Violet | Deep red | Deep red | Deep red |
| Crystal Violet. ............... | 140 | Violet | Violet | Red | Violet | .. | Blue | Violet |
| Victoria Blue B .............. | 99 | Blue | Violet | .. | Blue-violet | Red | .. | Red |
| Victoria Blue 4 R ............ | 80 | Blue | Violet | .. | Violet | Red | .. | Red |

—T. A. L.

*Mercerised Cotton Yarn, Dyeing of, with Diamine Colouring Matters.* Leipziger Färber- u. Zeugdr. Zeit. 1898, **47**, [6], 243.

Cassella and Co. give the following methods for dyeing mercerised cotton :—

After mordanting with tannin and antimony (tartar emetic) in a bath containing 25 per cent. less mordant than is required for ordinary cotton, basic colouring matters are applied in a cold bath well acidulated with acetic acid.

Diamine colouring matters are dyed as usual, but since mercerised cotton possesses a much great affinity for these dyestuffs than ordinary cotton, it is best to prepare the bath as follows :—1 per cent. of soda, and 1—2 per cent. of Turkey-red oil are dissolved in water, the dyestuff then added, and after treating the goods for half an hour, for light shades, 2—5 per cent. of sodium phosphate, for middle and dark shades 3—8 per cent. of Glauber's salts, and for black 15—20 per cent. of the latter is added. Light shades are entered lukewarm, and after the addition of the phosphate, the bath is heated to 50°—60° C. Darker shades are best obtained by entering the goods at 50°—60° C., and heating to the boil after the addition of the Glauber's salts. In some cases, soap must be used instead of Turkey-red oil, when the amount of soda is increased to 2—4 per cent. Topping with basic colours takes place best in a bath slightly acidulated with acetic acid.—H. I.

*Fastness of Dyestuffs on Cotton, Proposed Standard Tests for.* E. Blondel. Rev. Gén. des Mat. Col. 1898, **2**, 189—192.

With a view to securing uniformity in methods of determining the fastness to various agencies of cotton dyes, it is suggested that standard tests should be adopted. The following are proposed :—

For measuring the fastness to soap, a standard solution is prepared of 50 grms. of white Marseilles soap and 10 grms. of sodium carbonate in 10 litres of distilled water. 100 grms. of this are weighed out into a copper or porcelain vessel, to which an arrangement for the maintenance of a constant level is attached, the water evaporated during the test being replaced by distilled water. The test is made by boiling in this solution for 10 minutes, 1 grm. of the dyed cotton fabric to be tested, along with 0·5 grm. of the same kind of fabric in the bleached condition, the two being tied together. The samples are then rinsed in water, dried, and examined.

In the case of yarns, the fastness to soap may also be estimated by immersing the sample to be examined, together with bleached yarn, both in the form of hanks, in a sufficient quantity of water at 45°—50° C. to moisten the hanks thoroughly, and soaping them by hand. At the end of 10 minutes the water is renewed, and the operation repeated ; this is continued as long as any dyestuff is removed, after which the hanks are rinsed and dried.

Dyed yarns intended for the manufacture of mixed cotton and woollen tissues, which have ultimately to be milled, are subjected to a milling test, along with hanks of wool, on the small scale.

Fastness to light is determined by exposing under a frame, with free access of air, for 30 days in the months of May, June, July, and August, the frame being placed in a vertical position, facing the west.

Fastness to rubbing and to hypochlorites should also be tested. Precise methods for ascertaining this, however, are not given in the original article.—E. B.

*Turkey Red [Calico Printing], Alkaline Discharge of.* W. Trjapkine. Rev. Gén. des Mat. Col. 1898, **2**, 180—189. (See this Journal, 1898, 347.)

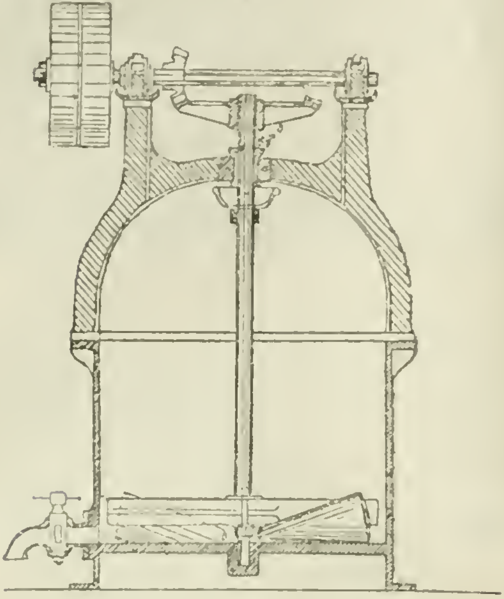
*Blue [Indigo] Discharge.*—This is obtained with indigo, reduced by means of glucose and caustic soda, under the influence of steam at 100° C. There are two ways of effecting the discharge. One is to prepare the tissue with glucose and to print a mixture of indigo and caustic soda. The other is to mix all three reagents together in the cold. Both methods are practised.

Java indigo is mostly used for the preparation of the discharge mixture, owing to its softness, which enables it to be readily ground, the large percentage of colouring matter it contains, and its freedom from calcium carbonate—a compound which in the course of the discharging operations is liable to lead to the formation of sparingly soluble alizarates, and thus to render the discharge dull in shade.

In addition to this kind of indigo, the better classes of Bengal indigo and refined natural indigos are employed.

The new synthetical product of the Badische Anilin und Soda Fabrik can hardly be said to have found a definite place in calico printing. Although its purity, constant composition, and fine state of division are greatly in its favour, preliminary trials made with it would seem to show

Fig. 1.



Indigo-grinding Machine.

that it is fixed on the fibre with more difficulty than natural indigo, and that it gives paler and greyer shades than the latter. These difficulties are, perhaps, to be ascribed to the absence of indirubin from it.

The important operation of grinding the indigo for printing is performed in either a cradle-mill or an apparatus of the kind shown in the figure.

For the first of the above-mentioned methods of printing, the tissues are prepared with glucose solution of 5°–8° B. at 50°—60° C.

On the tissues thus prepared, a blue discharge-mixture such as the following is printed :—2·5 kilos. of British gum, 7 kilos. of water, 10 kilos. of caustic soda at 50° B., 3·75 litres of indigo (78—80 per cent.) paste (40 per cent.), 2 litres of sodium silicate at 38° B., 1 litre of glycerin at 28° B., 0·4—0·5 litre of turpentine.

The following is an example of a printing mixture for the production of a discharge by the second method :—1 kilo. of dextrin solution (10 : 10), 1 litre of water, 10 litres of caustic soda at 50° B., 3·5—3·75 litres of indigo paste (40 per cent.), 1 litre of sodium silicate at 38° B., 8¼ litres of glucose solution at 32° B., 0·75 litre of glycerin at 28° B., 0·4—0·5 litre of turpentine. In this case only a small proportion of thickening agent is required, as the glucose serves in part to thicken the mixture. In adding the indigo paste to the thickened solution of caustic soda, care is taken that the temperature of the mixture shall not exceed 40° C.

*Green Discharge.*—A mixture of the blue and yellow discharge colours is employed for green.

The colour is generally printed on tissues prepared with glucose, as this method admits of the use for the green, of the blue colour-mixture which has become soiled by the transference into it of other colours (mainly the yellow) during printing, the blue discharge-mixture containing glucose not keeping sufficiently well to be used in this manner.

The printing colour may be prepared as below :—*Blue mixture for green :* 2·5 litres of dextrin solution (10 : 10), 2·5 litres of caustic soda at 50° B., 2·3 kilos. of indigo paste (40 per cent.). *Printing mixture :* 10 litres of yellow discharge-mixture (this Journal, 1898, 317), 3·5 litres of blue mixture for green, 0·25 litre of turpentine.

*Black Printing Colour.*—Vanadium and potassium ferrocyanide Aniline Black mixtures are used, the former being preferred for 5—6, the latter for 2—3 colour patterns.

More concentrated mixtures than those commonly employed are required. This is especially the case with those which are printed on tissues dyed with Alizarin, and which, when white and yellow discharges are to be produced, have to withstand treatment with bleaching powder after printing. For tissues prepared with glucose at 5°—7° B. the proportion of alkali chlorate needed is 75—80 grms. per litre.

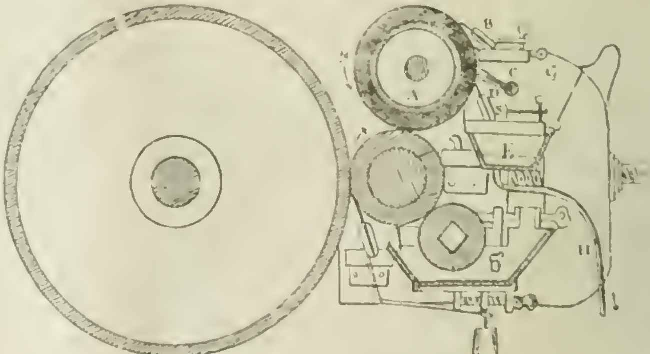
The black mixture is supplied to the printing roller by means of a brush-furnisher. To prevent as much as possible the alkaline colours transferred from the tissue from spoiling the black, an unengraved roller, working in an acidified gum solution, is inserted in the printing machine in front of the roller by which the black mixture is applied. This arrangement enables 20—30 pieces of tissue to be printed before the printing colour is renewed.

In the case of 4—6 colour designs, the apparatus shown in the figure is used with advantage.

It consists of a cast-iron cleaning-roller (A), coated with india rubber, placed in contact with the printing roller and driven by it. The colours transferred from the tissue to the printing roller are for the most part in turn transferred to this, from which they are removed by the doctor, D. C is a spirt pipe, and B a doctor, the function of which is to remove the excess of moisture from the surface of the cleaning roller. An india-rubber pipe, H, conducts away from the machine the colour and water as they flow from the cleaning roller into the trough, E.

With this arrangement 40—50 pieces of tissue can be printed before the printing colour becomes too much deteriorated to use.

Fig. 2.



Calico-printing Apparatus.

The colours are commonly printed in the order, pink, white, yellow, green, blue, black. Sometimes the order of the last two is reversed in order to obtain a brighter shade of blue.—E. B.

*Wool, Scouring and Bleaching of.* Leipziger Färber- u. Zeugdr. Zeit. 1898, **47**, [6], 271—273.

The oil, from the spinning and weaving operations may be removed from fabrics and yarns by subjecting them to the action of steam and water in a closed vessel. The oily matters rise to the surface, and are skimmed off before removing the goods. This treatment, though it suffices for wool to be dyed with acid colours, has to be modified when the goods require mordanting, as for alizarins. Such wool is scoured in a 2½—3 per cent. solution of soda, with the addition of 3—4 per cent. of soap, for 1—1½ hours at 40°—50° C., and is afterwards washed with dilute ammonia and water.

*Bleaching with Sulphur Dioxide.*—The objectionable spotting of pieces by the dropping of condensed moisture from the roof of the stoving chamber may be avoided by placing steam pipes in the upper part of the stoves.

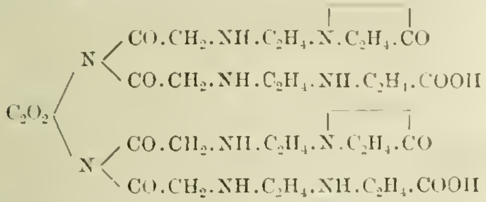
The last traces of sulphur dioxide, which are almost impossible to remove by washing with water, may be oxidised by treating the goods with dilute hydrogen peroxide solution. The sulphuric acid thus formed is readily removed during the washing processes.

*Bleaching with Sodium Peroxide.*—This material has, on account of its cheapness, replaced hydrogen peroxide.

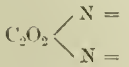
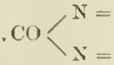
For every 100 kilos. of wool about 10 kilos. of sodium peroxide are required. Into a sufficient quantity of water to wet the wool, containing 1·33 kilos. of sulphuric acid (66° B.) for every kilo. of sodium peroxide, the latter is slowly added with continued stirring. The solution is then neutralised either with more peroxide, or more sulphuric acid, and 1—2 kilos. of water-glass (45° B.) run in until the solution is alkaline to litmus. When ready, the wool is introduced into the cold solution, which is then gradually heated to 40°—50° C. After working for 2—3 hours it is wrung out, washed with dilute sulphuric acid, and finally with water.—H. I.

### *Wool, On the Dyeing Properties and Constitution of.* M. Prud'homme. Rev. Gén. des Mat. Col. 1898, **2**, 209—213.

Schützenberger has shown that the products of hydrolysis of wool by baryta water are analogous to those of ordinary albumin; that is, they contain products having a formula similar to that of gelatin—



and to albumin, which contains the typical groups—

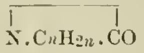
 and the carbamide group, 

The author points out that the ordinary operations of washing and bleaching wool with agents such as hydrogen peroxide, dilute acids, alkalis, and bleach, play an important part in determining the affinity of the wool for both acid and basic colouring matters.

Wool, after treatment in various baths (such as, for example, 500 grms. of sodium sulphite, 1,000 grms. of water, 30 grms. of sulphuric acid for about 24 hours, or this treatment followed by digestion with hydrogen peroxide solution, &c.), was dyed with typical acid and basic colouring matters. Taking 100 as the maximum intensity of colour, the following table shows approximately the results of the experiments:—

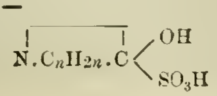
| Experiment. | Treatment. | Intensity of Colour. | |
|---|---|---|---|
| | | Basic Colours. | Acid Colours. |
| I. | $SO_2$ | 50 | 40 |
| II. | $SO_2 + H_2O_2$ | 100 | 50 |
| III. | $SO_2 + Na_2CO_3$ | 30 | 100 |
| IV. | $SO_2 + H_2O_2 + Na_2CO_3$ | 80 | 90 |
| V. | Water only | 20 | 70 |

These results are quite in accordance with wool having in its constitution the group—



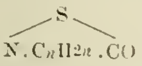
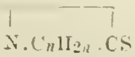
$$\overline{|\;N.C_nH_{2n}.CO\;|}$$

*e.g.*, on treatment with sulphurous acid this group would probably become—



thus the acid function of the molecule becomes accentuated, and therefore its affinity for basic colouring matters is increased, as is shown in the table. The other results can be explained in a similar manner.

The sulphur in wool is probably combined, either as—

 or 

$$\overline{|\;N.C_nH_{2n}.CS\;|}$$

it is also contained in the yellow natural colouring matter in wool. Experiments on the quantity of Magenta which untreated wool will take up, point to the conclusion that wool contains less than four carboxylic groups.

It is well known that wool will absorb nitrous acid and combine with phenols in alkaline solution; it is therefore supposed to contain $NH_2$ groups, whereas Schützenberger's formulæ contain no $NH_2$ groups, but only NH groups. It is pointed out that nitrous acid could form nitrosamines with the NH groups, which would also give colours with phenols.—C. M.

### *Paranitraniline Red, Perfected Method of Dyeing with.* A. G. Green. Rev. Gén. des Mat. Col. 1898, **2**, 214—216.

After several experiments on the development of *p*-Nitraniline Red on mercerised and unmercerised cotton, the author concludes that the β-naphthol, in certain quantity, combines intimately with both.

The maximum quantity of β-naphthol which gives the best shade is 2 per cent. for unmercerised and 1 per cent. for mercerised cotton; hence in the latter the combination is more intimate. The shades produced by these quantities of β-naphthol are practically identical, and it is claimed that, by the new method with mercerised cotton, in spite of the cost of preparation of the latter, the total cost is less, owing to the decreased quantities of paranitraniline and β-naphthol required.

The following is an example of the new method:—The cotton, bleached or not, is immersed for 1 hour in a cold solution of caustic soda, 20° B., and is then washed until most of the soda is removed. It is now passed, in quantities of 1 kilo. at a time, through a bath containing 11¼ litres of "Naphthol Solution P N" (Clayton Aniline Co.), and 11¼ litres of water; after each kilo., 85 c.c. of naphthol solution P N must be added to the bath. The cotton is now rolled and dried. The diazo solution is prepared thus:—For every 75 kilos. of cotton, 1 kilo. of paranitraniline X F (Clayton) is mixed with 2½ litres of cold water, 2 litres of HCl 35 per cent. or 2½ litres of HCl 28 per cent. The mixture is agitated, diluted with 5 litres of cold water, then 562 grms. of nitrite of soda, dissolved in 2½ litres of water, are added, all at once. The whole is diluted to 30 litres, and keeps well. To develop, to 2½ litres of this solution add 12½ litres of cold water and 2½ litres of sodium acetate solution, obtained by dissolving 1 kilo. 815 grms. of acetate and 345 grms. of carbonate of soda in 30 litres of water. Add the cotton in quantities of 1 kilo. at a time, and after each development add 375 c.c. of the diazo solution and 375 c.c. of the sodium acetate solution.

When 75 kilos. of cotton are thus developed, to finish they are soaped for 10 minutes with ¼ per cent. soap solution at 60° C.—C. M.

### *Paranitraniline Red and Allied Colours, Note on some Properties of.* E. Knecht. J. Soc. Dyers and Colourists, **14**, [6], 123.

In the discussion following the paper on "Paranitraniline Red in Dyeing and Printing" (this Journal, 1897, 139), J. Rhodes referred to the solubility of Paranitraniline Red in organic acids, such as acetic and tartaric.

Further investigations have been made by the author, who experimented upon the insoluble azo dyes obtained by precipitating diazotised paranitraniline, α-naphthylamine, and β-naphthylamine with sodium β-naphtholate. All three products are volatile, which confirms the results of practical experience with regard to hot pressing; for it is known that such colours interwoven with white, mark off badly when hot-pressed.

In accordance with the observations of Rhodes, Paranitraniline Red was found to be soluble in acetic, tartaric, and citric acids, but not in formic (50 per cent.). It is insoluble in boiling water, hydrochloric acid, and aqueous alkalis, but soluble in alcoholic caustic soda with a violet colour. It dissolves in concentrated sulphuric acid to a red solution, which on dilution with water turns red and then orange.

It is also more or less readily soluble in benzene, toluene, chloroform, carbon bisulphide, methyl and ethyl alcohols, and ether From some of these solvents it can be easily crystallised. The colours obtained from diazotised α- and β-naphthylamine behave similarly to Paranitraniline Red, being however more readily soluble in benzene and toluene.

α-Naphthylamine Maroon and β-Naphthylamine Red, in contradistinction to Paranitraniline Red, are extremely difficult to discharge by means of reducing agents. All three azo dyes mentioned are soluble in oils—*e.g.*, olive oil—and in molten paraffin wax.

In the discussion which followed this paper, Jansen stated that the action of nitric acid on Paranitraniline Red gave a remarkable result. It dissolved in the acid to a clear solution, which was found to contain diazotised paranitraniline and nitronaphthol.

Thus the failure of the author to produce Paranitraniline Red on the fibre, by treating with strong nitric acid cotton dyed with the orange benzene-azo-β-naphthol, is accounted for.—J. F. B.

*Paranitraniline Red, Tannin Resist Styles for.* J. Langer. Bull. de Rouen, 1898, [1]; Leipziger Färber- u. Zeugdr. Zeit. 1898, **47**, [6], 241—242.

The property of tannin to form insoluble lakes with diazo- and tetrazo compounds is employed.

For *white resist styles* the fabric, prepared with β-naphthol, is printed with a composition containing tannin, 100 grms.; water, 250 grms.; gum solution (1 : 5), 200 grms.; glycerin, 50 grms.; soap solution (made by dissolving 168 kilos. of commercial oleïne in 82 kilos. of caustic soda solution of 36° B., and diluting to 1,200 litres) 100 grms. On introducing the goods into the developing bath of diazotised paranitraniline, the tannin, combining with the diazo compound, prevents its action on the β-naphthol; on washing and soaping, the tannin lake is removed and white effects are obtained.

*Coloured styles* are obtained by printing along with the tannin, basic colouring matters, such as Methylene Blue, Auramine, Victoria Blue B, &c., tartaric or citric acid and acetin. Before entering the diazo-developing bath the tannin colours are fixed by several passages through the Mather and Platt machine, and after dyeing and washing follows a treatment in a tartar emetic bath before soaping. The basic colouring matters and tannin are dissolved in dilute acetic acid, the gum solution is replaced by a thickening made of starch and dextrin, and no soap solution is used.

—H. I.

*Paranitraniline Red, Solvents for.* C. Kurz. Rev. Gén. des Mat. Col. 1898, **2**, 188.

*See under* XXIII., *page* 698.

*Chromous Acetate, Action of Certain Carbonates on.* G. Baage. Comptes Rend. **126**, 1566—1569.

By treating an aqueous solution of an alkaline carbonate with chromous acetate, the authors prepared double carbonates containing protoxide of chromium and ammonium or sodium (Comptes Rend. **122**, 174, and **125**, 1177). In the present communication they show that similar double carbonates may be prepared, containing potassium or magnesium, the former being obtained as follows:—A current of carbonic acid is passed through the tubulated flask already described (*loc. cit.*) and chromous acetate suspended in water, introduced. After standing, the clear liquid is decanted and replaced by a 20 per cent. solution of potassium carbonate in water previously boiled. On shaking, the chromous acetate dissolves, and in a short time a yellow crystalline powder separates. The precise concentration mentioned is essential to success. The product, after decantation, is washed with a little boiled water until free from potassium acetate, then several times with alcohol, and finally is dried in the same flask on a water-bath. It may also be prepared by adding a tepid saturated solution of potassium bicarbonate to moist chromous acetate; in this case carbonic acid is evolved. The salt is a yellow powder, closely resembling ferrous oxalate in colour. Under the microscope it is seen in the form of citron yellow hexagonal prisms. It dissolves in water with a yellowish-brown colour, but is slowly deposited therefrom in a modified form, insoluble or only slightly soluble in water. The salt is also precipitated from its aqueous solution by the addition of potassium carbonate. Like the corresponding sodium and ammonium salts, it is an energetic reducing agent, decomposing water below 100° C. Heated *in vacuo* or in an inert gas it becomes brown, reverting to its original colour on cooling. This property is retained up to nearly 280° C., at which temperature the salt decomposes. It is stable in dry air for some time, but in moist air is oxidised, chromic hydrate being formed. Weak acids, if air be excluded, dissolve the salt with a blue colour. Analyses, details of which are given, agree well with the formula $(K_2CO_3CrCO_3)_2 3H_2O$. Another potassium salt has also been prepared, of uncertain composition, distinguished by its red-brown colour. A red-brown compound of uncertain composition is obtained by adding moist chromous acetate to a saturated solution of magnesium carbonate in water charged with carbonic acid. This compound decomposes water at 100° C., but attempts to form similar double salts with the carbonates of the alkaline earths, failed.—T. A. L.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Thirty-fourth Annual Report on Alkali, &c. Works, by the Chief Inspector.* Feb. 9, 1898.

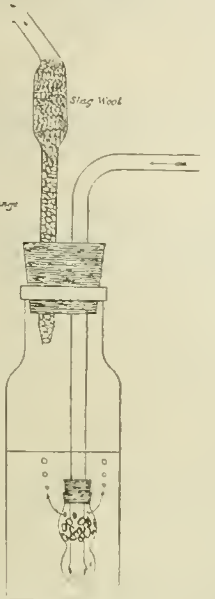
The present Report, the third presented by Mr. R. Forbes Carpenter, shows a diminution in the number of works registered, as compared with the preceding year, for England, Ireland, and Wales, of 16, the numbers being, for 1896, 1,074, and for 1897, 1,058, the difference being chiefly in alkali works, which were 87 in the latter year as against 98 in the former year. There is no material change, as compared with the two preceding years, in the amount of the escape of acid gases. Three prosecutions have been instituted under the Acts, in only one of which a substantial penalty was exacted. The system so wisely inaugurated when Lord Derby made the happy selection of the late Dr. Angus Smith to administer the then recently passed Acts, in consideration for the needs of the manufacturers, and of assisting them with such skilled advice as only experience distributed over wide areas, joined with high scientific attainments, could afford, has, as continued by his successors, enabled the working of the Acts to be not only, as primarily intended, a protection from avoidable nuisance to the general public, but an actual boon to the manufacturers themselves. Thus, the present inspector writes: "High as is the standard of management of the United Alkali Company, who control so large a portion of this industry [salt-decomposition], their highly-skilled staff do not feel that it would be desirable, even if it were possible, to dispense with the check on the conduct of their operations that is supplied by the constant visits of trained independent chemists, acquainted by long experience with the operations conducted, with the difficulties presented, and with their possible solutions."

Some interesting remarks are quoted from Dr. James Young, of Bristol, on the influences of alkali and of tar works on the health of the surrounding districts. It appears that the vapours evolved on the running-off of pitch are so highly valued as a remedy for whooping-cough that children suffering from this complaint are sent to such works to inhale the vapours. In such neighbourhoods, notifiable diseases are fewer, and epidemics seldom occur. A mild epidemic of measles that broke out in 1896 in the chemical works area had no fatal cases, while beyond this district the epidemic was severe and "very fatal." Pulmonary affections, and especially phthisis, are mitigated in such localities. The death-rates also bear a very favourable comparison with those of outlying districts. Vegetation, however, suffers from vapours which are harmless to the inhabitants.

Hargreaves' process for the manufacture of sodium chlorate from chlorine and sodium carbonate pentahydrate has been successfully worked on a large scale during the past year, compact tower absorbers of special construction having dealt efficiently with the waste gases. There is stated to be strong competition on the Continent in this manufacture, conducted by electrolytic processes, with water power as the source of energy. At home, chlorine is actively manufactured by electrolytic processes in four works, and the commercial practicability of the Hargreaves and Bird electrolytic cell, decomposing 10 lb. of salt per hour with 100 feet of diaphragm surface, is being tested, with a view to extensive working in the present year. In one of the four works where chlorine is produced electrolytically, a solution of zinc chloride is decomposed; in the others sodium chloride is used. The workpeople do not appear to suffer any injury to health in these processes, notwithstanding the use of mercury; and the absorption of chlorine in the manufacture of bleach is satisfactory. Three works in the United Kingdom (inclusive of one in Scotland) produce chlorine by the nitric acid process, liquid hydrochloric acid, obtained in the usual way, being employed, objections having arisen to the proposed use of the gaseous acid. The large quantity of dilute sulphuric acid resulting from the nitric acid process, which it is necessary to reconcentrate, makes special attention to the prevention of the escape of acid vapours of great importance.

In sulphuric acid works, and especially in such as are not provided with Glover and Gay-Lussac towers, the inspector recommends the use of Strype's form of the automatic-balanced damper. "The balance is obtained by use of a hollow bell-shaped float of lead, communicating freely with the exit flue below, and to which the damper or valve is attached; the liquid in use to float the luted bell being in some cases glycerin, where the apparatus is exposed to frost. When balanced by a counterpoised weight at the other end of the steelyard, this bell is very sensitive to changes of draught, and a great uniformity of pull on the chamber is, by this simple means, automatically attained." The Lunge-Rohrmann tower, though only used as yet in one works in this country, is coming into extensive use abroad, especially as a denitrator on the top of the Glover tower, whereby the latter is confined to the work of concentration only. In works using spent oxide as source of sulphur, and where consequently the nitrogen oxides are in danger of being destroyed by ammonia or cyanogen compounds, the application of the Lunge-Rohrmann tower is particularly valuable. The introduction of cooling shafts in sulphuric acid chamber space, has, in one works, increased the productive power 20 per cent. The advantages attending the use of the Delplace system of chambers, and of the interposition between chambers of a series of towers, as described by Hacker and Gilchrist (this Journal, 1894, 1142) are also stated. The Kessler form of concentrating apparatus continues to give satisfaction in Scotland, where its use has extended to three works.

Considerable difficulty has been found in working Hasenclever sulphuric acid concentrating pans, as well as in the system known as Webb's process, in effecting complete absorption of the acid vapours. In testing, it was found that to absorb the gases completely, required most violent and prolonged shaking of the bellows aspirator, as otherwise "the acid indicated by the test was often one-third less than that actually present," and as long as any of the "white vapour" absorbed with difficulty, remained, some hydrochloric acid also remained unabsorbed. The experiments instituted by the inspector showed that the formation of this fume depended on the rate at which the dry or moist air, $CO_2$, $SO_2$, &c., were introduced, rather than on the nature of the gas used. The introduction of a cold gas caused immediate condensation of the white fume, which, after disappearing in the neck of the retort, reappeared in the Woulff's bottle, and even traversed the final condenser and soda solution. After many experiments the scrubbing bulb and apparatus here sketched was adopted.



The solution in the bottle is water and hydrogen peroxide. It will be seen that the gas is broken up into bubbles by passing through the holes pierced in the inner tube, and then comes into contact with the rubber clippings in the space between the inner tube and outer perforated bulb. The rubber clippings in the exit tube are kept moistened with water coloured with methyl orange, which indicates at once the passage of any acid vapour. A series of tests is given, proving the satisfactory working of the apparatus. Curious anomalies were found in the testing of acid escapes, notably when made with different apparatus, and even to some extent when made with the same apparatus when the gases were drawn from different points in the diameter of the flue. These anomalies have been made the subject of careful investigation, details of which are given. Some ingenious experiments are also described having for their object the determination of the extent of area from which a tube could draw gases, moving at a known velocity, across the direction of the tube. The contributory area had, in the case of a tube of 1·5 mm. diameter, a maximum radius of 4 mm., and in that of a tube of 3 mm. diameter, a maximum radius of 6 mm. under the conditions of the experiment.

A statement in tabular form is given of the results of the working of oxide of iron purifiers with concurrent revivification by regulated steam and air admission, reference to which was made in the two previous reports. From 87·5 to 90·4 per cent. of free to total sulphur was obtained in the different runs, the total sulphur being respectively 46·87 and 57·05 per cent. of the dried oxide. Attention is directed to a method of disposing of hydrogen sulphide in works using the continuous process of distillation (but where the output is too small for application of the Claus process), which has been in use for some years at a factory in the South Midland district, and is likely to receive further extension. The well-dried gases are passed to a specially constructed furnace for complete combustion, and the resultant gases, after cooling, pass to the bottom of a tower packed with limestone, and fed with water by a tumbling-box arrangement. Solution of calcium bisulphite is thus collected. This process has also been successfully applied to the acid gases arising from the manufacture of Venetian red. The following table shows the—

*Amount of Sulphate of Ammonia produced in the United Kingdom.*

| —— | 1897. | 1896. | 1895. |
|---|---|---|---|
| | Tons. | Tons. | Tons. |
| Gas works | 132.724 | 127,498 | 119,645 |
| Iron works | 17.779 | 16,511 | 14,588 |
| Shale works | 37,153 | 37.822 | 38,335 |
| Producer gas, coke, and carbonising works. | 10,624 | 9,078 | 7.083 |
| Total | 198,280 | 190,909 | 179,651 |

These figures relate to production merely, not to what is marketable. In the manufacture of metallurgical coke, it is stated that by use of efficient plant there may be obtained, stated as values at present prices from a unit of fuel treated, 68 per cent. value of coke, 5 per cent. of tar, 9 per cent. of ammoniacal liquor (in terms of sulphate), and 18 per cent. of light oils, chiefly benzol.

The annexed table shows the results of the examination of ammoniacal liquors from various sources, but all from gas works, one being a Scottish factory in which special attention is paid to the removal of carbonic acid from the gas, by use of a washer of special construction :—

| Liquor. | ° Tw. | Cyanide. | Ferro-cyanide. | Chloride calculated as HCl per cent. | Distribution of Sulphur. As Sulphate. S per cent. | As Sulpho-cyanide. S per cent. | As Thio-sulphate. S per cent. | As Sulphide. S per cent. | Total Sulphur. | Per Cent. Sulphur to Ammonia. |
|---|---|---|---|---|---|---|---|---|---|---|
| Gas works, 1..... | 4 | Considerable traces. | Absent | 0·006 | 4·14 | 2·26 | 0·55 | 93·05 | 0·6330 | 34·64 |
| „ 2..... | 4·75 | Absent | Small traces | 0·620 | 0·59 | 7·91 | 5·66 | 85·81 | 0·6955 | 26·49 |
| „ 3..... | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| „ 4..... | 5·25 | Considerable traces. | Absent | 0·685 | 0·43 | 14·40 | 6·24 | 78·93 | 0·7500 | 30·29 |
| „ 5..... | 3·75 | „ | „ | 0·12 | 4·63 | 16·47 | 10·83 | 68·67 | 0·1366 | 9·88 |

| Liquor. | Ammonia. Total per Cent. | Fixed per Cent. | Free. Per Cent. | A. / H.E. | Carbonic Acid. Per Cent. | B. / H.E. | Sulphuretted Hydrogen. Per Cent. | C. / H.E. | Total Acids. B. + C. / H.E. | Difference Acids. B.+C.−A. / H.E. | Difference Acids. B.+C.−A. ($NH^3$, H.E.= 100). | Per Cent. of Acids as $H_2S$. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Gas works, 1 | 1·827 | 0·110 | 1·717 | 101·00 | 1·860 | 84·50 | 0·6256 | 36·77 | 121·27 | +20·27 | +20·07 | 30·3 |
| „ 2 | 2·630 | 0·395 | 2·235 | 131·47 | 2·023 | 91·95 | 0·6340 | 37·30 | 129·25 | − 2·22 | − 1·69 | 28·8 |
| „ 3 | 1·84 | 0·28 | 1·56 | 91·76 | 1·390 | 63·18 | 0·3970 | 23·35 | 86·53 | − 5·23 | − 5·69 | 27·0 |
| „ 4 | 2·476 | 0·490 | 1·986 | 116·82 | 2·019 | 91·77 | 0·629 | 37·00 | 128·77 | 11·95 | +10·23 | 28·7 |
| „ 5 | 1·382 | 0·120 | 1·262 | 74·24 | 1·493 | 67·86 | 0·100 | 5·88 | 73·74 | − 0·50 | − 0·67 | 7·9 |

The total amount of sulphate of ammonia used in the United Kingdom for agricultural purposes in 1897 was about 70,000 tons, an increase of about 2,000 tons over the preceding year. France took from us 10,000 tons more in the same period, and Germany and Belgium together 12,000 tons more. Continental production of the salt continues to augment, except in France, where the production is diminishing. Experiments have been made on Lord Rosebery's estate at Dalmeny on the comparative value of the nitrogen in sulphate of ammonia and nitrate of soda, which show an advantage for the former in respect to hay, potatoes, and turnips, both as to the weight and quality of the crops obtained. In oats the difference was not marked.

The following is an account of the—

*Tons of Mineral Phosphates, Guano, and Nitrate imported.*

| — | 1894. | 1895. | 1896. | 1897. |
|---|---|---|---|---|
| Guano.... .............. | 28,207 | 19,849 | 20,211 | 16,164 |
| Mineral phosphates..... | 379,000 | 359,000 | 291,000 | 325,000 |
| Nitrate of soda ......... | 125,000 | 122,000 | 106,000 | 105,000 |

The amount of nitrate of soda imported into Europe in 1897 was 1,100,000 tons. "This figure," it is remarked, "is of interest in connection with the use of sulphate of ammonia, and our exports of this to the Continent, as affecting recovery of ammonia."

The process of tar distillation has been watched from beginning to finish, and the results are tabulated. Three fatal accidents occurred in tar works during the year.

Dr. Hoepfner's electrolytic method for obtaining zinc from its ores is already in operation in this country. It is stated on Mr. J. W. Swan's authority that 1 E.H.P. hour produces 1 lb. of zinc, as compared with 0·5 in the case of copper, and that the displacement by electrolytical methods of the older smelting processes of this metal in the future is probable.

The report contains 129 pages, of which 9 pages are devoted to Scotland.—E. S.

*Calcium Chloride Solutions, Electrolysis of.* [*Preparation of Chlorates.*] H. Bischoff and F. Foerster. Zeits. f. Elektrochem. 1898, **4**, 464—570.

In his investigation of the electrolytic formation of chlorates, Oettel (this Journal, 1895, 492) refers to a difference in the behaviour of potassium chloride and calcium chloride solutions. In the former case the current efficiency rarely exceeded 60 per cent., whilst in the case of the latter salt it rose to about 87 per cent. Since no further details have been published, the authors decided to re-investigate the subject.

They first repeated Oettel's experiment, and found that, on electrolysing a solution containing 20 per cent. KCl and 1·5 per cent. KOH, the current efficiency was 52·2 per cent.; the current densities at the anode and cathode being respectively 9·4 and 13 ampères per sq. dem., whilst the E.M.F. employed was 4·9 volts. On now substituting for the potassium chloride an equivalent solution of calcium chloride (14·86 per cent. $CaCl_2$) the current efficiency increased to 81·1 per cent., but the E.M.F. necessary was 6·7 volts. The temperature in both of these experiments was 0° C. Further experiments were then made to determine the current density and concentration most favourable for the production of a good yield of calcium chlorate, and it was found that the solution should contain at least 10 per cent. of $CaCl_2$, but preferably much more. The current density should be about 10 ampères per sq. dem. at the anode, and at least twice as much at the cathode.

Neutral and alkaline solutions of barium chloride were also electrolysed, but the current efficiency was, in general, lower than in the case of potassium chloride. The authors consider that the exceptional behaviour of calcium chloride is to be attributed to the fact that during electrolysis, an insoluble layer or diaphragm of calcium hydrate is formed on the cathode and that this then prevents the reduction of the hypochlorite which is first formed. The bulk of the calcium hydrate on the cathode is converted into hypochlorite or chlorate by the chlorine liberated at the anode, but a considerable fraction invariably peels off and settles at the bottom of the cell. The resistance of the film of calcium hydrate on the cathode necessitates the employment of a higher E.M.F. for the electrolysis. In the above experiment, made at 0° C., the E.M.F. was 6·7 volts, whilst in the case of the corresponding potassium chloride solution 4·7 volts were sufficient.

If the yield of chlorate in both cases be referred to the actual electrical energy, and not merely to the current used, then the employment of calcium chloride for the electrolytic manufacture of chlorates no longer appears in such a favourable light. This difficulty may, however, be overcome by conducting the electrolysis at about 50° C., when the active diaphragm of calcium hydrate which forms on the cathode becomes so thin that its resistance no longer requires the application of such a high E.M.F. Thus, on repeating the experiments already described at 50° C. instead of at 0° C., the following results were obtained:—(1) KCl solution: E.M.F. = 4·4 volts; mean current efficiency, 55·1 per cent. (2) CaCl$_2$ solution: E.M.F. = 4·55 volts; mean efficiency, 76·6 per cent.

Only a slight increase of voltage was necessary for the electrolysis of the calcium chloride solution, but, on the other hand, the yield of chlorate had somewhat diminished, owing to the increase in the amount of reduction of hypochlorite which took place.

By increasing the concentration of the solution and the current density at the cathode in accordance with what had previously been found, the results obtained were as follows:—

The solution electrolysed, contained 222 grms. CaCl$_2$ per litre, whilst the current density was 7—8 ampères per sq. dcm. at the anode, and 18—20 ampères per sq. dcm. at the cathode, which consisted of two sheets of nickel foil, one on either side of the platinum anode. The E.M.F. necessary varied from 5·2—5·9 volts, and the mean current efficiency was 87·7 per cent.

These results show that the industrial manufacture of calcium chlorate by electro-chemical means is worthy of attention.—J. S.

*Ammonia-Soda Works, Effluent from.* G. Lunge. Zeits. f. angew. Chem. 1898, 488—489.

A reply to Jurisch (this Journal, 1898, 349), who criticises a statement of the author in his "Soda Industry" with regard to the determination of ammonia in the effluent from ammonia-soda factories, pointing out that although in several commercial analyses it is not possible to read with accuracy to the third or even second decimal place, this is by no means true of the small quantities of ammonia which are here in question (0·004 grm. per litre), amounting, however, in the aggregate to a considerable quantity. He points out that in potable waters, account is taken of differences of 0·000001 per cent., whilst, on the other hand, in cases in which large quantities of ammonia are concerned (*e.g.*, manures), differences of ±0·01 per cent. cannot be accurately determined.—T. A. L.

*Lead, Haloid Salts of.* V. Thomas. Comptes Rend. **126**, [19], 1349—1352.

Boiling hydrochloric acid, acting on lead iodide, gives a chloro-iodide, PbI.Cl, mixed with chloride or iodide of lead according to the proportions of iodide and hydrochloric acid used. By acting on lead chloride with an alkaline iodide, or on lead iodide with an alkaline chloride, there is always obtained the same double salt, mixed with an excess of lead chloride or iodide as the case may be. The author has repeated the experiments of Miss Field (Chem. News, 1893, **67**, 157), and finds that whether lead iodide and ammonium chloride or lead chloride and potassium iodide be used in the proportions mentioned by her, the same chloro-iodide is always obtained, any excess of lead chloride or iodide remaining simply mixed with the double salt. The double salt is distinguished from mixtures of chloride and iodide by the action of nitrogen peroxide, which is without action on the chloride, but decomposes the iodide with formation of lead oxide. Its action on a mixture of the two salts consequently gives rise to a mixture of lead chloride and oxide, but it decomposes the chloro-iodide with production of an oxychloride, $Pb_2OCl_2$.—J. T. D.

*Tartaric Acid, Analysis of Raw Material containing.* Chemische Fabrik vormals Goldenberg, Geromont, and Co. Zeits. Anal. Chem. 1898, **37**, [6], 382.

*See under* XXIII., *page* 698.

*Perchlorate* [*or Total Chlorine*] *in Sodium Nitrate* (*Chili Saltpetre*), *Estimation of.* O. Foerster. Chem. Zeit. 1898, **22**, [36], 357.

*See under* XXIII., *page* 694.

## PATENTS.

*Electricity Supply, The Combined Generation and Application of, and the Manufacture of Salt.* H. Tee, Liverpool. Eng. Pat. 12,665, May 22, 1897.

*See under* XI. A., *page* 672.

*Carbonic Acid Gas, Impts. in and Apparatus for the Manufacture or Production of.* H. Steinem, Clapton, and K. S. Murray, London. Eng. Pat. 13,825, June 4, 1897.

The waste gases from the furnace in which the carbonated lyes are boiled to recover carbonic acid, are passed in succession up two vertical washers, packed with limestone or marble chips, hot water being streamed down the first washer, and cold water down the second, sulphuretted hydrogen and other impurities being thus removed, and the gases cooled. From the cold-water washer the gases are drawn by separate pipes to a series of absorbing coke-packed towers, each tower having its own exhauster, and all being supplied with potassium carbonate solution from an elevated tank, kept full by pumping from an underground tank, which latter receives from the boilers the carbonate solution after expulsion of the absorbed carbonic acid. The lower tank is divided into a large and a small compartment by a partition reaching nearly to the top. The large compartment supplies lye to the upper tank, as stated, receiving the spent liquor from the boilers through coolers. The small compartment receives the carbonated lye from the absorbing towers, and from this compartment the lye is pumped through a preliminary heater to feed the boilers, the heater for the ingoing being also the cooler for the outgoing liquor. Any excess of carbonated lye in the smaller compartment overflows into the larger compartment, whence the absorbing towers are supplied, thereby enriching the lye. Two or more boilers are used, worked in succession, the lye being discharged from one into the other so as to get a proper circulation. Both boilers communicate with a main pipe leading to the carbonic acid gas reservoir. —E. S.

*Phosphates, Improved Process relating to the Treatment and Production of.* E. Masson-Polet, Hannut, Belgium. Eng. Pat. 15,976, July 5, 1897.

*See under* X., *page* 670.

## VIII.—GLASS, POTTERY, ENAMELS.

*Glass, New Applications of.* [*Bearings, and for Electrical Purposes.*] P. Marcillac. L'Éclairage Électrique, 1897, **11**, 585; through Proc. Inst. Civil Eng.

The author gives an account of experiments carried out in America with regard to the use of glass in place of metal for bearings. The most suitable kinds were found to be bottle and common window glass; and the bearings, which were obtained by fusion, were made in two pieces. The bearings should be mounted on elastic supports, and the revolving parts should be well balanced and the shafts accurately centred. It is stated that glass bearings require less lubrication, have less friction, and in many cases are advantageous, though in some instances they give less security than others. With regard to the friction of glass on glass, a trial was made by wrapping a sheet of glass while hot round the end of a shaft, and allowing this to run in a glass, bearing without lubrication: some trace of wear was found, but none when oil was supplied. The best degree of tightness of the bearings was found to be 25 per cent. less than in the case of metal ones. At Marseilles, glass tubes are being used as underground conduits for telegraph wires, and for carrying electric lighting wires in factories, theatres, &c. The tubes are 0·01 cm. (0·004 in.) thick, and from 40 mm. (about 1·5 in.) to 60 mm. (2·375 in.) bore are in lengths of from 1 m. (3·28 ft.) to 2 m. (6·56 ft.).

They can be produced at a price sufficiently low to enable them to be used in cases where safety and perfect insulation admit of a slight increase in cost, and they can take the place of wrought-iron, lead, zinc, and even cast-iron pipes. It is stated that with well-made joints, which are of spigot and socket kind, the tubes are completely air tight.—A. S.

*Glass, Dry Etching on.* Sprechsaal, **31**, [23], 709 (this Journal, 1898, 351).

The process may be expedited, for use on a large manufacturing scale, by impregnating the atmosphere of the work-room with moisture up to 80 per cent. of the point of saturation, or by laying the glass articles, coated with the etching material, face downwards at a distance of about 4 ins. over water maintained at a temperature of 25°—30° C. The latter method is preferable, and will reduce the time required for etching to about 10 minutes.

The printing medium is preferably compounded by mixing 70 parts of melted resin with 30 parts of purified olive oil or other fatty oil, colouring the mixture with a dark blue aniline dyestuff, and reducing its consistency with oil as occasion requires.—C. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Hydraulic Lime, Natural and Artificial; Theory of the Setting of.* K. Zulkowski. Chem. Ind.; Thonind. Zeit. **22**, [26], 285—291; [27], 316—320; [30], 363—365.

The investigations conducted by the author were divided into several sections, certain of which are given in this Journal, 1898, 353.

*The Examination of Blast-Furnace Slag and Slag-Meal.*—Here the object was to ascertain the cause of the different behaviour of granulated and of slowly cooled slag on exposure to the air. Dilute (1 : 5) acetic acid was employed as a solvent, and the results showed the slowly cooled slag to be less susceptible to the influence of this acid than when in the granulated form, the latter being more uniform in composition; whilst the assumption that the former—like de-vitrified glass—has undergone an alteration in composition and contains a large proportion of a tertiary compound (hydraulite) is confirmed. In explanation of the hydraulic property of granulated slag, the author believes that a basic calcium-aluminium di-silicate is present, which retains its composition when rapidly cooled, and is then capable of hydration, but breaks up when the cooling is allowed to proceed slowly.

*Behaviour of Granulated Slag towards Water and Alkaline Solutions.*—Experiments made with various samples of slag formed into balls and immersed in water, lime-water, and solutions of caustic alkali showed that although no absorption of the reagent occurs, its presence facilitates the absorption of water of hydration, and thus assists the hardening of the cement: presumably by attacking the inert materials forming the matrix and so admitting the water into contact with the embedded molecules of hydraulite. It was also found that hardening was facilitated by heat in the absence of lime, &c. in the steeping water.

*Composition of Portland Cement.*—Attempts were made to extract the free lime from cement and slag by means of an alcoholic solution of magnesium nitrate, but it was found that in presence of water—necessary for complete extraction—the combined lime was also drawn into the reaction when prolonged. Dilute hydrochloric acid was then resorted to, and from 10 to 16 per cent. of lime was extracted from a Portland cement, from Podol, containing 56·9 per cent. of CaO, the residue in each case being made up into balls and placed in lime-water to set hard. This effect was attained in about a month, though in water alone the samples deprived of more than 12 per cent. of lime refused to harden; nevertheless, analyses showed that there had been no re-absorption of lime from the lime-water. The proportion of lime removable by extraction, without injury to the setting power, varies with the kind of cement, a sample from Züllchow (Stettin) parting with up to 18 per cent.

The function of the free lime in Portland cement is merely to keep the absorbed water alkaline, and so facilitate hardening. As this lime only enters into combination with carbon dioxide in course of time, the regeneration of weathered cement appears feasible, and was effected by the author—thus confirming the researches of Michaëlis and Schott—though, apparently owing to the occlusion of carbon dioxide by the hydrosilicate, the original degree of hardness could not be regained, a higher kilning temperature being required to break up the combination.

*The hardening of hydraulic cements* occurs through the alteration, in shape and volume, of the individual granules during the absorption of water—changes noticed under the microscope and found to develop more quickly when the added water contained lime or free alkali, the originally crystalline mass becoming swollen, flocculent, and cohesive. This property is weakened when the cement has been deprived of part of its lime by hydrochloric acid, as already described; this treatment causing partial hydration, and reducing the hygroscopicity of the mass, and also—as shown by dynamimetric tests—lowering the tensile strength of the finished cement.

The additional conclusions drawn by the author, from his researches, are: that the free lime in Portland cement cannot be estimated by direct methods (extraction); and that, like slag cement, the natural cement is a mixture of free lime and a hydraulite analogous to that present in blast-furnace slag, the only difference being that slag cement requires an extra addition of free lime in the form of hydrate.—C. S.

*Hydraulic Lime, Natural and Artificial; Theory of the Hardening of.* K. Zulkowski and H. Löster. Chem. Ind. **21**, [11], 225—234.

The authors have experimented upon the production of hydraulic limes by exposing various mixtures of lime and silica, and lime, silica, and alumina to high temperatures. A mixture of silica and lime in the proportion of one molecule of the former to two of the latter failed, even when subjected to a white heat, to produce a compound of hydraulic properties. On the addition, however, of 5 per cent. of boric acid, complete fusion took place, resulting in the formation of a basic metasilicate of decided hydraulic properties. A mixture corresponding to the formula $2SiO_2 + Al_2O_3 + 4CaO$ behaved in a similar way. A mixture containing one molecule of boric acid to four molecules of lime, though not fusing to a completely homogeneous mass, yielded a hydraulic body on cooling with water. A mixture of the formula $B_2O_3 + Al_2O_3 + 4CaO$ likewise furnished a hydraulic body, notwithstanding that the fusion also in this case was incomplete. In all cases where, through imperfect fusion, the product was possessed of only weak hydraulic power, an increase in temperature produced more favourable results. Experiments were also made with phosphoric acid, but these were not carried far enough to furnish positive proof as to its capability of producing a hydraulic body.—H. H. B. S.

*Stoneware Pipes, The Jointing of. [Employment of Asphalt Joints.]* A. Unna. Gesundheits-Ing. 1897, 260; through Proc. Inst. Civil Eng., 1898.

It is stated that asphalt joints for glazed stoneware pipes are now used in all the chief cities of Germany. In making the joint, the heated asphalt is poured into the socket after the insertion of the spigot end of the pipe, a ring of india-rubber or of canvas filled with cork shavings being fixed round the spigot to confine the asphalt within the socket. As regards cost, there is little difference between cement and asphalt joints for small sizes of pipes. On testing the asphalt joints under hydraulic pressure, the pipes were found to give way before the joints showed any signs of leaking. In conclusion, asphalt joints are stated to possess advantages over other kinds of joints with regard to their impermeability, elasticity, and resistance to the action both of acids and alkalis; to the fact that they can be made in severe weather; that they require no time for induration; and that pipes thus jointed can be taken apart for repairs without injury by slightly heating them.—A. S.

*Slate Marble.* Eng. and Mining J., July 2, 1898, 5.

According to the *Bautechnische Zeitschrift*, Belgium exports a sort of black marble which is nothing else than prepared slate. The slate suitable for this purpose is first polished very smooth with a sandstone, so that no visible impression of the chisel remains on it; this is the rough polish. After this, polish finely with artificial pumice stone, and finally finish with extremely light natural pumice stone. The polished surface now presents a velvet-like, soft appearance. The surface is then dried and heated thoroughly, whereupon the finely polished surface is impregnated with a heated mixture of oil and fine lampblack. This is allowed to remain for 12 hours. According to whether the slate used is more or less grey, the process is repeated until it loses its grey appearance. Now polish thoroughly with emery, which is taken on a linen rag, and finally finish polishing with tin ashes, to which is added some lampblack. After the polishing is finished, spread wax, dissolved in turpentine, to which some lampblack is also added, on the polished plate warmed again. Allow it to remain some time, and then rub off vigorously with a clean linen rag. The slate thus treated now has a deep black appearance and looks like black marble. The polish is just as durable as the latter. The polished surfaces can be etched, engraved, gilded, and silvered, just the same as genuine marble.

## PATENTS.

*Composition, Mixture, or Cement, An Improved, for Lining or Re-lining and Jointing, Mending or Renewing Gas Retorts, Furnaces, and the like.* A. W. Crowther, Huddersfield. Eng. Pat. 13,000, May 27, 1897.

The composition consists of a mixture of fireclay or similar material, gannister or silica, and soda, the whole being ground, mixed together, and made into a paste with water. The mixture is applied to the parts requiring lining, mending, or jointing, with which it unites. For some classes of work, barilla is substituted for soda.—H. H. B. S.

*Blast-Furnace Slag, Process of Treating, for Use in Cement, Mortar, or the like.* A. D. Elbers, Hoboken, State of New Jersey, U.S.A. Eng. Pat. 14,299, June 12, 1897. (Under Internat. Convention.)

"Sulphurous blast-furnace slag" in a powdered state, after partially desulphurising, is impregnated with "nitrosyl," which, on the one hand, effects the partial decomposition of the sulphides impairing cohesion; and, on the other, prevents the formation of ferric hydrate, which interferes with the process of hydraulic cementation. The strength of the weak nitric acid solution to be employed is determined by experiment, but usually about one-fourth part by weight of a solution containing 1¾ per cent. of $N_2O_3$, and of a density of slightly over 2° Tw., answers the purpose.—H. H. B. S.

*Artificial Blocks or Plates for Building and other Purposes, Impts. in and relating to the Manufacture of.* H. H. Lake, Middlesex. From J. Seidler and A. J. C. Graf, both of Hamburg. Eng. Pat. 16,664, July 13, 1897.

Calcined dolomite is treated with a solution of hydrochloric acid; the pulp so obtained is mixed with an organic porous or fibrous substance, the mixture formed into blocks, slabs, &c., and then dried.—H. H. B. S.

*Fireproof Building Stones, A Process for the Manufacture of, and for the Production of Cement from the same.* P. Kleber, Malstatt-Burbach, Germany. Eng. Pat. 1769, Jan. 22, 1898.

Caustic lime in lumps is mixed, in the proportion of 1—12 to 1—15, with quartz or sandstone, and the whole ground to the size of grains of sand. The mixture is sprinkled with dilute hydrochloric acid and the mass mixed as intimately as possible, which results in the formation of chlorides of aluminium, iron, calcium, magnesium, &c., whilst gelatinous silica is separated. The plastic mass is pressed into moulds and subjected to the vapour of hydrochloric acid under a pressure of 7 atmospheres, and at a temperature of 160° to 170° C., by which it is hardened. It is further claimed that the finished product when ground may be used as a substitute for Portland cement.—H. H. B. S.

*Waterproof Material, Impts. in the Manufacture of; more particularly intended for Roofing and Insulating Purposes.* L. Hatschek, Vöcklabruck, Austria. Eng. Pat. 7678, March 30, 1898.

Fibrous material, more particularly asbestos, is intimately mixed with pulverised asphalt and the mass formed into sheets and heated until a partial melting of the asphalt takes place, thus spreading it uniformly through the fibrous material.—H. H. B. S.

# X.—METALLURGY.

*Combination Mill [Gold], in the United States.* W. McDermott. Inst. of Mining and Metallurgy, 1898.

At the Eureka Hill Mining Company of Utah, the pulp from the Fruevanner, after settling, is put in charges of 3,000 lb. into amalgamating pans, together with 5 per cent. of salt, 3 lb. of copper sulphate, 2 lb. of sulphuric acid, 2 lb. of iron borings, and 2 oz. of concentrated lye. The temperature is raised by steam in two hours to about 180° F., 200 lb. of mercury are added, and the whole is run for 6 hours. The original ore contains 20 oz. of silver, 2 dwt. of gold, 4 per cent. of lead, and a little copper, half the value of which is separated by the Fruevanners. The average extraction is 80 per cent., which is considered satisfactory for the raw treatment of a complex ore.—A. W.

*Soluble Gold. Zsigmondy.* Zeits. f. Elektrochem. 1898, **4**, [23], 546—547.

Red solutions of gold, similar to those of silver prepared by Carey Lea, have been obtained by adding formaldehyde to very dilute solutions of gold chloride made slightly alkaline. The solutions contain 0·005 per cent. of gold, and may be concentrated by dialysis until they contain 0·1 per cent. of gold. The addition of small quantities of salt or dilute acids changes the colour of the solution to blue, whilst an excess precipitates the gold entirely.—J. S.

*Telluride Ore [Gold] Experiments on Roasting.* E. D. Skewes. Eng. and Mining J. 1898, **65**, [17], 488.

Experiments in respect to the volatilisation of gold in telluride ores by roasting, were carried out in Western Australia, when it was found that by wet-crushing and roasting, the loss of gold and silver was much heavier than in the case of dry-crushing and roasting. In the former case the losses were as much as about 80 per cent. of the gold, and from 66 to 90 per cent. of the silver, whereas in the dry-crushing experiments the gold lost was less than 1 per cent., and the silver from 21 to 31 per cent. The mine water used was salt, and it is probable that some of the volatilisation in the wet process was due to the fact that the ore, when dried, retained some of the salt, which, on roasting, converted the silver into chloride, and thus assisted the tellurium in volatilising the silver as well as the gold. It is now considered advisable to wet-crush the telluride ores, amalgamate to extract from 20 to 25 per cent. of the gold, and then roast for treatment with cyanide.
—A. W.

*Steels, Electrical Resistance of.* H. Le Chatelier. Comptes Rend. 1898, **126**, [24], 1709—1711.

The author has determined the influence of carbon, silicon, &c., on the resistance of steel by measurements made on steel bars of known composition, and which had been heated at 600° for several hours. In the case of steels containing carbon, an increase of 1 per cent. of carbon raises the specific resistance—*i.e.*, the resistance in microhms of 1 cm. cube—by 7. The steels are made up of lamellæ of pure iron or ferrite intermingled with the carbide $Fe_3C$ or *cementite*; the specific resistance of these components is calculated to be 9·5 and 45 respectively. An increase of 1 per cent. of silicon increases the specific resistance by 14. According to the author, silicon steels do not contain a definite silicide $FeSi_2$, but are either homogeneous mixtures analogous to the steels containing manganese or nickel, which are isomorphous with iron, or contain the silicon in a dissolved state. Manganese steels can exist in two allotropic forms (this Journal, 1894, 1198—1199), which differ in magnetic properties. The specific resistance of the non-magnetic variety increases by 5, and that of the magnetic variety, which is obtained by heating the former at 550° C. for two hours, by 3·5 for each increase of 1 per cent. of manganese. Nickel steels also occur in two forms (Comptes Rend. 1889, **110**, 283; and 1890, **111**, 445), which differ in magnetic properties and also in specific resistance. The latter is greatly affected not only by the content of nickel, but also by the allotropic state of the alloy. In steels which contain less than 5 per cent. of nickel, an increase of 1 per cent. raises the specific resistance by 3—7. Chromium, tungsten, and molybdenum cause only a very slight increase in the resistance of steels, and hence, according to the author, are probably not homogeneously mixed with the metal, but occur isolated as definite compounds.—E. W. W.

*Nickel Steel.* B. Simmersbach. Berg- und Hüttenm. Zeit. **57**, 113—115; Chem. Centr. 1898, **1**, [19], 1078.

In connection with the use of nickel steel for boiler plates, the author found that it is greatly superior to other kinds of steel with regard to corrosion by the action of salt water and of steam. Nickel steel is also suited for the manufacture of armour plates on account of its ductility, lightness (compared with ordinary steel), and high resistance to tearing or rending. An addition of 3 per cent. of nickel with 0·3—0·4 per cent. of carbon gives the best material. Experiments were carried out on a large scale which illustrate the extraordinary power of resistance of nickel-steel armour plates towards the effect of shot. At the present time nickel steel is not very extensively used on account of its high price, but it is used for parts of marine engines, especially for the hollow shafts used on the latest transatlantic steamers.—A. S.

*Iron-Nickel Alloys, Micro-structure of.* F. Osmond. Comptes Rend. **126**, [19], 1352—1354.

Up to 8 per cent. of nickel the structure of the alloy is like that of ordinary steel; from 12 to 25 per cent. of nickel the structure is fibrous, and the non-magnetic alloys containing above 25 per cent. of nickel have a purely crystalline structure. The author has observed that forging produces in all of these alloys a schistose structure, which is independent of, and superposes itself on, the structure characteristic of the class as indicated above.

—J. T. D.

*Alloy of Lead-Tin-Bismuth: Conditions of Equilibrium in the Ternary System.* G. Charpy. Comptes Rend. **126**, 1569—1573.

The three metals, lead, tin, and bismuth, mix in all proportions, forming at a suitable temperature a homogeneous liquid containing no definite compound. The system is a simple case of three constituents in which, in the terminology of Gibbs, there is a single liquid phase and three solid phases. To determine what compositions of the liquid phase can be in equilibrium with the solid phase—that is, with the pure metals in the solid state—the author has determined the temperature of commencement of solidification for 56 alloys, and for studying the results graphically has constructed a surface founded on the triangular diagram of Thurston. The projection of this surface is figured in the paper. The eutectic alloy of lead and bismuth contains 45 per cent. of Pb and 55 per cent. of Bi, and melts at 127° C. The eutectic alloy of lead and tin contains 37·5 per cent of Pb. and 62·5 per cent. of Sn, and melts at 182° C. The eutectic alloy of bismuth and tin contains 58·5 per cent. of Bi, and 41·5 per cent. of Sn, and melts at 133° C.; whilst the ternary eutectic alloy—that is, the alloy which fuses at the lowest temperature of all (96° C.)—contains 32 per cent. of Pb, 16 per cent. of Sn, and 52 per cent. of Bi.—T. A. L.

*Nickel Assay, New Caledonia.* H. W. Edwards. Eng. and Mining J., May 28, 1898.

*See under* XXIII., *page* 696.

*Lead in Ores, Analytical Determination of.* F. Moldenbauer. Chem. Zeit. 1898, **22**, [26], 256.

*See under* XXIII., *page* 695.

*Gold Assay, A Semi-Electrolytic Process of.* A. Bock. Chem. Zeit. 1898, **22**, [36], 358.

*See under* XXIII., *page* 695.

## PATENTS.

*Aluminium or Metallic Alloys, Impts. in the Manufacture of Various Articles from Alloys containing a Large Percentage of Aluminium, and Apparatus to be employed in connection therewith.* G. A. Dick, London. Eng. Pat. 8917, April 7, 1897.

The alloy is formed into tubes or hollow bars whilst in a heated or plastic state, by means of plungers, &c. working within a pressure chamber.—J. H. C.

*Aluminium, Solder and Flux for.* G. Hammond, San Francisco, and T. Flint, jun., San Juan, California, U.S.A. Eng. Pat. 2567, Feb. 1, 1898.

The solder consists of an alloy of tin, 100; silver, 20; zinc, 10; and aluminium, 0·1 to 6 parts; or in such proportion that there is more tin than silver, and more silver than zinc. Iodine, or the iodides of tin and mercury, serves as a flux, and may be used with or without the addition of a hydrocarbon such as vaselin.—W. G. M.

*Aluminium, A Process for Tinning and Soldering.* C. V. Burton, Chelsea. Eng. Pat. 3296, Feb. 9, 1898.

A "soldering alloy" is used consisting entirely or substantially of tin about 12 parts and aluminium about 1 part by weight.—J. H. C.

*Alloy of Iron and Hydrogen, A New and Useful, and Means for Producing the same.* G. W. Gesner, Brooklyn, U.S.A. Eng. Pat. 9103, April 13, 1897.

Jets of hydrogen are passed through molten metal contained in a stationary or movable converter, at first alone, then mixed with hydrocarbon vapour, and finally alone once more. Approximately, 1,000 cubic feet of hydrogen, at the temperature of 60° F. and Bar. 30 in., are sufficient for the alloying of 2,000 lb. of iron or steel. The temperatare of the molten metal should be about 2,800° F., and the depth 15 inches. In some cases the alloy is produced direct from the ore.

The alloy possesses unusual properties for resisting oxidation and corrosion; it can be worked like iron, and it has nearly the same strength; its specific gravity is somewhat less.—J. H. C.

*Steel or other Compounds of Iron and Carbon, A Process for Decarburetting.* R. H. Thwaite and H. V. Holden, London. Eng. Pat. 10,785, April 30, 1897.

The metal is subjected to the action of heated carbon dioxide at a high temperature; a bright-red heat is often suitable.—J. H. C.

*Steel, Impts. in the Manufacture of.* J. Parry and G. H. Llewellyn, Newport, Monmouth. Eng. Pat. 13,308, May 29, 1897.

A hearth is charged with alternate layers of crude iron and refining material, consisting of ore, cinder, lime, alkali, or other purifying agents. Heat is supplied sufficient to melt out the slag, and then increased so as to melt the refined metal. Sometimes two hearths are used alternately, in which case the lower heat for the first hearth is derived from the waste heat during the melting of the refined metal.

—J. H. C.

*Phosphates, Improved Process relating to the Treatment and Production of.* E. Masson-Polet, Hannut, Belgium. Eng. Pat. 15,976, July 5, 1897.

The claim is for "the process of transforming natural phosphated calcium carbonates or grey chalk into Thomas' slag or tetraphosphates, consisting in melting them in the presence of metals or ores so as to extract from them phosphoric acid, which passes into the metal, and treating phosphor metal thus obtained by the Thomas process in order to transfer the phosphoric acid to the slag and form tetraphosphates."—E. S.

*Steel, An Impt. in the Manufacture of.* A. Reynolds, Sheffield. Eng. Pat. 26,063, Nov. 9, 1897.

In the manufacture of crucible steel the metal is melted before placing it in the crucible for the usual treatment, instead of melting it in the crucible. By this means the capacity and the life of the crucible are increased.—A. W.

*Patina, A New or Improved Method of Coating Copper or Copper Alloys with.* A. J. Boult, London. From A. Lismann, Munich. Eng. Pat. 10,459, April 27, 1897.

The objects to be treated are made to form the anodes or cathodes of an electrolytic bath, consisting of water containing carbonates or compounds giving off carbonic acid. In some cases they are oxidised by exposure to the air before being placed in the bath.—J. H. C.

*Iron and Steel and other Metals, Process for Refining,* W. P. Thompson, London. From B. Talbot, Pencoyd. Pa., U.S.A. Eng. Pat. 3810, Feb. 15, 1898.

Impure material (*e.g.*, pig-iron) in the molten condition is charged into a bath of purified metal on the hearth of a tilting reverberatory furnace, and a portion of the charge is drawn off as soon as the whole is sufficiently pure. The time required for the refining of the added portion varies with the amount added, which usually ranges from 40 to 20 per cent., the original charge of course varying from 60 to 80 per cent. of the furnace capacity. Slag may or may not be left on the bath before the addition of the fresh metal: but if the slag be of suitable character it will assist in the refining operation. The process may be used for the production of steel or refined iron. The furnace, if not of the tilting type, must be provided with several tap-holes at different levels. The portion of the charge left on the hearth is used for the treatment of a fresh quantity of impure material.—W. G. M.

*Crucible Steel, Impts. in the Manufacture of, and its Alloys.* F. Schadeloock, Trieste, Austria. Eng. Pat. 8245, April 6, 1898.

To remove the oxidising materials, which affect the regulation of the exact amount of carbon required in the steel, the material is hand-picked, scraped, and when melted, treated with small quantities of aluminium, magnesium, or similar reducing metals, before the oxidising bodies can have time to act on the carbon.

The materials used are Siemens-Martin iron and grey pig, both free from sulphur and phosphorus, and they are put in the crucible in pieces of 1 to 5 c.c. in volume.—A. W.

*Roasting and Calcining of Minerals and the like Substances, Impts. in and relating to.* G. H. Blenkinsop, Swansea. Eng. Pat. 11,151, May 4, 1897.

The ore under treatment is charged into a closed revolving or stationary vessel or furnace provided with inlet and outlet holes and valves; heated air is then introduced under pressure, whereby the ore is oxidised, and a gas (or gases) is produced containing a high percentage of sulphurous or arsenious acid. The ores are sometimes subjected to a preliminary heating in the furnace, the inlet and outlet being being opened during this process.

For operating on magnetic iron, a temperature of 1,000° F. is necessary, and a pressure of 10 lb. to the square inch. For a monosulphide a temperature of 700° F. and a pressure of 3 lb. per inch is usually sufficient.—J. H. C.

*Alloys, Impts. in.* J. C. Bull, Erith. Eng. Pat. 12,325, May 18, 1897.

The alloys form a novel series of white metals with melting points varying from about 700° F. to about 2,000° F. They are composed of copper, lead, and nickel, with sometimes a little phosphorus added in the form of phosphor-copper. An alloy of copper, 70 parts, and nickel, 30 parts, is first prepared, to which the lead is added.

The most useful proportions of nickel are 5 to 15 per cent. of the whole; of lead, 30 to 70 per cent.; and of phosphorus, $\frac{1}{4}$ to $\frac{1}{2}$ per cent.—J. H. C.

*Furnaces, Impts. in, for Treating Ores containing Precious Metals and other Ores.* S. Alley, Glasgow. From J. Campbell, Sydney, N.S.W., and J. W. H. James, New York, U.S.A. Eng. Pat. 15,115, June 24, 1897.

This furnace consists of a closed horizontal cylinder, lined with firebrick, heated from the outside by gases and heated air. The ore or charge is fed in at one end through a closed hopper, and moved along by blades on a hollow rotating shaft passing through the end walls to the other end, whence it is discharged into a closed water-trough connected with a condensing apparatus. Water is run through the hollow shaft to keep it cool. Water-gas is forced through the cylinder at a slight pressure whilst the furnace is in action.—A. W.

*Furnaces, Impts. in and connected with, for Treating Ores containing Precious Metals and other Ores.* S. Alley, Glasgow. Eng. Pat. 15,203, June 25, 1897.

A circular furnace or oven, heated by gases in flues underneath, is supplied with hollow ploughs or stirrers attached by hollow arms to a revolving hollow shaft, through which a gas, such as water-gas, can be conveyed to the bed of the furnace on to the ore or charge.—A. W.

*Precious Metals, Impts. in Apparatus for Extracting, by Chlorination.* G. F. Smith, Sydney, N.S.W. Eng. Pat. 3807, Feb. 15, 1898.

The apparatus consists of a wooden barrel, the interior of which is divided into two chambers by a diaphragm of asbestos, protected by perforated wooden shields.

The larger or extracting chamber, into which the stuff under treatment is fed, is lined with carbon anode plates fixed by carbon bolts or screws; the smaller or depositing chamber is provided with metallic cathodes. The extracting chamber having been charged with powdered ore, salt, and water, the barrel is made to revolve whilst an electric current passes from the anodes to the cathodes. The gold present in the ore is deposited in the cathode chamber as a brown powder.—J. H. C.

*Treatment of Slimes and Tailings: Improved Apparatus for the Extraction of Gold and other Precious Metals therefrom.* H. H. Lake, London. From H. S. Denny, Johannesburg. Eng. Pat. 28,013, Nov. 27, 1897.

The slimes or tailings are delivered into a series of mixing or solution chambers, and thence into precipitating vats in an upward direction, beneath the surface of the water or liquids contained therein. The mixing chambers are sometimes closed in, compressed air being used to aid the solution. Automatic arrangements are provided for supplying and removing the slimes at a proper rate.—J. H. C.

*Complex Ores: Impts. in the Treatment of Ores containing Intimately Mixed Copper, Zinc, and Lead.* G. D. Bechi, Paris. Eng. Pat. 285, Jan. 5, 1898. (Under Internat. Convention, July 5, 1897.)

The crushed ores are subjected to a chlorinating roasting, followed by fractional precipitation of the copper and zinc as hydrated oxides. The evolved gases and vapours are condensed in suitable towers, and the roasted products are lixiviated with water to which is added the hot or acid waters from the condensing towers after precipitation of the sulphuric acid by means of chloride of calcium.
—J. H. C.

*Zinc, Improved Process for the Recovery of, from Refractory Zinc-bearing Ores.* J. Jones, Melbourne, Australia. Eng. Pat. 4296, Feb. 21, 1898.

Sulphate of zinc solution, obtained from the roasting and solution of the ores, and sulphate of ammonium, are placed in the anode compartment of an electrolytic cell divided by a porous diaphragm, and acid zinc sulphate, either alone or with a little sulphate of ammonium, is put in the cathode compartment. In the latter the zinc is deposited, and the ammonium also produced combines with the free acid, thus reducing the liability of the zinc to redissolve.—A. W.

*Gases from Molten Metal, Impts. in the Arrangement and Construction of Apparatus for Exhausting.* J. T. Wainwright, Chicago. Eng. Pat. 2293, Jan. 28. 1898.

The claim is for various combinations of an exhauster, a vacuum chamber, self-regulating trap inlets and outlets, and in some instances an internally cooled condenser.
—J. H. C.

*Smelting Plants, Impts. in.* O. Imray, London. From The Pennsylvania Salt Manufacturing Co., Philadelphia, U.S.A. Eng. Pat. 7008, March 22, 1898.

The object of the invention is to provide a continuous smelting plant for smelting ores and mattes, especially those containing copper with or without silver. A pair of blast-furnaces, with dust-catcher, elevator, and the usual appliances, are arranged in combination with two series of reverberatories, so that the matte from the blast-furnaces flows, by gravitation, to each of the reverberatories successively, for the different reducing and refining operations. A wheeled settler is provided for the blast-furnace, and a settler is placed between each pair of reverberatories, the settler being either on wheels to facilitate the transfer of the charge to the next furnace, or provided with a spout for the same purpose. The blast-furnace may have a porous bottom, through which the lead (in lead-smelting) oozes into a cavity with a sloping bottom, which discharges the metal into a receptacle in front. This forms the third claim; whilst the fourth, and last, is the combination with a smelting furnace of a slag pit having a water supply, and containing a receptacle, which may be removed by a crane, to catch the particles of slag that fall into it, granulated by the water.—W. G. M.

*Ore-Dust, or Perished Ore, Impts. in or relating to the Utilisation of.* P. Kleber, Mainz, and D. Timar, Berlin. Eng. Pat. 9610, April 26, 1898.

The ore-dust is mixed with lime, and, if necessary, with silicates, and is worked into a plastic mass with a hot solution of a flux; the mixture is pressed into blocks, which are then hardened in pressure chambers with high-pressure or superheated steam, and may afterwards be smelted in the blast-furnace. If refuse coke-dust be incorporated with the ore, no extra coke need be used in smelting. The process is specially applicable to iron ores.—W. G. M.

*Welding of Metals, A Compound for Facilitating the, and for Preventing the Deterioration of the same by Heat, or Restoring the Qualities of Injured.* T. Odlum, Richmond, Virginia, U.S.A. Eng. Pat. 1027, Jan. 13, 1898 (under Internat. Convention, Foreign Applic., June 23, 1897).

The "welding compound" is formed of an intimate mixture of sand, 32 parts; ground marble, 50 parts; borax, 12 parts; salt, 4 parts; and ground charcoal, 2 parts.
—J. H. C.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (A.)—ELECTRO-CHEMISTRY.

*Calcium Chloride Solutions, Electrolysis of.* [*Preparation of Chlorates.*] H. Bischoff and F. Foerster. Zeits. f. Elektrochem. 1898, 4, 464.

*See under* VII., *page* 666.

*Chlorine, Bromine, and Iodine, A Quantitative Electrolytic Separation of.* H. Specketer. Zeits. f. Elektrochem. 1898, 4, [23], 539.

*See under* XXIII., *page* 695.

### PATENTS.

*Electric Accumulators, Impts. in.* A. W. and P. de Kildnchevsky, London. Eng. Pat. 12,443, May 20, 1897.

The claims are: I. "In an electric accumulator the employment as an electrolyte of a solution containing the combination of cadmium and magnesium sulphates with another sulphate," zinc, iron, chromium, nickel, and cobalt, as specified. II. . . . "the employment as negative electrodes, in combination with the electrolyte above specified, of thin plates covered with an alloy of cadmium, magnesium, and zinc, or other metal specified, which is deposited thereon in charging." III. . . . "the employment as negative electrodes in combination with the said electrolyte, of grids of antimony-lead (or other metal unattacked by sulphuric acid) pasted with a compound of spongy metallic lead, zinc, and cadmium, in the pores of which the alloy above specified and hydrogen are deposited from the electrolyte."—J. C. R.

*Electricity Supply, The Combined Generation and Application of, and the Manufacture of Salt.* H. Tee, Liverpool. Eng. Pat. 12,665, May 22, 1897.

In the drawings annexed to this specification, modifications of pans or boilers are shown adapted to make salt and generate steam under a comparatively high pressure, utilising the steam so generated in, and driving, a steam engine, generating electricity by said engine, and employing such electricity for the production of heat in suitable apparatus, upon substances as described—carbides—electro-chemical or electro-metallurgical manufacture. Specifications 6565 and 6565A of 1895 are referred to (this Journal, 1896, 356).
—J. C. R.

*Batteries, Secondary, Plates for; Impts. in and relating to.* W. J. S. Barber-Starkey, Bridgnorth, Salop. Eng. Pat. 15,879, July 3, 1897.

The claim is: "The process of manufacturing storage battery electrodes, which consists in first immersing them in sulphuric acid, and passing an electric current through them, and then permitting them to dry with access of air, these operations being repeated alternately until the electrode has 'formed' to a suitable depth."—J. C. R.

*Batteries, Impts. in or connected with Electric.* [*Carbon, Positive.*] A. D. Seton, Aberdeen, and J. L. Dobell, Harlesden. Eng. Pat. 15,903, July 3, 1897.

The cell consists of an outer vessel, preferably of iron, and within this, supported in any suitable manner so as to be clear of the bottom, is a porous pot formed of magnesite as described in Eng. Pat. 4142, 1897, the whole being provided with a lid. Surrounding the porous pot is bismuth or a mixture of lead and bismuth, and within it a salt capable of parting with its oxygen to the surrounding molten metal, such as an arsenate, arsenite, vanadate, chromate, manganate, plumbate, zincate, or stannate, or a mixture of such salts.

The producer gas, granulated coke, or other carbonaceous matter from the oxidation of which the cell derives its energy, is fed into the molten metal continuously or intermittently, and means are provided for heating the cell and for admitting air to re-oxidise the salts in the porous pot.
—G. H. R.

*Batteries, Impts. in Primary Reservoir Electric.* [*Pressure Regulation.*] W. Rowbotham, London. Eng. Pat. 17,158, July 20, 1897.

This invention relates more particularly to the class of batteries described in Eng. Pat. 2378, 1897, and consists in constructing the acid-supply reservoir in two chambers, one of which is larger than the other, and contains an aqueous solution of sulphuric acid, whilst the smaller is filled with a similar solution, strongly charged with nitric acid or other suitable depolariser. The two chambers are connected by a valve which closes with pressure from the small chamber. Within the large chamber is a float with jointed or other pipe connection, leading by a pipe to the upper part of the acid compartments of the battery cell. The nitrous or other depolarising fumes pass up through this pipe, and bubble through the sulphuric acid by way of the float and a pipe within it, this arrangement of valve and pipes forming a constant-pressure trap to bring the depolarising gases in contact with the acid in the supply chamber.—G. H. R.

*Batteries, Primary, Impts. in or relating to, for Producing Electric Currents; also applicable for the Purification of Water.* [*Zinc-Carbon.*] A. J. Boult, London. From J. Cerpaux, Brussels, Belgium. Eng. Pat. 7193, March 24, 1898.

This cell, the electrolyte of which is rain or salt water, consists of a column of broken graphite, traversed by a copper rod with screw-threaded ends, having a copper cross or strip with depending sectioned arms mounted on it. This is enclosed in a fabric jacket which is surrounded by a sectioned zinc cylinder, held at a suitable distance from it by two covers of insulating material fixed on the ends of the cylinder and rod by means of nuts, the covers being provided on their inner sides with recesses to receive the column and zinc cylinder. Terminals are provided, and a device for suspending and immersing the apparatus in a water reservoir or stream, so that it can be utilised simultaneously for the generation of the electric current and for the purification of water.—G. H. R.

*Calcium Carbide, Impts. in Electric Furnaces for Manufacturing.* [*Hand-Feeding.*] W. P. Thompson, London. From C. L. Wilson, C. Muma, J. W. Unger, H. Schneckloth, A. P. Brosius, and J. C. Kuchel, Holstein, Iowa, U.S.A. Eng. Pat. 7575, March 29, 1898.

*See under* II., *page* 654.

## (*B.*)—ELECTRO-METALLURGY.

### PATENTS.

*Ores, Sulphide; Impts. in Treatment of.* J. Swinburne, London. Eng. Pat. 10,829, May 1, 1897.

Sulphide ores are heated and subjected to the action of chlorine, the temperature being so high that chloride of sulphur is not formed, the sulphur being recovered as such. The chlorine may suffice to chlorinate all, or only a portion, of the metals present, and a separation of different metals may be effected by fractional chlorination. The chlorides may then be electrolysed in the fused condition, or, if the metal to be deposited be relatively infusible, from aqueous solution; infusible metals may be deposited from fusion in a melted cathode of more fusible metal, and may thus form alloys; otherwise they would be deposited in a pulverulent condition. Thus a continuous process may be used: the sulphide is decomposed by chlorine, and the separated sulphur is recovered, the fused metallic chloride is run into a separate vessel, and is there electrolysed, the metal being run off from time to time, and the anode chlorine being employed to attack more ore. The precious metals may be recovered from the fused chloride by the introduction of more electro-positive metals; and impure metals may be refined from the more electro-positive metals present, by placing them in a fused bath of the chloride of the former. Thus, zinc blende may be treated with insufficient chlorine in order to separate it from iron, and the fused zinc chloride may then be treated electrolytically until too great an accumulation of impurities has collected in the electrolyte, when the zinc chloride may be distilled off. Lead sulphide may also be treated, the precious metals being recovered by treating the mixed chlorides afterwards with melted lead. There are 15 claims.
—W. G. M.

*Ores, Sulphide; Impts. in Treatment of.* J. Swinburne, London. Eng. Pat. 10,829A, May 1, 1897.

This is a modification of Eng. Pat. 10,829 of 1897 (see preceding abstract). Sulphide ores are run into a bath of fused chlorides and electrolysed, sulphur being evolved at and recovered from, the anode. Zinc sulphide, as free as possible from iron, may thus be treated in zinc chloride; gold and silver are then deposited by means of metallic zinc, and the residual charge is electrolysed for zinc and sulphur. Lead sulphide is run into fused lead or zinc chloride (or a mixture of the two); if sufficient zinc chloride be present to decompose the whole of the lead sulphide, no precious metals can remain locked up, but all may be recovered by treating the bath with metallic lead. The fused mass is then run into a second vessel, where it is brought in contact with the lead deposited in the succeeding process, so that any zinc and iron in that lead may be removed by exchange; the mixture of chlorides and sulphides is then electrolysed in another vessel for lead and sulphur, electrolysis being stopped before zinc and iron are deposited in any quantity; this lead is then refined as above described. The electrolyte is returned to the first vessel for the treatment of fresh ore; when too foul for this, it is treated for the recovery of the zinc chloride. Modifications of the process are described for the treatment of complex sulphides. The process is specially applicable to sulphide ores, which contain but little gangue. Complex ores may be treated by simple exchange in chloride solutions by subjecting them successively to the action of the different component metals, beginning with the more electro-negative, the most electro-positive metal being deposited electrolytically at the end. Metals of high melting point may be deposited in, and so alloyed with, cathodes of melted metals of greater fusibility, or they may be deposited in the pulverulent form. There are 10 claims.
—W. G. M.

*Metals, Electro-Deposition of; Impts. in certain Revolving Apparatus to be employed in connection with the.* [*Insulated Bearings.*] J. B. Brooks and J. Holt, Birmingham. Eng. Pat. 15,850, July 3, 1897.

The apparatus consists of a suspended perforated and freely rotating barrel, immersed or partly immersed in the electrolyte, and supported on insulated bearings. Within it is a helical conductor making contact with the work and with a conductor passing through the axle of the barrel, but insulated from it and the bearings, and continued into a flexible insulating jointed shaft, which may consist of an insulated wire or rod, part of which is formed into a coil the outer terminal end of which is directed upwardly at an angle out of the electrolyte, and is in wiping or rolling contact with a terminal of the return-circuit wire.—G. H. R.

*Electrical Resistances* [*Metal Films*], *A New and Improved Method of making High.* F. B. Fawcett, Bristol. Eng. Pat. 18,284, Aug. 6, 1897.

Instead of using covered wire or carbon for electrical resistances, it is proposed to use a very thin film of metal deposited *in vacuo* by electrical methods on glass or some other suitable non-conductor.

The preparation of this is thus described. "In order to enable the film to arrive at a constant electrical resistance, I boil it in heavy mineral oil under reduced pressure for a considerable time." To increase its effective resistance and adjustment to the required value, the film is cut by a hard-tempered steel style.—J. C. R.

*Production of Pliable and Elastic Bodies by Electrolysis, An Improved Process for the.* A. Krüger, Baden Baden, Germany. Eng. Pat. 26,102, Nov. 9, 1897.

"The invention consists in the metallic deposition upon a mould, model, or a framework forming part of the article to be produced, layers of the metal separated by a layer of composition such as plumbago; or in the deposition of layers of different metals. The invention further consists in the specific mechanism employed to produce homogeneity and solidity in the different layers of metal." This mechanism is shown, and is in the nature of "hard balls" held in a box the bottom of which is cut to meet the configuration of the article under treatment, upon the surface of which the balls are pressed by a spring. "The balls rotate, and, while polishing, also compress and render homogeneous and solid the deposited metal; and it is to be understood that a series of cylinders or other suitably shaped bodies may be used as a substitute for the balls."—J. C. R.

# XII.—FATS, OILS, AND SOAP.

*Oils and Fats, Fractional Saponification of.* R. Henriques. Zeits. f. angew. Chem. 1898, [15], 338—345.

The author has applied his method of cold saponification (this Journal, 1896, 299 and 476; 1897, 746) to the elucidation of the changes which occur during the saponification of fats and oils. Finding that on treating certain oils with one-third of the requisite amount of alcoholic potash and separating the unsaponified portion, the latter was readily soluble in alcohol, and that practically the whole of the glycerin had been liberated, and could be determined in the lye, systematic experiments were carried out with glycerides of known composition.

On treating pure tribenzoin with one-third of the alcoholic potash necessary for saponification, and extracting the unsaponified portion with ether, the latter was found to consist of the ethyl ester of benzoic acid, whilst, as in the preliminary experiments, nearly the whole of the glycerin had been set free. Even with so little as 10 per cent. of the requisite alkali, nine-tenths of the tribenzoin was converted into the ester.

Experiments were next made on a mixture of tristearin and tripalmitin, freed from oleins by recrystallisation from benzene. On saponifying this with one-third of the necessary alkali, the unsaponified portion was identified as the mixed ethyl esters of the fatty acids (m. pt. 21° to 23° C.; saponification value, 199·4), whilst 10·64 per cent. of glycerin was found in the lye (theory, 11·05). Similar results were obtained in all other experiments with saturated glycerides.

For the separation of ethyl esters from glycerides, the author has made use of the fact that the former are volatile in a current of steam at 250° to 270° C., whilst the glycerides are only very slowly decomposed into fatty acids, which can readily be separated in the distillate. His apparatus consists of an ordinary fractionating flask, the outlet tube of which is connected with a Liebig's condenser, and which is fitted with a cork having two holes. Through one of these passes a thermometer, whilst the other is for a tube which reaches nearly to the bottom of the flask, and through which dry steam is admitted. Between the steam-generating flask and this admission tube is a short copper tube inclined slightly upwards. This is heated with a small flame, and effectually prevents any particles of water from being mechanically carried forward. It is stated that with this apparatus the temperature is kept constant, and that 100 grms. of an ester can be distilled in from 20 to 30 minutes.

For the experiments on unsaturated glycerides, the oils chosen as types were (1) linseed, (2) olive, almond, and rape, (3) castor. In the case of linseed oil treated with one-third of the necessary alkali, the same results were observed as in the other experiments. With 25 per cent. of the alkali the reaction was equally complete, and even with 15 per cent. in a more concentrated solution. The ethyl esters separated by distillation, freed from fatty acids, and dried in a current of carbon dioxide, gave the following constants: — Saponification value, 180·8; mol. weight, 309·7; iodine value, 168·4; acid value of liberated fatty acids, 198·7; mol. weight, 281·8. As was also observed in other instances, the difference between the molecular weight of the esters and of the fatty acids closely approximated to 28. No difference in the iodine and saponification values of the esters obtained by fractional saponification with varying quantities of alkali (33½ to 5 per cent.) was observed, and from this the author concludes that fractional saponification is of no value as a means of differentiating the various fatty acids in linseed oil.

The constants of the residue left in the flask after distilling off the ethyl ester formed by saponification with only 10 per cent. of the necessary alkali, were:—Acid value, 1·1; saponification value, 185·8; iodine value, 174·4; acetyl value, 51·7. These figures corresponded to a mixture of di- and triglycerides; but whether the substance was really a mixture of impure diglycerides, and whether it was formed during the fractional saponification or by the action of the steam during the distillation of the ester, was left as an unsettled question.

By using methyl or amyl alcohol in place of ethyl alcohol in the alcoholic potash, the corresponding methyl and amyl esters are obtained. The latter are much less volatile than the ethyl esters, and the temperature in the distilling flask must be raised to from 270° to 280° C. The ethyl esters of linseed oil fatty acids are unstable and readily oxidisable, so that when left in contact with a little air in a closed vessel, the saponification value rises, the iodine value sinks, and a considerable acetyl value is produced. When left exposed to the atmosphere for several weeks, the solubility of the esters in petroleum spirit gradually decreases, until finally they become almost insoluble. The same result is obtained more rapidly by passing air through the ester heated on the water-bath. When the heating is carried out in an atmosphere of carbon dioxide, no change takes place. The following figures illustrate the changes which occur during the oxidations of the mixed ethyl esters of linseed oil fatty acids:—

| — | Acid Value. | Saponification Value. | Iodine Value. | Acetyl Value. |
|---|---|---|---|---|
| Two days old ... | 0 | 181·7 | 170·4 | 42·3 |
| About three weeks old. | 4·2 | 213·6 | .. | 85·1 |
| About six weeks old. | .. | 220·4 | 83·5 | 104·9 |
| Portion insoluble in petroleum spirit. | .. | 271·9 | 36·1 | .. |

The acetyl values were determined by Lewkowitsch's method (this Journal, 1897, 503). The author suggests that the increase in the saponification value is due to a disruption of the molecule, with the formation of acids of a lower molecular weight. So long as the oxidised esters are soluble in petroleum spirit they do not appear to react with phenyl hydrazine, but the insoluble substance gives a marked reaction, which is attributed to the presence of aldehydic or ketose groups. Unlike the linoxyn formed by linseed oil on drying, the oxidation products of the esters are not solid substances.

Almond oil behaved like linseed oil, the glycerin being almost entirely liberated by saponification with 15 per cent. of the requisite alkali, whilst the reaction was incomplete with 10 per cent. of alkali. The distilled ethyl esters gave the following constants:—Saponification value, 185·0; mol. weight, 302·7; iodine value, 91·1. From rape and olive oils similar esters were obtained by cold saponification, and in the case of the former by hot saponification. The esters were also oxidisable, but much more slowly and incompletely than those of linseed oil.

The glycerides of castor oil (saponification value, 182·9) were still more readily converted into esters than those of the other oils examined. The distilled ethyl esters had a saponification value of 174·9 and an iodine value of 84·1. They differed from the original castor oil in having a smaller viscosity, and in being completely, though not very readily, soluble in petroleum spirit. Their taste and physiological properties were found by Dr. Aronson to be the same as those of castor oil.

The author concludes from his experiments that during the saponification (either cold or hot) of all triglycerides with alcoholic alkali, the ethyl esters of the acids present are first formed as intermediate products, with the complete liberation of the glycerin. Further investigation is required to determine to what extent the rule applies to other esters. In the case of waxes an analogous behaviour is regarded as probable, but where lower alcohols are in question, the reaction may only occur within certain limits.—C. A. M.

### *Japanese Wood Oil.* [*Tung Oil.*] J. H. B. Jenkins.

Analyst, 1898, **23**, [266], 113—118.

The analysis of a sample of this oil gave results which were as a general rule considerably lower than those found by the author for a previous sample (this Journal, 1897, 193 and 195). The figures for the two samples were as follows:—

| — | Present Sample. | Earlier Sample. |
|---|---|---|
| Specific gravity at 60° F. ........ | 0·9343 | 0·9385 |
| Insoluble fatty acids............ | 95·6 per cent. | 96·4 per cent. |
| Unsaponifiable matter .......... | 0·63 „ | 0·41 „ |
| Glycerin ........................ | 10·6 „ | 10·4 „ |
| Free fatty matters (as oleic) .... | 1·83 „ | 3·84 „ |
| Solidifying point................. | Below − 17° C. | Below − 15° C. |
| Bromo-thermal rise (= $x$) ..... | 21·4° C. | 23·4° C. |
| Calc. iodine value ($x$ × 5·7)..... | 122·0 | 133·4 |
| „ „ ($x$ × 7·0)..... | 149·8 | 163·8 |
| Iodine value (Hübl) ............ | 149·7 | 165·7 |
| Saponification value............ | 192 | 194 |
| Specific temperature reaction ... | 298 | 330 |

The insoluble fatty acids gave—

| —— | Present Sample. | Earlier Sample. |
|---|---|---|
| Melting point | 30°—31° C. | 37° C. |
| Bromo-thermal rise (= $x$) | 21·0° C. | 22·1° C. |
| Calc. iodine value ($x \times 5·7$) | 119·7 | 126·0 |
| „ „ ($x \times 7·0$) | 147·0 | 154·7 |
| Iodine value (Hübl) | 144·1 | 150·1 |

With regard to these results the author mentions that the viscosity was determined in Redwood's instrument, and that in the Maumené test olive oil was used as the diluent and a correction applied. With Valenta's test the two samples became turbid at 44° and 47° C. respectively. In the elaidin test the latter sample darkened considerably, and, after standing for 24 hours, showed signs of partial solidification at the bottom of the tube. Unlike the earlier sample the later one did not reduce the silver nitrate in Becchi's test. Negative results, however, were given by both samples in Halphen's test for cotton oil (Rev. de Chim. Ind., Feb. 1898). On exposure to the atmosphere in a shallow dish heated on the water-bath, the later sample increased in weight during the first eight hours' exposure at the rate of 0·26 per cent. per hour, and in six hours the surface of the oil was covered with a skin. The earlier sample gained during four hours at the rate of 0·36 per cent. per hour, and was covered with a skin in two hours. A sample of linseed oil similarly treated, gained during eight hours at the rate of 0·10 per cent. per hour, and at the end of the period showed no sign of any skin on the surface. The refractive index of the later sample with sodium light was 1·503 at 19° C. On heating this oil out of contact with air (which was effected in a flask with a mercury valve) it remained liquid for two hours at 200° C. The temperature was then raised to 250° C., and maintained at that for two hours, during which time the oil thickened and some gas was evolved. When cold it was found to have been converted into a semi-solid product. The author suggests that the difference in the viscosity of the two samples may have been due to a similar partial polymerisation of one of them. It is stated that, by an error of calculation in the former paper (this Journal, 1897, 193), it was found that for the bromo-thermal test the factor 5·7, applicable to all other known fatty oils, could be used with the fatty acids of Japanese oil; but it is now established that the fatty acids, as well as the oil, require the factor 7·0. On treating Japanese wood-oil with a saturated solution of iodine in chloroform or other solvents, a jelly-like mass is produced, the stiffness of which is proportional to the amount of oil taken. No similar action was observed in the case of bromine, whether by itself or in solution.—C. A. M.

*Butter and Lard, Detection of Foreign Fats in.* C. B. Cochran. Rev. intern. falsific. **11**, 49; Chem. Centr. 1898, **1**, [19], 1069.

*See under* XXIII., *page* 697.

*Seal Oil in Cod-Liver Oil, Detection of.* E. Dowzard. Pharm. J. 1898, [1458], 532.

*See under* XXIII., *page* 696.

*Hübl's Method of Iodine Absorption, The Reactions in.* J. J. A. Wijs. Zeits. angew. Chem. 1898, [13], 291.

*See under* XXIII., *page* 698.

*Copper Alkali-Glycerin Compounds.* F. Bullnheimer. Ber. 1898, **31**, [10], 1453.

*See under* XXIII., *page* 700.

### PATENTS.

*Continuous Extraction, Process and Apparatus for.* E. Schliemann and E. von Boyen, Hamburg, Germany. Eng. Pat. 6431, March 16, 1898.

*See under* I., *page* 651.

*Wool Grease, Impts. in or relating to the Extraction of.* A. J. Boult, London. From L. E. Vial, Brussels. Eng. Pat. 19,014, Aug. 17, 1897.

The process claimed is one for extracting raw wool grease from the watery magma obtained on acidulating wool washings. For this purpose the magma is concentrated as much as possible in vats, after which it is mixed in a boiler with an equal quantity of wool grease and heated until all water is driven off. The anhydrous fatty residue is then extracted with a suitable volatile solvent to separate the wool grease from mineral matters. The latter, it is stated, form a good manure, containing about 4 per cent. of nitrogen.—W. P. S.

*Soap Powders, Impts. in the Manufacture of. [Prevention of Cohesion.]* W. P. Thompson, London. From E. Sieglin, Dusseldorf, Germany. Eng. Pat. 7932, April 2, 1898.

The improvements consist in subjecting the broken-up material as it comes from the breaking-up rollers, to a current of air. The latter is sufficiently strong to carry along with it dry soap powder, which forms a coat or crust over the broken particles and prevents cohesion of the same. —W. P. S.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (*A.*)—*PIGMENTS, PAINTS.*

### PATENT.

*Anti-Corrosive Composition or Paint, as a Preventative against Corrosion on Metals.* W. Peel, Liverpool. Eng. Pat. 110, Jan. 3, 1898.

The base of the composition claimed, consists of a mixture of 10 parts of "gum euphorbium" dissolved in fusel oil or other solvent, 1 part of gum dammar dissolved in "benzoline," and 5 parts of coal tar previously incorporated with 1 part of slaked lime. One part of graphite and one of zinc oxide, together with the necessary pigment, are now added, and the whole is thinned down to the required consistency with "benzine," spirits of turpentine, or other spirits. —W. P. S.

### (*B.*)—RESINS, VARNISHES.

*Oils and Varnishes, Absorption of Oxygen by Drying.* W. Lippert. Zeits. angew. Chem. 1898, 431.

Continuing his investigations on the oxygen-absorbing power of linseed oil, &c. (this Journal, 1898, 588), Lippert now describes the behaviour of varnishes (boiled oils) containing manganese resinate or lead oxide.

1. "*Manganese Resinate Varnishes.*" * — The first sample of drier was prepared by precipitating a weak alkaline solution of colophony with manganese acetate, extracting the deposit with ether, and distilling off the bulk of the solvent. As the resinate obstinately retained some ether, it was heated to 100° C. for several hours, which caused a certain amount of oxidation to occur, and finally yielded a yellow, resinous, friable mass, not entirely free from ether, and containing about 4 per cent. of metal. Varnishes were then made up by heating linseed oil to 150° C. for 15 minutes with such quantities of the drier that they ultimately contained (*a*) 0·02 per cent., (*b*) 0·06 per cent., (*c*) 0·15 per cent., and (*d*) 0·2 per cent. of manganese. The colour of the products deepened with increasing proportions of siccative, but no appreciable sediment was noticed, even on prolonged keeping. The drying tests were carried out during cool and cloudy April weather in a room maintained at 11° or 12°, the weight of the films being 0·11 to 0·13 grm. per 100 sq. cm. After 12 hours (*a*) had gained 2·1 per cent. of oxygen, (*b*) 4·89, (*c*) 6·6, (*d*) 6·46; after 36 hours the corresponding figures were 15·97, 15·48, 14·45, and 14·02 per cent. respectively; (*a*) continued to gain for 55 hours, the total increase being 17 per cent.;

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* By "metallic resinate varnishes" ("Metallresinatfirnisse") are to be understood boiled oils which, as siccatives, contain the lead or manganese salts of the resin acids of colophony; also, according to Bottler, the salts of the resin acids of the copals. It should be also noted that in Germany the term "firniss" includes "boiled oil."

(*h*) for 10 hours, absorbing 15·69 per cent. altogether. The results show that the higher the proportion of drier, the sooner is the maximum of absorption reached, and the sooner, also, the varnishes begin to lose weight again. Conversely, the smaller the proportion of drier, the larger is the amount of oxygen taken up. On testing the films with the finger, it was found that they remained tacky all the time they were gaining weight; and they could not be called dry until after they had commenced to lose. (The elasticity of the skin prevents linseed oil from ever feeling so hard as the dried film of a resin-varnish, so that the finger test is always subjective.) After four weeks (*d*) had softened again, and stuck to the palm of the hand, whereas the others did not exhibit this property. Although Lippert has obtained no direct evidence to bear out Weger's statement that above a certain percentage the drier becomes inoperative, yet his view is probably correct, for among the above varnishes (*c*), with 0·15 per cent. of Mn, was distinctly the best.

Other experiments were tried with a "pure" commercial sample of precipitated manganese resinate. The material was dissolved in ether, thrown down with alcohol, washed several times with the latter, and dried *in vacuo*. It then formed a chocolate-coloured powder containing 8·5 per cent. of metal. This was dissolved in linseed oil as before, the varnishes being arranged to have (*e*) 0·007 per cent., (*f*) 0·04 per cent., (*g*) 0·12 per cent., and (*h*) 0·33 per cent. of metal in them; but as a considerable amount of sediment was left behind in the basins, the two latter figures are only nominal. The drying tests took place in bright, warm July weather, the thickness of the films being 0·06 to 0·07 grm. per 100 sq. cm. In six hours (*e*) had gained 0·76 per cent., (*f*) 5·59 per cent., (*g*) 14·0 per cent., and (*h*) 11·16 per cent.; in 21 hours, 5·51, 11·83, and 12·1 per cent. respectively. (*e*) continued to increase in weight for 42 hours, the total gain being 16·16 per cent. (*g*) and (*h*) were also heated to 200° with the deposit, but complete solution could not even thus be effected. The varnishes, however, turned darker in colour, the solid matter changed to a flocculent substance which adhered to the walls of the vessel, whilst the power of absorbing oxygen remained practically unaltered. In general, the results were identical with those yielded by the specially prepared resinate, but no softening of (*h*) took place.

II. *Litharge Varnishes.* — These were made up by warming from 0·34 to 6·8 per cent. of litharge with linseed oil at a temperature of 220° or 230°, some undissolved matter remaining in every instance. As with the resinate varnishes, the larger the proportion of drier, the sooner the varnish attained its greatest weight, and the less oxygen was absorbed. In dull, warm July weather a sample containing 0·34 per cent. of PbO gained 8·5 per cent. in 23 hours, and 11·1 per cent. in 39 hours. Another, with 1·1 per cent. of PbO, gained 13·5 and 13·9 per cent. in the same times. Two others, with 2·5 per cent. and 6·8 per cent. respectively, gained 12·7 per cent. and 12·3 per cent. in weight during the first 23 hours. All the films were equally hard to the touch when they had reached their maximum weight, but after four weeks they softened again, this being particularly noticeable with the specimens containing much of the drier. In other respects the phenomena of drying were the same as before. Additions of litharge above 2·5 per cent. made no appreciable difference to the absorbing power, whilst, for practical work, the proportion of lead evidently ought to be kept lower than that permissible in the case of manganese salts. There is no advantage in employing red lead, as advised by Mulder; in fact, its use was abandoned by oil boilers a long while ago.

The reason why some varnishes soften again after becoming dry is not yet known: it may possibly depend on the presence of an excess of drier or of an unsuitable one. The previous experiments, however, distinctly prove that it is not evidence of adulteration with rosin or rosin oil. Indeed, it has long been recognised that the addition of a powerful siccative, even if dissolved in turpentine, will cause rapid drying, but may induce subsequent liquefaction. According to Mulder, the acid which results from the oxidation of lead linoleate, is a liquid turpentine-like body in the free state, whereas its lead salt is hard and brittle. It is conceivable, therefore, that if too much of this substance be present, prolonged exposure to air may lead to partial decomposition and the formation of the liquid acid within the film.

III. *Influence of Rosin and Rosin Oil.*—The foregoing observations show that the amount of oxygen taken up by a varnish is chiefly dependent upon the proportion of drier in it; but in all materials which are of industrial value, this absorption only ranges between 12 and 16 per cent. Products yielding lower figures are generally sophisticated; although even here, as the annexed table indicates, 12 per cent. may easily be reached:—

| Manganese Oxide Varnish Adulterated with | Maximum Increase in Weight. | | |
|---|---|---|---|
| | I. | II. | III. |
| | Per Cent. | Per Cent. | Per Cent. |
| 5 per cent. of rosin oil...... | 10·09 | 11·14 | 7·31 |
| 15 " " ...... | 8·63 | 10·19 | 11·35 |
| 25 " " ...... | 7·46 | 10·13 | 11·23 |
| 5 per cent. of colophony.... | 7·86 | 7·01 | 11·73 |
| 15 " " .... | 7·08 | 7·20 | 10·21 |
| 25 " " .... | 9·53 | 10·07 | 11·06 |

The problem of detecting adulteration in this manner is complicated by the increased viscosity of the varnishes (actual varnishes here) thus prepared; so that it is difficult to obtain thin and uniform films; still, if they are tested with the finger after they begin to lose weight, notable proportions of impurity will be shown by abnormal tackiness. It should be remarked, however, that the tests in the table were made with common rosin, which causes tackiness in very small amounts. When it has been previously neutralised with chalk, as has lately been recommended, at least 5 per cent. may be present without development of this property; wherefore, if there be grounds for suspecting that this neutralised rosin has been added, the varnish should be tested for calcium.

There is on the whole no better way of examining a varnish than by testing its drying power with the finger in the usual manner; nevertheless it is advisable to combine such a test with a determination of the amount of increase in weight on exposure. To find the exact time any sample requires to attain its maximum weight is troublesome, because the initial gain (as already explained) is followed by a loss. This may be best overcome by starting one test overnight when the action of light will be eliminated, weighing the film early in the morning; then preparing a second sample which can be weighed at intervals during the day. Gross adulteration will thus be detected by tackiness, or absorption of too little oxygen; lack of driers by diminished speed of drying; while the specimens may be preserved for a month to see if they become soft on standing. The whole process is, of course, greatly influenced by the weather and the prevailing temperature; and all experiments must be regarded as comparable only if they are carried out simultaneously.

—F. H. L.

## (C.)—INDIA-RUBBER, &c.

### *The Brazilian Process of "Smoking" Rubber.* Bd. of Trade J., July 1898, 24.

H.M. Consul at Pará states that the juice or milk of the *Hevea braziliensis* may be caused to coagulate by various means. By the addition of a solution of alum, sodium chloride, or various other salts, or by simply allowing the milk to stand exposed to the air for 24 hours, or by artificial evaporation, the watery portion may be separated from the solid residue known to commerce as india-rubber or caoutchouc.

To produce, however, a substance having that consistency and elasticity which is desirable, it is of the first importance that this coagulation should take place in such a way that as little water as possible remains imprisoned in the mass.

When the milk is allowed to coagulate by simply standing in contact with the air, a considerable amount of water remains in the mass; the rubber is flabby, inelastic, and deteriorates rapidly. It is classed together with scraps and *débris* as "sernamby," and fetches but a low price.

When alum is used, the result is better. In the province of Matto Grasso the milk is always treated with that reagent, and the rubber fetches but 3*d*. or 4*d*. per lb. less than the finest Pará rubber.

The best rubber, known as Pará fine, is produced, however, by rapidly evaporating the juice in the smoke produced by burning the nuts of the Urucury palm (*Attalea excelsa*). This is effected by throwing a quantity of the nuts on a small wood fire; a dome-shaped vessel is then inverted over this fire, and the smoke pours out through a hole at the top of the dome. A piece of wood, sometimes cut to the shape of a rough spoon or paddle, is dipped into the milk and then held in the smoke until the viscous liquid has dried, leaving a layer of rubber on the stick. The stick is then dipped again into the liquid, and the process repeated until layer by layer a large pellet of rubber is built up.

The exact action of the smoke has not yet been satisfactorily explained. It is probably due, however, to the acid (pyroligneous) present in the smoke. All the apparatus employed may be seen in the Kew Museum. But there is no doubt that the milk thus treated yields a very elastic, and, at the same time, firm rubber that is free from all tendency to flabbiness, and does not lose weight when stored. The smoke of an ordinary wood fire is found to produce what is known as "wet rubber," that loses from 5 per cent. to 10 per cent. in weight whilst in the hold on the way to Europe. A considerable amount of rubber is, however, probably prepared by this means in localities where the Urucury palm is scarce.

The method of smoking, though very primitive, enables a man to prepare as much as 10 to 15 kilos. of rubber in one hour.

The good effects produced by the smoke of the Urucury nuts are probably due, in part, to the fact that the smoke is a dry smoke; but it seems probable that there is also some chemical reagent at work, the nature of which has not yet been ascertained.

A machine, designed on the principles of a centrifugal cream separator, to separate the watery portion of the milk has been tried, but without good results at present.

A feasible method would be to dissolve the rubber in some liquid that will not mix with water. Carbon bisulphide, for instance, if it could be sufficiently cheaply produced, might revolutionise the present somewhat primitive system.

*Waterproof Cloth, On the Vulcanisation of.* C. Otto Weber. Dingler's polyt. J. **308**, [2], 42—48.

The vulcanisation of waterproof cloth with sulphur chloride offers a number of advantages which effectually counterbalance the undeniable disadvantages of this method, and will, in the opinion of the author, probably secure it a permanent place in the manufacture of waterproof cloth. But to obtain good results with this vulcanising agent, the great reactive energy of sulphur chloride with substances other than india-rubber should always be remembered. To begin with, this reactive energy of sulphur chloride necessitates its application to the india-rubber in a state of solution. The solvent generally employed for this purpose is carbon bisulphide, but it is questionable whether this very obnoxious solvent is really the best for the purpose. Any solvent to be used in conjunction with sulphur chloride for the vulcanisation of india-rubber must conform to the following conditions:—(1.) The solvent must be indifferent to sulphur chloride. (2.) The solvent must be a homogeneous compound. (3.) The boiling-point of the solvent should not be less than 70° C. nor exceed 100° C. (4.) The solvent for sulphur chloride should also be a solvent for india-rubber, or at least cause it to swell. Of these conditions the first is a matter of course. The second becomes at once obvious if we remember that the cold vulcanising process is a continuous one, exposing the solution to continuous evaporation when in use, so that a solvent consisting of a mixture of compounds of different boiling points in course of employment would suffer an alteration of its mean boiling point owing to gradual evaporation of its more volatile components. This would find expression in an alteration, generally an increase, of the surface tension of the remainder of the solution, which would result in a stronger vulcanisation of the cloth than was intended. A solution of sulphur chloride in petroleum spirit shows this effect of a heterogeneous solvent very strongly. The observance of the third condition, as far as the upper temperature limit is concerned, is quite obvious. The lower temperature limit is suggested by the consideration that the lower the boiling point of a solvent the greater is its liability to very rapid volatilisation, causing such fall of temperature as to lead to the deposition of dew upon the india-rubber surface. This is indeed what very frequently happens when carbon bisulphide is the solvent employed. The fine film of water thus formed, causes decomposition of sulphur chloride, which not only imparts a most objectionable smell to the cloth, but also renders it peculiarly liable to decomposition. The fourth condition is required to make the vulcanisation as homogeneous as possible. To achieve this the solvent must almost instantly penetrate the whole thickness of the india-rubber that is to be vulcanised, but only solvents or energetic swelling agents of india-rubber possess a rate of penetration sufficiently high for this purpose. Among solvents conforming to these conditions our choice is limited practically to two: benzene and carbon tetrachloride. The latter is still a rather expensive article, and could therefore only be used in a machine enabling us to recover at least 80 per cent. of the solvent. We therefore would have to fall back upon benzene, which indeed is an excellent solvent for the purpose. In using benzene as a solvent, account must, however, be taken of the fact that its surface tension, as ascertained by the drop method, exceeds by 25 per cent. that of carbon bisulphide. Consequently, for the same degree of vulcanisation, only three-quarters of the amount of sulphur chloride should be used of that which is employed with carbon bisulphide as the solvent. This, even at low benzene prices, renders the use of this solvent rather more expensive as compared with carbon bisulphide, but as a set off we have more homogeneously vulcanised and less offensively smelling goods; and moreover, the benzene vapours, although if not absolutely innocuous, certainly have not the disastrous effects that the vapours of carbon bisulphide have upon the workmen. The superiority of benzene to carbon bisulphide as a solvent becomes very apparent in the vulcanisation of ornamented (printed) rubber surfaces, the purity of the colour effects being far better preserved when the first-named solvent is used. This is no doubt due to the absence of formation of dew upon the fabric when benzene is employed, and shows, moreover, the importance of using the india-rubber and all materials in an absolutely dry state. Many substances, such as ferric oxide, can be used with perfect impunity in the manufacture of india-rubber goods, provided all the components are dry; otherwise, on vulcanisation, basic ferric chlorides are formed, which are a prolific source of decomposition. The danger of compounds of manganese and copper, in this respect, is well known. On account of this danger of moisture in india-rubber, no compounds should be used for incorporation with india-rubber, which are capable of combining with hydrochloric acid with liberation of water, or which form hygroscopic chlorides.

—C. O. W.

## PATENTS.

*Vulcanising Apparatus, Impts. in.* H. J. Doughty, Providence, Rhode Island, U.S.A. Eng. Pat. 5470, March 5, 1898.

The apparatus consists of a vulcaniser, comprising two platens, with suitable means for heating the same and for moving them to and from each other. A separate "mold" is provided, which is inserted between the platen and contains the articles to be vulcanised. The apparatus is more particularly intended for vulcanising hollow articles, and is therefore fitted with an inflating needle, which works automatically when the platens are brought together round the "mold."—W. P. S.

*Leather and India-Rubber, and for Floor Coverings and the like; Impts. in the Manufacture of a Compound or Material applicable for use as a Substitute for, and* *in Apparatus therefor.* P. Reidel, Mannheim, Germany, and E. Kuhn, Birmingham. Eng. Pat. 11,911, June 19, 1897.

The material claimed, consists of two layers of cellulose, linen, or other long-fibre material, with an intermediate layer of cotton wool, the whole being bound together with india-rubber solution. The cellulose or long-fibre material is used in the form of a watery pulp. On a layer of this pulp the cotton wool is placed, and then another layer of pulp is run on. After passing through rollers the triple layer is dried by hot-water pipes and then runs through the rubber solution. A substitute may be used for this latter, consisting of rubber solution, scrap-rubber, glue, rosin, and linseed oil.

The material may be improved and rendered waterproof by treatment with tannin and alum.

More than the above-mentioned number of layers may be used if a thicker article is required.—W. P. S.

*India-Rubber, Leather, or other Substances, or for any other Purposes to which it may be applicable; A New or Improved Compound for use as a Substitute for.* H. C. P. Graves, London. Eng. Pat. 12,829, May 24, 1897.

The compound consists of a mixture of Trinidad asphalt (47—80 per cent.), oxidised oil (20—30 per cent.), vaselin (5 per cent.), sulphur (15 per cent.), chloride of sulphur (3 per cent.), and vegetable oils or other greasy material (10—30 per cent.).—W. P. S.

*Artificial India-Rubber and Gutta-Percha-like Substances, Further Impts. in the Production of.* F. Fenton, London. Eng. Pat. 15,269, June 26, 1897.

The patentee improves the substance prepared according to Eng. Pat. 26,518 (this Journal, 1897, **16**, 450) by the addition or admixture of sugar of lead, zinc oxide, potassium nitrate, or gum copal. These ingredients are used separately or in conjunction with gum copal. The mixing is carried out, with or without the aid of heat, in a bath containing a quantity of dilute nitric acid. From 5 to 7½ per cent. of the above materials added to the india-rubber substitute produce great hardness in the same, and they may be vulcanised without the addition of real india-rubber.

—W. P. S.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Bates and New Bateing Materials.* Der Gerber, 1898, **24**, [565], [566], [567], [568], [569], [570].

*The Removal of the Lime, together with the Scud and Dirt, from the Pelt.*—Most of the other skins, except those referred to above (this Journal, 1898, 361), require more treatment previous to tanning than the simple removal of the lime; the scud and dirt must also be removed.

Under the term scud and dirt, the tanner understands the thick coloured liquid which exudes from the skin or hide when worked over on the scudding beam. This consists of particles of the hair root or bulb, the pigment cells of the semi-liquefied hair sacs, together with the young hairs. These are not removed on the unhairing beam, but only on the scudding beam, on which a more intense pressure can be exercised. This, however, can only be satisfactorily done when the skins or hides have been brought down (lost their swelling). The liquid portion which flows out of the hides on the scudding beam is generally considered by the tanner to be milk of lime. It, however, really consists of several compounds, among which compounds of lime and hide substance, lime soap and emulsified fat are the chief. Many of these compounds of lime and hide are soluble in water, and are dissolved out, but can be rendered insoluble by dilute acids. Hence, goods which have been de-limed with acid will give a greater weight of leather than those which have been merely scudded. It will be noticed that on scudding the goods out of an acid bate, the liquid scudded out is no longer milky, but clear.

When it is necessary to remove all scud, dirt, and lime, mechanical means are generally used in conjunction with some kind of bate. In France, old waters in which the skins have been allowed to soak, are used. As these contain a considerable quantity of dissolved hide substance, they soon ferment, forming a very weak bate. Calf skins are scudded several times out of this liquor, which rapidly reduces the swollen condition of the skins, and less hide substance is dissolved out than when dung bates are used. This method, on account of the expense of hand labour, can only be used for the very finest classes. The usual method is to put the skins in large drums through which water is constantly flowing. In some cases the skins are given a mild stocking.

*Bates for the so-called "Liquefication" of the Pelt as a Preparation for the Production of Pliable and Mild Leather.*—The part-liquefication of the pelt is necessary as a preparatory process for the production of some kinds of leather; the methods which are employed also serve the purpose of removal of lime, scud, and dirt.

As was pointed out above, the scud and dirt consists not only of hair sacs and hair residues, which should be removed, but also compounds of lime with hide substance. For some varieties of leather, where pliability is an essential quality, these compounds should all be removed, but for sole leather or other class of leathers where firmness and solidity are required, these must be left in the pelt, as they unite with the tannin to form leather.

It must be understood that lime acts upon the raw pelt in two ways: firstly, it liquefies a portion which may easily be removed by rinsing in tepid water and scudding previous to their going into the acid bate; secondly, lime softens a part of the pelt which is not removed by scudding, and which, if afterwards treated in an acid bate, is rendered more solid. The first product, the liquefied portion, if treated with dilute acid, is decomposed and rendered insoluble, but this combines to a brittle mass with tannin. The second compound, which is only softened by the lime, when tanned produces a milder mass, not in any way brittle. The possibility, then, of removing much or little of both of these products—the liquefied pelt and softened pelt—depends largely on the liming that the goods have received. By long liming in old and mellow limes, more pelt is liquefied and more softened than by liming in strong, sharp limes; and the removal of these two products is accomplished by what is known as the dung bate.

The dung bate, in spite of its being one of the most objectionable compounds, and, to those not inured to it, the most disgusting material, used in the leather manufacture, is still found in nearly every branch of the leather trade; from the preparation of the heaviest buffalo hide to the finest lamb skin some use is found for the dung bate, because, with different materials to act upon, the results achieved vary. When limed goods are put into the bate liquor they soon lose their plump and swollen condition. This, however, is not due to the neutralisation of the lime, as in the case of the acid bate, because all dung bates are alkaline, except sometimes in the case of dog-dung when the dogs have been fed on bones; then the bate is at first slightly acid, owing to the presence of salts of phosphoric acid. It is not essentially on the lime that the bate works, but upon the hide substance swollen by the lime. This portion of the hide is liquefied by the action of the bate, and oozes out of the pelt as a slimy mass, carrying with it the lime and lime water which kept the hide in its swollen condition. As this fluid oozes out the pelt becomes flat, and the silky appearance is explained by the slimy mass referred to. This fluid is alkaline, and consists of compounds of lime and hide substance, together with saponified and emulsified fats. These products are, after bateing, thoroughly removed by simply swilling in tepid water or a light scudding on the beam.

In the tannery two sorts of dung bate are used—that made with bird-dung (hen and pigeon) and that made from dog-dung. The former has a sharp and penetrating action; the pelt, rapidly losing its plumpness, becomes silky, and the leather, when tanned, tough and without spring. The latter has a more softening action, consumes more pelt, producing a hollower leather. Bird-dung is therefore used when stability without spring is required; on the other hand, for glove work, in which the greatest possible spring is required, the dog-bate is employed. One can therefore define the action of the bird-dung bate as solvent for the dirt and scud, that of the dog-dung bate as solvent for the hide tissue.

The active principle of the bate is not, as is generally understood, the action of phosphoric acid salts, butyric acid, and other weak organic acids, in the removal of the lime, but a solvent action on the pelt and intercellular substance of the hide. If we mix together one part of potassium phosphate, one part of magnesium sulphate, and two parts of ammonium succinate, in 100 parts of water, then add to this two parts of ordinary bate liquor, and allow it to stand two or three days in a warm place, it will be found that this bate acts similarly to a dung bate; but without the small addition of the bate liquor as ferment, it has no action whatever upon pelt, proving that for the bateing process the bacteriological fermentation is necessary. The fermentive organisms play the chief part, and the products of fermentation only a secondary part. The micro-organisms liquefy certain constituents of the pelt; so, according to the activity, the hide cells are more or less emptied. The products of the action are chiefly peptones, which also have a solvent action, but only in a very minor capacity.

The difference in the effect produced by the bird-dung bate and the dog dung bate is accounted for by the difference in the nutrient materials of the bate. The active bacteria formed in the two bates differ from one another, although some bacteria are common to both, but on account of the different nutritive materials they are generated in different ratios. A substitute which has been suggested for the dog-dung bate is to make a broth from the fleshings scraped from hides or skins. This, if kept at a temperature of 30° to 35° C., or by the addition of a small quantity of bate liquor, will ferment quickly, and works well, although it is apt to putrefy rather rapidly, and requires to be made fresh for each batch of skins.

There are many different kinds of bacteria generated in bates, and if the process is not properly understood and looked after, the wrong sort are generated in larger quantities and obtain the ascendancy, and tend to destroy the leather. One of the worst of this class is that of the common putrefactive bacteria. When its action sets in, not only is the intercellular substance dissolved, but also the hide fibres themselves, thus producing a soft, flat, and weak leather. The best temperature for the generation of bacteria is the temperature of the blood, 37° C., but it is found in practice that it is safer to work at a temperature not exceeding 30°. Even at this temperature great care must be taken, as the bate is more liable to turn. This is less likely if the temperature does not exceed 25°. Another important factor in the working of the bate is the concentration. If the bate is made very strong, more nutritive material is present, and the bacteria generate with greater rapidity. The texture of the skin to be bated is also important, whether it be a mild, soft skin, or a harsh and more resistant one.

It is not easy to regulate the bate and prevent what is known as the turning. When putrefactive fermentation sets in, this cannot be seen until serious damage is done, the skins turning blue and then a darker colour; often small pin holes are formed; but more serious damage is done before these effects are visible, which can only be noticed on the finished leather by various characteristic marks and effects. The most common cause of the bate turning, and putrefaction setting in, is that of allowing the bate to stand without being used for some time, or if the dung has not been collected in a fresh condition and has been allowed to go rotten; and although it is impossible to absolutely safeguard oneself and prevent these faults, the following general rules should always be observed. For making the bate liquor good, pure water should be used, the dung should be dissolved in a tub of warm water, and when it is thoroughly extracted and has started to ferment, should be poured into the pit of water at the required temperature through some filtering material, such as coarse sacking, so that the solid material, which would stain the skins, is not allowed to go into the bate. The material, previous to use, should be kept either spread out in a dry place or dried, otherwise it will be liable to heat, and become dangerous for use by going rotten.

In spite of the many substitutes for the dung bate which have been suggested at various times, and although it is acknowledged on all sides that the dung bate is most objectionable and can be most dangerous, yet so far no materials have been suggested which can take its place; none have been found to perform all the functions of the dung bate; and for some classes of leather it would appear that the use of the common dung bate will always be a necessity.—J. G. P.

## PATENTS.

*India-Rubber, Leather, or other Substances, or for any other Purposes to which it may be applicable; A New or Improved Compound for Use as a Substitute for.* H. C. P. Graves, London. Eng. Pat. 12,829, May 24, 1897.

*See under* XIII. C., *page* 678.

*Leather, Impts in or relating to Tanning of.* C. J. Dunn, Watercourse, Cork. Eng. Pat. 14,076, June 9, 1897.

This invention relates to improvements in that stage of the manufacture of leather in which the green hides are suspended in vats, and kept in movement in the tanning liquor. This movement is preferably the intermittent one, and the object of the invention is to provide automatic means of regulating the duration of the periods of time during which the hides are kept alternately in motion and at rest. The motion may be conveniently given by means of an electric motor of any suitable kind, to which the supply of electricity is controlled by a clock, so that the current may be automatically switched on and off at any desired moment of time.—J. G. P.

*Leather and India-Rubber, and for Floor Coverings and the like; Impts. in the Manufacture of a Compound or Material applicable for Use as a Substitute for, and in Apparatus therefor.* P. Reidel, Mannheim, Germany, and E. Kuhn, Birmingham. Eng. Pat. 14,911, June 19, 1897.

*See under* XIII. C., *page* 677.

## XV.—MANURES, Etc.

*Ammonia, Loss of, in the Preparation of Farmyard Manure.* P. P. Dehérain. Comptes Rend. **126**, [19], 1305—1310.

The dissociation of ammonium carbonate, as Berthelot long ago showed, is determined by the loss of carbon dioxide, so that if the salt be kept in an atmosphere of carbon dioxide, there occurs practically no loss of ammonia. The author has shown this to be the case also with urine, and the conclusion he draws is that to avoid the loss of the nitrogen in the urine of farm animals, there should be plenty of litter in their stalls, so as to soak up the urine completely; that this litter should be removed regularly and frequently to the manure heap; and that fermentation in the manure heap should be encouraged, as the evolution of carbon dioxide during this fermentation prevents the dissociation of the ammonium carbonate formed from the nitrogenous constituents of the decaying matter and thus prevents loss of ammonia. All channels and drains in the stalls should also be frequently washed, so as to carry away the liquid to a reception tank and avoid exposure to the air.—J. T. D.

*Bone Superphosphate.* F. Poquillon. Monit. Scient. 1898, **51**, 408—409.

Genuine bone superphosphate is distinguished from mineral superphosphate by determining the nitrogen, but the method fails in the case of mineral superphosphates adulterated with nitrogenous matter. The method now proposed depends on the fact that when bones which have been extracted with hot water, carbon bisulphide or light petroleum, are converted into superphosphate, a fresh quantity of fat is liberated, and that this fat, which is determined by extracting with light petroleum, bears a fairly constant relation to the amount of total phosphoric acid. In "dissolved bones" (or superphosphate prepared from fresh bones) the relation is similar, provided, as is almost always the case, that the bones have been degelatinised. In England the bones are not always degelatinised, but that is when the bones are already practically free from fat, as is the case with those obtained from La Plata.

The amount of fat, per cent. of total phosphoric acid, in superphosphate (1) from bones extracted with water, and (2) bones extracted with light petroleum, is 10 to 12 and 6·5 respectively. When the fat amounts to over 12 per cent. of the phosphoric acid, it is an indication that the superphosphate was made with bones which had not been extracted.

With regard to the adulteration of mineral superphosphate with substances containing both nitrogen and fat, analyses of the usual nitrogenous matters (green and roasted horn, dried blood and roasted leather) show that none of them, added to mineral superphosphate, could give a product containing, at the same time, 0·75 per cent. of nitrogen and 1·76 per cent. of fat, as the nitrogen is enormously in excess. The relation could only be obtained by adding some exceptional nitrogenous matter, or by the separate addition of fat; and the homogeneous admixture of fat in sufficiently small quantity would be a matter of great difficulty.

The method is not considered perfect, but it will detect adulteration in most cases.—N. H. J. M.

### *Various Fertilisers and Manures.* Report of the Connecticut Agric. Expt. Station for 1897, 138—146.

*Cotton-Hull Ashes.*—This material is largely used as a source of potash for fertilising tobacco lands. Of 55 samples analysed, the highest percentage of potash was 36·45; the lowest, 10·26; and the average, 22·4. The composition of commercial cotton-hull ashes, as determined from 185 analyses, is:—Water, 9 per cent.; coal, sand, soil, and silica, 11·04 per cent.; oxide of iron and alumina, 2·07 per cent.; lime, 8·85 per cent.; magnesia, 9·97 per cent.; potash, 23·40 per cent.; soda, 2·58 per cent.; sulphuric acid, 2·56 per cent.; phosphoric acid, 9·08 per cent.; and chlorine, 1·60 per cent.

It is recommended that cotton hull ashes be bought under guarantee of the percentage of water-soluble potash.

*Cotton Hull and Hard-Wood Ashes.*—A sample gave on analysis:—Water-soluble phosphoric acid, none; citrate-soluble phosphoric acid, 1·78 per cent.; insoluble phosphoric acid, 1·92 per cent.; and water-soluble potash, 3·94 per cent.

*Lime Kiln Ashes.*—One ton of the ashes contained 868 lb. of lime, 341 lb. being as carbonate, and 521 lb. chiefly as hydrate.

*Wood Ashes.*—These ashes besides acting as a potash fertiliser, are often valuable as a source of lime in the form of carbonate. The average composition, ascertained by the analysis of 21 samples is:—Water-soluble potash, 5·5 per cent.; phosphoric acid, 1·2 per cent.; lime, 32·5 per cent.; sand and soil, 10·5 per cent.; and charcoal, 2 per cent. The potash insoluble in water need not be determined, as it is practically unavailable.

*Leached Wood Ashes.*—An average sample gave on analysis:—Water-soluble potash, 0·36 per cent.; phosphoric acid, 1·68 per cent.; lime, 15·12 per cent.; carbonic acid, 18·90 per cent.; sand and soil, 6·01 per cent.; charcoal, 1·70 per cent.; and water, 31·80 per cent.—A. S.

### *Double Sulphate of Potash and Magnesia.* Report of the Connecticut Agric. Exp. Station, 1897, 95.

This compound is usually sold as "Sulphate of potash" or "Manure salt," and contains 46—50 per cent. of potassium sulphate, over 30 per cent. of magnesium sulphate, chlorine equivalent to 3 per cent. of sodium chloride, a little sodium and calcium sulphates and varying quantities of moisture.
—A. S.

### *Perchlorate in Sodium Nitrate (Chili Saltpetre), Estimation of.* O. Foerster. Chem. Zeit. 1898, 22, [36], 357.

*See under* XXIII., *page* 694.

## XVI.—SUGAR, STARCH, GUM, Etc.

### *Beetroot Juice, A Resinous Substance from.* E. O. von Lippmann. Ber. 1898, 31, [6], 674—678.

In 1880, the author met with a substance similar to, but not identical with the resinous body described by Anderlik and Votocèk (Böhm. Zeits. 20, 248).

Towards the end of the season, in working up the last beets, a white, frothy, very light mass separated from the juice running on the char filter. This mass was not again soluble in water or sugar juice, and was not even wetted by water. It proved to be a calcium compound which, when decomposed by sulphuric acid, gave a brittle, yellow resin, melting at 278° C. with previous sintering. The substance swelled in water somewhat, could not be crystallised, gave with lime a white compound, and with alkali a solution with a strong lather. These salts could not be crystallised. The substance was dextro-rotatory, and in alcoholic solution showed about $\alpha_D = + 65^{\circ}$, but constant results could not be obtained. On oxidation with nitric acid it gave isophthalic acid, and on fusion with alkali, protocatechuic acid. After boiling with barium hydroxide, an acid was separated which was identified as caffeic acid. Further examination was impossible from lack of material. Wachtel (Oesterr. Zeits. f. Zuckerind. 9, 681, and 10, 205) observed a similar substance on working rotten beetroots.—L. J. de W.

### *Furfural, Formation of, from Cellulose and its Derivatives.* L. Vignon. Comptes Rend. 126, [19], 1355—1358.

Cellulose, boiled for an hour with dilute hydrochloric acid, hydrochloric acid and stannous chloride, hydrochloric acid and potassium perchlorate respectively, yields in the first two instances hydrocellulose, and in the third oxycellulose. These products, as also starch, cotton bleached by chlorine, and oxycellulose produced by using chromic acid as oxidising agent, were distilled with hydrochloric acid of sp. gr. 1·06, and the furfural in the distillate determined by converting it into the hydrazone. From 100 grms. of the various substances were obtained about 0·8 grm. of furfural in the case of the hydrocelluloses and the starch, 1·8 grm. in the case of the bleached cotton, and 2·1 and 3·5 grms. respectively from the oxycellulose from chlorate and from chromic acid. Larger amounts of this substance were then prepared by the use of each oxidiser, the products treated for five hours in the cold with caustic potash solution, and the insoluble residue, the precipitate formed on neutralising the potash with hydrochloric acid, and the substance remaining in the neutralised solution, separately distilled with acid and the furfural in the distillates determined. The following table gives the results:—

| — | By $KClO_3$ |  | By $CrO_3$ |  |
|---|---|---|---|---|
|  | Amount from 25 grms. of Cellulose. | Furfural from 100 grms. | Amount from 25 grms. of Cellulose. | Furfural from 100 grms. |
| Insoluble in potash. | 16·20 | 0·86 | 11·16 | 0·76 |
| Precipitated from solution in potash by acid. | 2·45 | 4·35 | 1·42 | 5·11 |
| Remaining in solution. | 6·55 | 1·10 | 12·42 | 1·51 |

Thus potash appears to break up the oxycellulose molecule into an insoluble part, with all the characters of cellulose, and a soluble part, aldehydic, containing a substance precipitable by acids, which yields relatively large amounts of furfural.—J. T. D.

### *Sugar from Orange Peel.* J. Flatau and H. Labbé. Bull. Soc. Chim. 19, [9], 1898, 408.

Fresh orange peel is macerated in water for 5—6 days, and the filtrate is evaporated in a vacuum. A gum is obtained, very soluble in water, very sparingly soluble in alcohol, and insoluble in ether, which reduces Fehling's solution in the cold. When treated with phenylhydrazine acetate, it yields a product crystallising from alcohol (80 per cent.) in yellow needles, melting at 200° C. It gives a precipitate in a neutral solution with lead acetate, and does not restore the colour to magenta solution decolorised with sulphurous acid. These tests are characteristic of mannose, which, according to Fischer gives a hydrazone, melting at 195°—200° C.—T. A. L.

### *Sugar and Liquids Rich in Sugar, Determination of Ash in.* G. Morpurgo. Chem. Zeit. 1898, **22**, [26], 257.

*See under* XXIII., *page* 699.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### *Alcoholic Fermentation without Yeast Cells.* (Fifth communication.) G. Buchner and R. Rapp. Ber. 1898, **31**, 1084—1090.

For the determination of the alcohol and carbon dioxide produced in the fermentation of sugar solutions by pressed yeast extract, the authors employed Soxhlet and Jodlbauer's method (Zeits. ges. Brauwesen, 1888, 252). 26 grms. of cane sugar were heated with 100 c.c. of pressed yeast and 2 per cent. of arsenious acid (as $AsO_2K$) at 15° C., the temperature being finally raised to 50° C. After 23 hours the sugar was completely fermented, and 12·2 grms. of carbon dioxide, and 12·4 grms. of alcohol were obtained. Some conceivable sources of error, such as the spontaneous disengagement of carbon dioxide from the albuminous bodies in the pressed yeast, the formation of carbon dioxide from the fermentation of glycogen, &c. are discussed, and shown to be of little importance.

The consideration that zymase could not be extracted from the yeast cell by water has been settled by treating yeast with water for 2 and for 20 hours, the fermentative power of the pressed yeast extract being in each case similar. The extract was prepared from washed and unwashed brewery yeast, and the respective fermentative powers showed hardly any difference. When, however, more potassium arsenite was added to the fermentation the washed yeast extract had a higher fermentative value than the unwashed.

When to a 10 per cent. solution of cane sugar in yeast extract a 2·2 per cent. solution of ammonium sulphate, nitrate, chloride, or azo-imide is added, a strong fermentation sets up in three hours. Ammonium azo-imide in a solution of 0·1 per cent. concentration has been shown by Loew and Schattenfroh to arrest the growth of yeasts and moulds, whereas, the authors show that solutions of the last-named salt of concentrations 0·36 and 0·71 per cent. have but slight influence on the fermentative action of the expressed yeast extract. A 6·7 per cent. solution of ammonium sulphate only slightly hinders fermentation. A 0·55 per cent. solution of ammonium fluoride completely stops the action of yeast extract. It is also shown that toluene acts as an antiseptic without interfering with the fermentative power of the extract.

The fermentation of dextrose, galactose, and the sugars yielding dextrose on hydrolysis, *viz.*, maltose and glycogen, is retarded by the addition of potassium met-arsenite. On the other hand cane sugar, an equimolecular mixture of dextrose and levulose, or a mixture of dextrose and cane sugar, ferment rapidly in presence of the arsenite, and thus the addition of a second carbohydrate nullifies the unfavourable action of the arsenite on dextrose. Levulose ferments more slowly with the addition of arsenite than without it, but the difference is much less than with the first mentioned aldoses.

Pressed yeast extract prepared from yeast, which had been kept for some days at 5° to 10° C. showed no fermentative power when a 2 per cent. solution of arsenite was added to it; similarly arsenite appears to repress the activity of pressed yeast extract which has been dialysed for some time and diluted with water.—J. L. B.

### *Alcoholic Fermentation without Yeast Cells.* (Sixth communication.) G. Buchner and R. Rapp. Ber. 1898, **31**, [8], 1090—1094.

The authors have investigated the behaviour of pressed yeast extract towards the more common carbohydrates with addition of potassium met-arsenite, with toluene, and without antiseptics, also in sugar solutions of different strengths. Maltose, cane sugar, *d*-glucose, and *d*-levulose are rapidly fermented, raffinose slowly, and *d*-galactose and glycogen still more so. Lactose and *l*-arabinose, as in the case of ordinary yeast, are unfermentable. It is known that dextrose and levulose do not ferment with the same rapidity when ordinary yeast is used, and as levulose ferments more slowly than dextrose a fermenting invert sugar solution is lævo-rotatory, the reason for this being that the dextrose diffuses through the cell wall more quickly than the levulose, and so comes into contact sooner with the zymase of the living yeast cell. Pressed yeast extract, however, acts uniformly on both of these sugars. Potato starch also appears to undergo a very slow fermentation, this point the authors intend to investigate further.—J. L. B.

### *Fermentation, Alcoholic, without Yeast Cells.* Seventh Communication. E. Büchner and R. Rapp. Ber. 1898, **31**, [10], 1531—1533.

For the isolation of the solid matter from pressed yeast extract, the authors take 500 c.c. of the crude extract and drop it into an exhausted Soxhlet's evaporation apparatus, afterwards quickly concentrating to a syrup at 20° to 25° C. The syrup is then placed in thin layers on glass plates, and dried in a partial vacuum at 35° C., or in an ordinary drying oven at 22° to 35° C. After about a day, the dried material is removed from the glass plates, powdered, and placed in a vacuum over sulphuric acid. From the above-mentioned quantity of yeast extract about 70 grms. of a yellowish powder were obtained, which resembled dried egg albumin. It was for the most part soluble in water, and the solution had the same fermentative power as the original yeast extract. It is immaterial whether the concentrated yeast extract is dried under diminished pressure or in the air; oxygen would therefore appear to exert little action on the zymase. The experiments were carried out partly in presence of toluene, and partly in that of potassium arsenite ($AsO_2K$), and with the latter some remarkable irregularities occurred, as have been observed before. The fact that the temperature of the yeast extract can be raised to 35° C. without consequent destruction of its activity is in harmony with the view that the inactivity of yeast extract is due to the presence of a proteohydrolytic enzyme, for the activity of this is as a rule checked in highly concentrated solutions.
—J. L. B.

### *Diastase, Barley-Malt; Researches on.* Seyffert. La Bière. **6**, [5], 68—73.

Finding that the diastase precipitated by lead acetate, instead of having a high saccharifying power gave values of only F = 14 to 25 the author determined to re-investigate the subject of the preparation and powers of this enzyme in barley malt.

With this object, a solution was prepared by digesting 500 grms. of pale dry malt for 20 hours with 1 litre of a 2 per cent. solution of neutral lead acetate, then adding 200 c.c. of water and filtering the liquid expressed from the mass. The clear filtrate was entirely free from lead, thus proving the decomposition of lead acetate by some constituent of the malt. The diastase thrown down by alcohol and purified by re-solution and a second precipitation by alcohol and ether, gave on drying *in vacuo* over sulphuric acid 9·218 per cent. of N, 8·04 per cent. of $H_2O$, and 5·36 per cent. of ash; or 10·65 per cent. of N calculated to ash-free dry substance. The diastatic power F = 175.

To explain this surprisingly high value of F, check experiments were made by digesting 3 kilos. of crushed malt with 4 litres of 30 per cent. alcohol, expressing the liquid and digesting it anew with another 3 kilos. of malt, this treatment being repeated once more. The resulting diastase contained 9·599 per cent. of N, 8·20 per cent. of $H_2O$, and 6·15 per cent. of ash, the diastatic power being F = 260. On treating this product with neutral lead acetate a diastase was obtained, containing 11·62 per cent. of N., 10·29 per cent. of $H_2O$, and 0·86 per cent. of ash; diastatic power, F = 280. The precipitate was very abundant, but the diastase obtained from the filtrate after treatment with hydrogen sulphide had a much higher diastatic power.

By steeping washed green malt in several waters until it had absorbed 58·85 per cent. of moisture, and pressing the grain, a diastase having the power F = 160 was obtained, the aqueous solution of which gave, with neutral lead acetate, a slight turbidity but no deposit.

To solve the problem of these discrepancies, a series of experiments was undertaken with the object of ascertaining whether malt contains more than one enzyme capable of breaking up starch into less complex bodies. In comparing the results the author employs the term "diastatic unit" (D.E.) to express the saccharifying effect of 1 grm. of diastase with a diastatic power F = 1, so that by multiplying the weight (or volume) of a product by its power F the total saccharifying power is obtained in grms. (or c.c.). Thus, with the standard employed by Lintner, 0·1 c.c. of diastase solution (F = 1000), corresponding to 0·005 grm. of malt, or 0·00012 grm. of diastase, the values F for diastase, and $f$ for malt are compared, by multiplying the weight of the former by the factor $\frac{0\cdot00012}{0\cdot005} = 0\cdot024$.

The material employed was de-germed green malt, dried at room temperature, water content 10·37 per cent.; value of F = 111; $f$ = 2·666 (0·09 c.c. being required to reduce the Fehling solution).

Four series of experiments with their results are given in detail.

From these trials, the conclusions drawn by the author are, in the first place, that the total number of diastatic units extracted in a given time from malt, is nearly the same, whatever solvent be employed. Next, that neither acid- nor basic lead acetate precipitates diastase, nor is an excess injurious thereto, the loss in extraction with neutral acetate being due to the mechanical action of other substances present. The loss sustained on treatment with alcohol is in all cases large, ranging from 29·2 per cent. (Series II.) to 71·1 per cent (Series IV.), only a small portion of which is due to mechanical influences. The injurious action of hydrogen sulphide is considerable, the loss ensuing when this reagent is employed as precipitant, being also great.

He also considers that the substance hitherto regarded as pure diastase is not so in reality, a much higher diastatic power being exhibited by certain of the products than was attributed to pure diastase by Lintner. The nitrogen content is higher than hitherto believed, and would indicate identity with leucosine, but for the difference in behaviour in presence of lead acetate.

A difference was noticed in the character of the ash in the various products, that from Series I. being fusible and probably containing phosphates, whereas the other series yielded an infusible ash.—C. S.

*Amylohydrolytic Ferments, The Chemical Properties of.* A. Wróblewski. Ber. 1898, **31**, [8], 1130—1136.

By means of a fractional "salting out" with ammonium sulphate, the author has isolated a very active diastase, which is not associated with a pentosan (this Journal, 1897, 1031). The preparation dissolved fairly readily in water and its solution did not coagulate either by boiling *per se* or in the presence of a small amount of acid; a flocculent precipitate resulted, however, on adding a considerable amount of hydrochloric acid and boiling. It gave reactions with nitric acid, Millon's reagent; it gave also the xanthoprotein and biuret reactions. Diastase forms a voluminous precipitate with tannic acid, which is soluble in very dilute alkali, and it is shown that under these conditions the diastase does not lose its activity. A carefully prepared diastase contained 16·53 per cent. of nitrogen, and this, taken in conjunction with its properties, leads the author to conclude that diastase is a protein.

Taka-diastase has been investigated, and by means of Brücke's reagent (this Journal, 1897, 1031) the presence of a carbohydrate was proved. The chemical behaviour of taka-diastase is in consonance with its being a protein; it, however, remains to be proved whether it is identical with the diastase derived from malt.

Invertase was prepared from pressed yeast, and Brücke's reagent shows that a considerable amount of a sugar is also present. This strongly dextro-rotatory sugar, which reduces Fehling's solution after inversion, may be precipitated with lead acetate, has no iodine reaction, and gives a brown coloration with hydrochloric acid and phloroglucinol. These properties, it will be seen, are different to those of any of the known carbohydrates. A specimen of invertase containing no sugar, inverted cane sugar very rapidly; it gave Millon's and the biuret reaction. The author considers it to be a protein, and the low amount of nitrogen found by Barth, Donath, and others, is accounted for by the presence of sugar in the preparations they analysed.—J. L. B.

*Fermentation of the Sugar Solutions obtained from Wood, Cause of the Incompleteness of, noted by Simonsen.* B. Tollens. Zeits. f. angew. Chem. 1898, [15], 337—338.

In the course of his experiments on the production of alcohol from cellulose and wood (this Journal, 1898, 365 and 181), Simonsen found that, whereas the reducing sugars obtained from the former, yielded almost theoretical amounts of alcohol, those obtained from the inversion of sawdust yielded only 57 to 62 per cent. of the theoretical quantity. The author's explanation of this is, that pentoses derived from pentosans in the wood were present, and that these, whilst reducing Fehling's solution like glucose, were unfermentable by ordinary yeast. In support of this, he states that in similar experiments on the inversion of peat (though at a lower temperature and pressure than Simonsen employed), liquids were obtained containing pentoses (xylose or possibly other unknown substances). These, estimated in the usual way and deducted from the total sugar determined by Fehling's solution, left a residue of fermentable sugar which yielded almost the theoretical amount of alcohol:—

| | 1. | 2. | 3. | 4. | 5. |
|---|---|---|---|---|---|
| Peat. | Total Sugar before Fermentation of Peat. | Pentoses. | Fermentable Sugar. Total Sugar Minus Pentoses. | Alcohol calculated from No. 3 | Alcohol found. |
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| I. | 27·87 | 14·49 | 13·38 | 6·84 | 6·79 |
| II. | 22·71 | 11·08 | 11·63 | 5·94 | 5·46 |
| III. | 11·22 | 6·67 | 4·55 | 2·33 | 1·48 |

Since pinewood, like peat, contains a considerable percentage of pentosans (7 to 10), and since these, on heating with dilute sulphuric acid under pressure, are more or less completely converted into pentoses and go into solution, the author considers that there can be no doubt as to the cause of the deficient yield of alcohol in both cases.—C. A. M.

*Yeast (Low Fermentation), Ascent of in the "Head" during Fermentation.* Gambrinus. Allgem. Anzeig. für Brau. **14**, [20], 661.

The presence of low fermentation yeast cells in the "head" forming on the yeast during fermentation is an indication of insufficient nutrition, causing the cells to remain of low specific gravity and hence easily carried upwards by the bubbles of carbon dioxide. The occurrence is thus a sign of degeneration and is accompanied by defective reproduction, the absence of "break," and by other characteristic indications. It is frequently manifested in large breweries where the same stock of pure yeast is continuously employed on a single class of worts, and in such case a change of wort should be resorted to if it is desired to retain the particular stock yeast.—C. S.

*Grapes, The Progressive Development of.* Girard and Lindet. Comptes Rend. **126**, [19], 1310–1315.

The authors have made fortnightly analyses (July 13 to Sept. 9) of grapes from the same vines, and give the results of these analyses for the grapes of Aramon, in the department of Hérault.

The stalk does not grow with the grapes. Its composition varies but slightly with time, though it gradually becomes less watery. The amount of sugars increases very notably till the time of ripening begins, after which it again decreases. A gradual diminution also takes place in the amount of acid potassium tartrate and of other acids; but the nitrogenous and mineral matters and the cellulose remain practically constant. The authors now consider that a brown substance found in the stalks, which they formerly called "resinous matter," belongs to the class of *phlobaphenes*, which, according to Etti are tannic anhydrides. The substance, extracted by ether, had the same properties and composition as the phlobaphene extracted by Etti from oak-bark or from hops. This substance and tannin appear to be complementary to one another in quantity in the stalks, and the authors suggest that the phlobaphene forms a reserve of the material, while the tannin represents it in the process of being used.

Separate analyses are given of the skin, the pulp, and the seeds of the grapes, and the following fortnightly figures of the weight of 100 grapes show that the pulp alone increases notably during ripening :—

| | | | | | |
|---|---|---|---|---|---|
| Skin | 24·10 | 23·10 | 27·50 | 27·20 | 28·69 |
| Seeds | 6·80 | 9·25 | 8·95 | 8·00 | 7·11 |
| Pulp | 87·10 | 202·70 | 288·50 | 361·80 | 414·20 |
| Total | 118·00 | 235·05 | 324·95 | 400·00 | 450·00 |

Though the weights of the skin and the seeds do not vary much, yet considerable changes in their composition occur, as well as in that of the pulp. In the early stages, the pulp accumulates chiefly acids; but when ripening begins, sugars accumulate rapidly while acids diminish. These sugars are chiefly monoses, but the authors have detected a sugar capable of inversion. The analysis of different layers of pulp has shown that while in the young grape the layers next the seeds are richest in sugar and poorest in acids, in the ripe grape these characters are borne by the outer layers. Nitrogenous and mineral matters both show steady increase in the pulp up to the time of maturity. The skin slowly loses water as the fruit ripens; the amounts of tartrate and of acids show similar variations to those in the pulp. Tannin and its anhydride increase till ripening begins, and then remain stationary, while the aromatic substance which gives the peculiar bouquet to the wine seems not to be formed till ripening has well set in. The seeds show much less variation in composition than either the skin or the pulp; the loss of weight which occurs is due partly to loss of water and partly to that of oil. Towards the end of the ripening process the seeds also lose some of their nitrogenous and mineral matters, and some of their cellulose, while at the same time there is a production of fatty acids, partly free, partly as glycerides.—J. T. D.

*Must and Wine Grown on the Saline Soil of Oran (Algiers), The Natural Presence of Large Amounts of Potassium and Sodium Chlorides in.* E. Boujean. Comptes Rend. 1898, **126**, [18], 1275—1277.

By a French statute (July 11, 1891, sec. 2), wines containing more than 0·607 grm. of chlorine per litre are to be regarded as sophisticated. But since it has often been raised as a defence that the wines derived from certain districts of Algiers naturally contain more than that amount, the author was commissioned in Sept. 1897 to establish on the spot the truth or falsehood of the contention. The juice from grapes grown in various places in the vicinity of the salt lakes of Oran were analysed, and also the wine obtained from the must. Contrary to what has been stated, the author found that the vines bear fruit for from 8 to 10 years and yield on the average 70 hectolitres per hectare. Of 28 samples of wine examined only two contained less than 0·607 grm. of chlorine per litre; one with 0·315 grm. derived from a district 15 kilometres distant from the salt lakes; the other with 0·582 grm. derived from well-irrigated soil. The remaining samples contained from 0·60 to 4·50 grm. per litre. The following is an example of the comparative results obtained with the juice of such grapes and with the wine derived from it :—

| — | Chlorine. | Potassium Oxide. | Sodium Oxide. | Phosphoric Acid. |
|---|---|---|---|---|
| Grape must | 4·552 | 3·591 | 2·291 | 0·715 |
| Wine | 4·500 | 2·165 | 2·719 | .. |

In the author's opinion the foregoing figures establish the fact that the chlorides of potassium and sodium are readily absorbed by the vine from the soil in large proportion, and that they remain in the wine after fermentation of the must.—C. A. M.

*Wine, Bitterness of.* F. Bordas, Joulin, and de Raczkowski. Comptes Rend. 1898, **126**, [18], 1291—1293.

The bacillus causing the bitterness of wine (this Journal, 1898, 260) has a terminal spore and ciliæ at one of its extremities, and moves very rapidly. It grows well in a culture medium containing potassium nitrate, but does not convert that salt into nitrite, nor does it produce indol when grown in a medium containing peptones. It is able to withstand a dry temperature of 100° C. The most suitable temperature for its cultivation is from 25° to 37° C. The addition of calcium carbonate to a peptone culture medium caused the amount of volatile acid produced and the bitterness to become very marked. The quantity of tartrate added to a medium was observed to have slightly decreased after 15 days.

The analysis of two portions of the same sterilised wine, of which the second was inoculated with the bacilli, gave the following results after six months :—

| — | Sterilised Wine. | Wine rendered Bitter. |
|---|---|---|
| Alcohol, per cent. by vol. | 10·4 | 10·6 |
| Reducing sugar, as glucose per litre | 3·32 | 2·80 |
| Tartrate | 3·43 | 1·30 |
| Glycerin | 7·50 | 4·80 |
| Acids, total, as $H_2SO_4$ | 3·92 | 6·61 |
| „ volatile, as acetic | 1·03 | 2·57 |
| „ fixed, as $H_2SO_4$ | 2·89 | 3·38 |
| Ammonia | 0·008 | 0·037 |
| Nature of volatile acids | Acetic | Acetic, butyric |
| „ fixed acids | Succinic | Succinic |

From these and other results the author concludes that the bacillus acts chiefly on the glycerin in the wine with the formation of acetic and butyric acids. In one instance, in which lactic acid was found in a bitter wine the author attributed its formation to the action of the bacilli on the glucose. The diminution in the amount of tartrate was more noticeable in the wine than in the artificial culture media; but inasmuch as this is a point on which Pasteur, Vergnette-Lamothe, and Glenard were not in agreement, the author intends to make further experiments in the matter.
—C. A. M.

*Natural Wine, The Presence of an Alkaloidal Substance in.* G. Guérin. J. de Pharm. et de Chim. 1898, **7**, 323—324.

A body, of an alkaloidal nature may be obtained from wine by evaporating a litre of the latter on a water-bath until the alcohol is driven off, a small quantity of tartaric acid being first added. After cooling, the liquid is made alkaline, and shaken with ether in a separating funnel. On drawing off the ethereal layer and evaporating, a residue is obtained which, when dissolved in a few drops of water slightly acidulated with sulphuric acid, gives all the general reactions of an alkaloid. The author suggests that this is the same as the substance found in the liquid obtained by the fermentation of sugar with brewers yeast.—W. P. S.

*White Wines Manufactured from Red Wines by Bleaching the latter with Potassium Permanganate.* L. Hugounenq. J. de Pharm. et de Chim. 1898, **7**, 321—323.

The author lately examined a sample of white wine and found that it contained 0·59 grm. of manganese oxide (MnO) per litre. Inquiries showed that a considerable business is done in bleaching red wine with a mixture of animal charcoal and potassium permanganate in order to obtain white wine.

A simple way of detecting wine so made is to add a little caustic soda solution to the suspected sample, and then a small quantity of hydrogen peroxide, and shake; the liquor is immediately coloured a deep mahogany red if manganese be present.—W. P. S.

### *On Beer Sarcina.* F. Schönfeld. Wochenschr. für Brau. 1898, 285—293.

The author concludes, from his experiments, that normal yeasts show different degrees of resistance to the growth of sarcina, but are less resistant than wild yeasts. Under suitable conditions a sarcina can be acclimatised to a certain yeast, and will destroy a beer prepared by this yeast. Sarcina is more anærobic than ærobic. The growth of sarcina is favoured by a medium temperature, absence of air, and agitation; and is hindered by a high hopping, a high alcohol percentage, and the presence of air.—A. L. S.

### *Volatile Acids produced in Fermentation, Formula for the Estimation of, by Duclaux's Method.* F. Bordas and de Raczkowski. J. de Pharm. et de Chim. 1898, 7, [10], 479.

*See under* XXIII., *page* 700.

### *Cider Vinegar.* E. F. Ladd, Gov. Agric. Exp. Stat., N. Dakota. Bull. 32, 278.

Cider vinegar should be the fermented juice of apples. It usually contains from 2 to 5 per cent. of solid matter and from 0·3 to 0·5 per cent. of ash. The latter consists largely of potassium carbonate and phosphates, and gives a strong potassium flame. The acids average about 4 per cent. This vinegar is of a dark brown colour; it has a decidedly fragrant odour and a characteristic flavour.
—W. P. S.

### *Vinegar Bacteria.* W. Henneberg. Zeits. für Spiritusind. 1898, 180—181.

The author divides the vinegar bacteria into four classes:—

1. The quick vinegar bacteria, *B. acetiginum*, is the only one known at present.

2. Beer bacteria. This includes those forms which have been isolated from beer. *Termobacterium aceti*, *B. aceti*, *B. Pasteurianum*, *B. Kutzingianum*, and *B. acetosum*.

3. Wort bacteria, *B. oxydans* and *B. industrium*.

4. Wine bacteria, *B. xylinum* and *B. ascendens*.

*B. acetigenum* forms a thin fairly tough slightly lustrous skin. The cells are motile, small, round, and not in chains. It gives a blue coloration with iodine and sulphuric acid. It oxidises alcohol and dextrose. The solution is usually bright. It grows best at 33° C. and can oxidise solutions containing 4·8 per cent. alcohol.

*B. (Termobacterium) aceti Zeidler* forms a very tender skin. The cells are motile and occur generally singly and seldom in long chains. It oxidises alcohol and dextrose. The solution is very turbid. It grows best at 25° C., and the acetic acid is very slowly oxidised. This bacteria can oxidise solutions containing 8 per cent. of alcohol.

*B. aceti Hansen* forms a slimy wrinkled skin. The cells are not motile and occur singly and in chains. It oxidises alcohol and dextrose, grows best at 34° C., and can oxidise solutions containing 11 per cent. of alcohol. The solution is generally bright.

*B. Pasteurianum* forms a fairly thick finely folded skin, The cells are not motile, and are usually in chains, and become coloured blue by iodine. It oxidises alcohol and dextrose, grows best at 31° C., and can oxidise solutions containing 9·5 per cent. of alcohol. The solution remains quite bright.

*B. Kutzingianum* forms an easily broken slimy skin. The cells are not motile, small, single, and coloured blue by iodine. It resembles B. Pasteurianum in its properties, but the solution is turbid.

*B. acetosum* forms a strong skin, at first smooth, but becoming folded. The solution remains bright and the cells are not motile. It oxidises alcohol, galactose, and dextrose; grows best at 28° C., and can oxidise a solution containing 11 per cent. of alcohol.

*B. oxydans* forms a tender slimy skin. The cells are large and motile, and at first in chains. It oxidises alcohol, glycerin, mannitol, levulose, dextrose, galactose, maltose, and dextrin. It grows best at 20° C., and can oxidise solutions containing 7 per cent. of alcohol. The solution is usually turbid.

*B. industrium* forms a slimy transparent skin which is easily broken. The solution remains bright. The cells are motile and do not form chains. It oxidises alcohol, glycerin, mannitol, galactose, levulose, dextrose, cane-sugar, maltose, lactose, dextrin, and starch. It grows best at 25° C., and can oxidise solutions containing 4·7 per cent. of alcohol.

*B. xylinum* forms a very thick tough skin. The cells are not motile, occur singly, and give the cellulose reaction when treated with iodine and sulphuric acid. It oxidises alcohol and dextrose, grows best at 33° C., and can oxidise solutions containing 6 per cent. of alcohol.

*B. ascendens* forms a very tender skin, which climbs high up the side of the vessel. The solution becomes very turbid. The cells are large, not motile, and seldom in chains. It can oxidise solutions containing 12 per cent. of alcohol, but does not oxidise dextrose, and grows best at 27° C.—A. L. S.

### *Tertiary Alcohols, A New Reaction of.* G. Denigès. Comptes Rend. 1898, 126, [18], 1277.

*See under* XXIII., *page* 697.

### *Beer, Estimation of Alcohol in.* L. Bousquet. La Bière, 1898, 6, [5], 65.

*See under* XXIII., *page* 700.

### *Reversible Zymohydrolysis.* A. C. Hill. Proc. Chem. Soc. 1898, [197], 156—158.

This investigation was undertaken to ascertain whether zymohydrolysis is a reversible process or not. The hydrolysis of maltose by maltase was selected for the purpose, the enzyme being obtained by extracting dry and finely-powdered low-fermentation yeast, which had been heated under certain precautions, as detailed in the paper. It was found that the extract could be sterilised without destruction of its activity by filtration through a Pasteur-Chamberland filter.

The analytical methods for estimating the extent of hydrolysis were (1) determination of the cupric-reducing power of the mixed sugars; (2) determination of the specific rotatory power. The details of the methods and limits of accuracy are dealt with fully in the paper. To simplify calculations, maltose is reckoned as maltose hydrate, $C_{12}H_{22}O_{11}.H_2O$.

The zymohydrolysis of maltose into glucose is hindered by the presence of glucose and is incomplete, and both these effects are more marked the more concentrated the solution of sugar. The retardation is shown by two sets of experiments. In the first set, the initial velocities of conversion are compared in a solution of maltose of a certain concentration with that in a solution of half maltose and half glucose of like total concentration. The ratio of these velocities should be not greater than 2 if the glucose were without influence; actually it is much greater, being about 3 for a 4 per cent., and 4·85 for a 20 per cent. sugar concentration. In the second set, the time curves are compared for two flasks, each containing initially the same concentration of maltose, whilst to one, glucose has been added in equal concentration. If glucose were without influence, the time curve of conversion of maltose into glucose should be identical for each flask, but a much slower conversion occurs in that containing the added glucose, and the effect is much more marked with a total sugar concentration of 20 per cent. than with one of 4 per cent.

When the enzyme acts in a 20 per cent. solution of glucose, a small reverse action can be detected by the polarimeter. With a 40 per cent. concentration, the reverse action is well marked, and when sufficient time has been allowed for this action, which is a slow one, to approach completion, the reversion indicated by the change in rotatory and cupric-reducing powers amounts to 15 per cent. of the sugar. Control flasks show no change. With a 40 per cent. solution of sugar, of which 75 per cent. was glucose and 25 per cent. maltose (hydrate), hydrolysis proceeded till 83·25 per cent. of the total sugar was glucose, and the equilibrium point, therefore, for this concentration is not far from 84 parts glucose to 16 parts maltose (hydrate). Equilibrium points for various concentrations have been determined;

and it is found that in dilute (2 per cent.) solutions hydrolysis is practically complete. The same equilibrium point is approached either with a solution of maltose or with one of glucose of the same concentration.

Maltose has been detected as a product of the reverse action by the use of phenylhydrazine. An osazone having a greater solubility in boiling water than glucosazone has been separated, and this crystallises in plates, has the same percentage of carbon and nitrogen as maltosazone, but melts at a temperature somewhat lower than maltosazone from pure maltose, though not lower than that prepared from ordinary maltose under similar conditions, *viz.*, from a mixture of maltose with excess of glucose to which some boiled enzyme solution has been added, the proteid being then removed as completely as possible before treatment with phenylhydrazine. The synthesised maltose has not yet been isolated, but it is considered that the evidence of its presence, coupled with the increase of optical rotatory power and corresponding decrease in cupric-reducing power observed in concentrated glucose solutions in the presence of maltase, justify the conclusion that the zymohydrolysis of maltose is a reversible process.

### PATENTS.

*Beer, Process and Product for the Preparation of Certain Kinds of.* O. Trachmann, Kiel, Germany. Eng. Pat. 9018, April 8, 1897.

An extract which contains all the ingredients of beer before fermentation, is prepared by concentrating the boiled hopped wort, prepared as usual, to a jelly-like consistency. A pure yeast is dried in a vacuum over sulphuric acid, and preserved in sterilised glass tubes. In order to prepare beer, a weighed quantity of the extract is dissolved in a suitable quantity of water, and a little of the dry yeast added. A vigorous alcoholic fermentation takes place and beer is produced.—A. L. S.

*Cereals for Use in Brewing and Distilling, Impts. in and Apparatus for the Preparatory Treatment of.* B. Willcox, London. From The Action Maschinenbau-Anstalt, late Venaleth and Ellenberger, Darmstadt, Germany. Eng. Pat. 10,899, May 1, 1897.

This relates to an apparatus for washing barley or other grain. The grain is mixed with water in a funnel-shaped chamber, and then, by means of a jet of water introduced at the bottom, forced up a central tube, the water and dust separated by a strainer, and the grain uniformly distributed in the said vessel, and sprinkled with fresh water. A valve is placed in the apex of the vessel so that it may be used as a steeping vessel, the valve serving for the discharge of the grain when steeped.—A. L. S.

*Yeast, Impts. relating to the Production of, and to Apparatus therefor.* J. H. Folkerts, London. Eng. Pat. 11,515, May 8, 1897.

This is an improvement of the inventor's patent, 18,296 of 1895 (this Journal, 1896, 824), and relates chiefly to an improved method of separating the grains from the wort. This is done by bag filters made of filtering cloth, which, the author states, give better results than the method usually adopted.—A. L. S.

*Alcohol, Impts. in Apparatus for Treating and Maturing Fluids containing.* A. J. Boult, London. From G. R. Besser, Berlin, Germany. Eng. Pat. 13,435, May 31, 1897.

This is an improvement of patent 7813 of 1895 (this Journal, 1895, 879). The special electrodes are omitted, and the cooling coil and the walls of the receptacle are used as electrodes.—A. L. S.

*Spirit Stills, Impts. in and relating to Continuous Working.* J. E. Dunn, Rendham, Suffolk. Eng. Pat. 2980, Feb. 5, 1889.

The trays of the column are fitted with partitions which cause the wash from its entry to exit to make a series of revolutions; the vapour tubes are arranged zig-zag throughout each of these channels, and are provided with deflecting hoods. The upper part of the column consists of hollow, flat drums, so arranged that the spirit vapours travel inside in one direction and the condensing water outside in the opposite. The wash-heater consists of three compartments, the upper and lower communicating by a series of vertical tubes, the wash and the spirit vapours passing in opposite directions through the chambers.—A. L. S.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Cereal and Leguminous Seeds, Composition of the Proteids of: Practical Results of the Researches of E. Fleurent.* A. Livache. Bull. Soc. d'Encouragement Ind. Nat. 1898, **97**, 547—550.

The varying quality of bread made from pure, white flour is not due, as might be supposed, to the amount of gluten in the flour, but to the composition of the gluten. By means of a modification of the ordinary methods of analysis, avoiding the use of too strong reagents, Fleurent was able to separate and determine the two constituents of gluten, glutenin, and gliadin. The results show that the gluten of good wheat-flour contains 18 to 34 per cent. of glutenin which is dry and pulverulent, and 82 to 66 per cent. of gliadin. The latter is a viscous substance, and is the cause of the adhesiveness of gluten.

In the case of rye, rice, maize, barley, and buckwheat flour there is considerably less gliadin in relation to the glutenin; this prevents the gliadin from binding, and renders the extraction of the gluten, by the methods usually employed for wheat flour, impossible. For a gluten to be extractible there must be a minimum and maximum relation between the amounts of glutenin and gliadin present.

For obtaining well-developed and readily digestible bread, the gluten should contain 25 per cent. of glutenin and 75 per cent. of gliadin. With as little as 20 per cent. of glutenin the bread develops well, but becomes compact during baking; whilst if the glutenin amounts to 34 per cent. the bread does not rise well, and, when baked, is compact and indigestible.

The amount of water retained by the bread when baked depends on the amount, and not on the nature, of the gluten.

Inferior wheat flour may be improved by the admixture of flour of different composition. When, for instance, the flour contains an excessive amount of gliadin, the proper relation of glutenin to gliadin (namely 1:3) may be obtained by adding a calculated amount of leguminous flour, the nitrogenous matter of which consists chiefly of legumin (which resembles glutenin) and gluten, which contains very little gliadin.

In examining mixed flours, in order to ascertain whether the addition has been made for the purpose of improvement or of adulteration, it would be sufficient to determine the relation between the amounts of glutenin and gliadin.

By means of a specially constructed crusher, it was found possible to separate wheat grains into three distinct zones. The products were then ground and the gluten examined. The results, obtained from a large number of wheat samples, showed that the percentage of gluten increases from the centre of the seed to the outside; the increase of glutenin in the gluten is in the same direction, whilst the gliadin, of course, diminishes as the glutenin increases. When the composition of the central part of the grain, which amounts to 42 to 58 per cent. of the whole, is known, it is possible to decide how much of the remaining portions of the grain should be included or rejected, in order to obtain the right proportions of gliadin and glutenin.

—N. H. J. M.

*Leguminous Plants and Cereals, Albuminoids in the Flour of.* E. Fleurent. Comptes Rend. **126**, [19], 1374—1377.

The author has extended his researches on cereals to leguminous plants, especially to horse-beans. The flour of these beans contains 25 to 32 per cent. of nitrogenous matters: a sample gave—Legumin 18·92, vegetable albumin 0·2, gluten (composed of glutenin 9·52, gliadin 2·40) 11·92. Putting together as analogous in their properties the legumin and glutenin, and the albumin and gliadin, the composition of the nitrogenous matters may be put as—

| | | | |
|---|---|---|---|
| Caseins ...... | Legumin....... | 60·95 | 91·60 |
| | Glutenin ...... | 30·65 | |
| Fibrins ...... | Albumin....... | 0·64 | 8·40 |
| | Gliadin ........ | 7·76 | |
| | | | 100·00 |

The proportion of glutenin to gliadin in samples of wheat-flour of the highest value for bread-making is about 25 to 75; and the auther concludes that the practice of adding to certain wheat-flours 2 or 3 per cent. of bean flour, is based on the fact that the wheat-flours in question contain too high a proportion of gliadin, and that the addition of the bean-flour of low gliadin content makes the proportion of glutenin to gliadin in the mixture nearly that just quoted.—J. T. D.

*Milk, Human, Report on the Chemical Examination of Two Hundred Individual Specimens of.* V. Adriance and J. S. Adriance, New York. 1898, A Clinical Report, 1—42.

The specimens of human milk analysed by the authors were taken at periods varying from 2 days to 15 months of lactation. Of these, 120 were regarded as normal, or in other words, as having no ill effect upon the child. The average percentage variation in the constituents was:—Fat, 3 to 4; carbohydrate, 6 to 7; proteids, 1 to 2; salts, about 0·20; and total solids, 12. The reaction of the milk was uniformly alkaline.

During the first two weeks the change in the composition of the milk was very marked. As a general rule a ratio of about 3 to 1 was observed between the amounts of fat and of proteid, but occasionally, especially during the colostrum period, the proteids were abnormal and higher than the fat. The extreme percentages of fat in normal samples were 7·61 and 1·31 per cent.; the average 3·83 per cent. The carbohydrate was more constant than the other constituents and though steadily increasing in amount from the commencement to the end of lactation, showed only a slight total increase. The lowest amount found in normal milk was 5·35; the highest 7·95 per cent. The percentage of proteids rapidly fell after the commencement of lactation, starting with an average of 3·31 on the second day, becoming 2·20 on the third day, and then gradually diminishing to about 1 per cent. after 8 months, and finally to about 0·6 per cent. after 15 months. The salts also slowly diminished from the commencement. On the second day they averaged 0·27 per cent., whilst in the fifteenth month they amounted to only 0·14 per cent. The total solids remained very constant in amount throughout lactation, the increase in the carbohydrate and decrease in proteid very nearly compensating each other. The average during the first seven months was 12·20 per cent., falling gradually to 11·50 per cent. in the fifteenth month. The average specific gravity throughout lactation was 1·030. Any increase in the amount of fat lowered the gravity, whilst in the case of proteids, the greater the percentage the higher the gravity. Hence the specific gravity fell as the proteids diminished during the later months of lactation.

The average percentage composition of human milk during the course of lactation is shown in the following list selected from a fuller table given by the authors:—

| Period. | Fat. | Carbohydrates. | Proteids. | Ash. | Total Solids. | Water. |
|---|---|---|---|---|---|---|
| 2 days ..... | 3·83 | 5·80 | 2·77 | 0·27 | 12·20 | 87·80 |
| 3 " ..... | 3·83 | 5·90 | 2·20 | 0·27 | 12·20 | 87·80 |
| 7 " ..... | 3·83 | 6·22 | 1·90 | 0·24 | 12·20 | 87·80 |
| 14 " ..... | 3·83 | 6·63 | 1·70 | 0·20 | 12·20 | 87·80 |
| 4 weeks .... | 3·83 | 6·68 | 1·78 | 0·19 | 12·20 | 87·80 |
| 6 months .. | 3·83 | 6·78 | 1·25 | 0·16 | 12·20 | 87·80 |
| 9 " .. | 3·83 | 6·84 | 0·94 | 0·18 | 12·04 | 87·96 |
| 12 " .. | 3·83 | 6·95 | 0·84 | 0·15 | 11·77 | 88·23 |
| 15 " .. | 3·83 | 6·90 | 0·63 | 0·14 | 11·50 | 88·50 |

The latter part of the paper deals with the subject of abnormal milk and its composition, as influenced by such factors as the constitution, environment, and nutrition of the mother.—C. A. M.

*Milk, Analysis of. [Determination of the Amounts of Added Water.]* A. Villiers and M. Bertault. Monit. Scient. **12**, April 1898, 270—271.

In the original paper a number of analyses are given of milks from various sources, and also the refractive indexes of the wheys obtained from these milks. The results show that, while the amount of the different constituents vary considerably, the refractive index remains almost constant. The whey was obtained by adding to the milk half its volume of dilute acetic acid (1—100), heating, and filtering after cooling. After correcting for this dilution, the refractive index was found to be between 40 and 41 divisions of the oleo-refractometer scale (Jean and Amagat's instrument).—W. P. S.

*Adulteration in Belgium.* M. de Molinairi. Rev. intern. falsific. **11**, 35; Chem. Centr. 1898, **1**, [21], 1148.

1. *Cattle Foods.*—It was found that linseed meal was mixed with flour refuse, that cotton-seed: oil and cake, contained a considerable quantity of cotton fibres, and that dried malt was adulterated with 1 to 15 per cent. of earth nut shells. Barley meal showed mildew and "brand spores," whilst maize was decayed. The so-called "Pondéine" consisted of wheaten flour, chaff, buckwheat, &c., and small pieces of dried blood.

2. *Foods.*—Butter was adulterated with 30 to 75 per cent. of margarine, and milk with 20 per cent of water. Mustard contained flour and turmeric; buckwheat flour was mixed with wheaten flour and yeast with potato starch. "Apple syrup" was adulterated with beetroot molasses, and essence of cloves contained a large quantity of sesamé oil. Wine was found to contain an excessive quantity of gypsum; chicory was too rich in mineral matter.—A. S.

*Grapes, The Progressive Development of.* Girard and Lindet. Comptes Rend. **126**, [19], 1310.

*See under* XVII., *page* 682.

*Colouring Matters in Milk, Detection of Added, especially "Orleans."* J. Froidevaux. Ann. Chim. Anal. Appl. **3**, 119; Chem. Centr. 1898, **1**, [19], 1068.

*See under* XXIII., *page* 697.

*Formaldehyde in Articles of Food, Detection of.* E. Rimini. Ann. di Farmacol. 1898, 97; Chem. Centr. 1898, **1**, [21], 1152.

*See under* XXIII., *page* 697.

## PATENTS.

*Milk, A Method of Treating, and obtaining therefrom a Gelatinous Proteid Substance and thus Forming a Nourishing Food for Invalids and Others.* A. S. Poyser, London. Eng. Pat. 7220, March 19, 1897.

The milk (whole, or skimmed) is coagulated, the mass sterilised by scalding with steam or boiling water, and the curd, previously pressed, or pressed and dried, mixed with a solution of an alkaline salt, such as the carbonate or bicarbonate of potassium, or sodium, or borax, and allowed to stand in the cold or at a gentle heat, until gelatinisation occurs. This "milk jelly" may be used alone, but for commercial purposes the patentee prefers to mix it with gelatin and clarify it as an ordinary jelly, after the addition of colouring, sweetening, or flavouring matters. It can be used in admixture with food substances, such as ground rice, or in beef tea, tea, coffee, brandy, sherry, and the like. Claim is also made for its reduction to powder or flakes by drying either by itself, or after the addition of food material such as the substances referred to above.

—C. A. M.

*Albuminoud Substances, Organic; An Improved Process for the Solution of, and for the Production of Soluble Preparations of Albumin.* H. Bremer, Munich. Eng. Pat. 12,712, May 22, 1897.

The inventor claims a process for rendering soluble organised albuminous substances whether of animal or vegetable origin, stating that the methods hitherto described have only had reference to the production of soluble compounds from unorganised albuminous bodies such as casein and certain vegetable albumins. The albuminous substances (meat, cocoa, cereals, &c.) are made soluble by being treated under a high temperature (*e.g.*, 130° C.), and pressure (*e.g.*, three atmospheres) with a solution of ammonia or of a readily dissociated ammonium salt such as the carbonate. The solutions thus obtained are evaporated *in vacuo* at a temperature below that at which the albuminous matters coagulate either directly or after the addition of soda or potash lye, milk of lime, or alkaline carbonates. If the evaporation temperature be too high the albuminous compounds are rendered insoluble in the absence of ammonia in excess. Claim is also made for the separation of the dissolved albuminous substances by saturating the liquid with salts, such as sodium chloride, ammonium sulphate, and sodium sulphate, by precipitating them with alcohol in excess, or by neutralising the solution with acid, and also for the treatment of the substances thus separated by evaporating them at a low temperature with a sufficient quantity of an acid or of an alkaline carbonate.—C. A. M.

*Milk, Separated; Impts. in or relating to the Treatment of, for Alimentary Purposes.* H. Higgins, Cambridge. Eng. Pat. 13,141, May 27, 1897.

Claim is made for the precipitation of caseinogen from separated milk by heating the latter to from 55° to 60° C., and adding hydrochloric acid in the proportion of 5 lb. to 100 galls. of milk. The supernatant liquid is drawn off into a second vessel containing milk heated to the same temperature, where only 2 lb. of additional hydrochloric acid are required to precipitate the casein from 100 galls. of milk. Similarly the supernatant liquid from the second vessel reinforced by 4 or 5 lb. of hydrochloric acid will precipitate the caseinogen from 200 galls. of heated milk in the third vessel, and so on as often as may be found convenient. —C. A. M.

*Milk, Separated; Impts. in or relating to the Treatment of, for Alimentary Purposes.* H. Higgins, Cambridge. Eng. Pat. 13,142, May 27, 1897.

The caseinogen precipitated from the milk, preferably by means of hydrochloric acid, is washed in about 150 galls. of boiling water, which causes it to assume a sticky condition in which it can be drawn out into strings or rolled into sheets. Soon after withdrawal from the water it becomes friable and can then be powdered. It can be used in this state by itself or mixed with other food substances such as rice, meal, sugar, glucose, and the like.—C. A. M.

*Milk, Separated; Impts. in or relating to the Treatment of, for Alimentary Purposes.* H. Higgins. Eng. Pat. 13,143, May 27, 1897.

The caseinogen obtained from separated milk and washed in boiling water (see preceding patent) is formed into blocks which are allowed to dry partially, and are then ground into powder. On arranging this powder in layers, and allowing it to dry further, the particles adhere to one another, and a material resembling thin slices of bread is obtained, which on further drying, becomes crisp. Other food substances (rice, flour, &c.) may be added to the caseinogen before it is formed into blocks.—C. A. M.

*Eggs, The Manufacture of a Substitute for.* A. Bernstein. Boston, U.S.A. Eng. Pat. 29,189, Dec. 9, 1897.

The albumin and casein obtained from skimmed milk as described in the inventor's English patent 14,336 (this Journal, 1897, 930), or by other means, are worked into a paste by mechanical means, such as rolling, after the addition of a certain proportion of salt as a preservative. When used as a substitute for eggs in baking, this curd paste is stirred up with milk into an emulsion before being added to the dough.—C. A. M.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Bacteria, The Conveyance of, by Underground Water.* E. Pfuhl. Zeits. für Hyg. 1897, 549; through Proc. Inst. Civil Eng.

The author gives the results of experiments carried out with a view of ascertaining whether it is possible for water situated deep in the soil to become contaminated by means of bacteria penetrating downwards from the surface. Two pits were excavated in a gravel soil situated in a clearing of the forest bordering the Rhine, and adjacent to the Strassburg waterworks. In the first pit, which was 1 m. (3·28 ft.) deep, the subsoil water stood at a level of 50 cm. (1·64 ft.) below the surface. The second pit which was 1·5 m. (4·92 ft.) deep, 1 m. (3·28 ft.) wide, and 12 m. (39·37 ft.) long, was 8 m. (26·25 ft.) distant from the first, and was perpendicular to the direction of the flow of underground water. Bouillon cultures of the *Micrococcus prodigiosus* and of the fluorescent *Vibrio* were introduced into the first pit, and at intervals of about half an hour, samples were withdrawn from the second pit lower down, and cultivated in the usual way on gelatin or on agar plates. It was found that in one hour, the micrococci, and in two hours the vibrios had passed through the 8 m. (26·25 ft.) of intervening gravel which separated the two pits. In further experiments, the *prodigiosus* bacilli were found to pass into the supply of a tube well, drawing its water through gravel from a distance of 3·7 m. (12·14 ft.) from the point where the cultures of bacteria were introduced into the water near the surface.—A. S.

### PATENTS.

*Sewage and Polluted Water, Impts. in connection with the Purification of.* F. P. Candy, Bowdon, Cheshire. Eng. Pat. 12,984, May 27, 1897.

This invention provides for automatically changing the delivery to and discharge from filter beds, the same being brought about by floats which, by alternately rising, work a lever to which valves or "changers" are attached. Secondary valves, also actuated by floats, automatically regulate the speed of the discharge into and from the filters. The sewage is delivered over the filter bed through a conduit which is caused to travel to and fro over the bed by a water-wheel which in turn is fed from a supply of the liquid to be filtered. An alternative method of spreading the water on the filter beds is by means of two or more conduits running on slopes, and so connected that the running down of the full one draws up the other empty one, and so on.—W. P. S.

*Sewage, Impts. in or relating to the Purification of, and Apparatus therefor.* W. D. Scott-Moncrieff, London. Eng. Pat. 13,400, May 31, 1897.

In the process claimed, the sewage is first submitted to the combined action of aerobic and anaerobic organisms, secondly to the action of anaerobic organisms, and lastly to that of organisms working under highly aerobic conditions, this final treatment being repeated or continued in successive vessels. In carrying out the process, the sewage is led under a bed of coke or flints (contained in a suitable tank) through which it rises and is then run off into a closed chamber. This chamber has a false bottom on which stand inverted open-mouthed vessels in which the gases produced by the organisms are occluded, providing highly anaerobic conditions. From this chamber the sewage runs into an automatically tipping trough, which delivers it through a series of sprinkling trays on to open tanks filled with coke. After being sprinkled over a successive number of these coke tanks the effluent is conducted to a suitable place of discharge.—W. P. S.

*Purifying Water and Steam, and for Heating and Softening Water; Impts. in and relating to Apparatus for.* W. J. Baker, Scarborough. Eng. Pat. 15,384, June 28, 1897.

The plant claimed, consists essentially of three parts; an oil separator, a water heater, and a series of settling tanks.

The exhaust steam is blown on to the surface of a layer of water contained in the separator, when much of the oil in the steam separates and floats on the surface of the water. After leaving the surface of the water, the steam passes through a set of angle-iron "bafflers," whereby it is deprived of the remainder of the oil. From the oil separator the steam then passes into the second part of the apparatus, where it meets with a spray of the water to be softened, the two being thoroughly mixed by running over alternate cups and cones contained in the vessel. If necessary, a softening solution is also mixed with the water as it runs through this vessel and the water may be strained as it enters. The heated water then runs into the settling tanks, from which it is drawn off for use.—W. P. S.

*Furnaces, Refuse-consuming; Impts. in, and Means for Utilising the Heat therefrom.* T. R. Berkley, London. Eng. Pat. 19,718, Aug. 26, 1897.

*See under* II., *page* 653.

## XIX.—PAPER, PASTEBOARD, Etc.

### *Waste Lyes from Cellulose Pulp Mills, Utilisation of.* Papier Zeit. 1898, 23, [19], 687.

Franz Müllner, of Hugohütte, has obtained an Austrian patent for a process of recovering by-products from waste lyes (soda and sulphite) from wood pulp mills. By adding bauxite or other minerals containing alumina to the waste lyes, and injecting $CO_2$ or $SO_2$ (according as to whether soda or sulphite liquor has been used), the alkali of the waste lye may be obtained in a form suitable for re-use, and also hydrate of alumina. The organic matter may be removed by evaporation and ignition. The salts containing sulphur, in the lyes are, by this treatment, converted into sulphur compounds of the alkalis, which cause a further precipitate of hydrate of alumina when the residue is lixiviated, giving rise to losses.

The treatment is as follows:—The waste lyes and wash water of the cellulose mill making any kind of pulp, are evaporated in the ordinary manner. At any time during the evaporation, pulverised bauxite or other substance containing alumina is added in sufficient quantity to convert the entire alumina into alkali aluminate by the action of the alkali contained in the waste lye. If the lye contains sulphur compounds, a corresponding amount of limestone or caustic lime should also be added.

The waste lyes contain caustic and carbonated alkalis (which are uniformly converted into alkali carbonates on ignition), and alkali sulphides, with other alkali sulphur salts. On calcining the mixture, these salts combine with the bauxite, according to the following equations:—

$$6NaOH + Al_2(OH)_6 = Al_2(ONa)_6 + 6H_2O.$$
$$3Na_2CO_3 + Al_2(OH)_6 = Al_2(ONa)_6 + 3H_2O + 3CO_2.$$
$$Na_2SO_4 + 4C = Na_2S + 4CO.$$
$$Na_2SO_4 + 3C = Na_2S + 3CO.$$

There is more than sufficient carbon contained in the organic matter in the lye to account for the following reaction:—

$$3Na_2S + 3CaCO_3 + Al_2(OH)_6 = Al_2(ONa)_6 + 3CaS + 3H_2O + 3CO_2.$$

The final products contain the alkali mostly as alkali-aluminate.

The calcined mass is lixiviated in the ordinary manner, and the clear liquid, containing the alkali aluminate in solution, is treated in either of the two following ways:—

A. *For Soda Pulp.*—$CO_2$ is passed into it, when aluminium hydrate is precipitated—

$$Al_2(ONa)_6 + 3CO_2 + 3H_2O = Al_2(OH)_6 + 3Na_2CO_3.$$

The solution of alkali carbonate is causticised with lime and the resulting lye used for making the cellulose. The aluminium hydrate is separated by a filter press. The $CO_2$ may be obtained from a lime kiln, and the burnt lime used for causticising.

B. *For Sulphite Pulp.*—$SO_2$ is passed into the solution, precipitating aluminium hydrate as follows:—

$$Al_2(ONa)_6 + 3SO_2 + 3H_2O = Al_2(OH)_6 + 3Na_2SO_3.$$

These are separated as under A, and the recovered $Na_2SO_3$ again used for boiling cellulose. The $SO_2$ is obtained by burning sulphur or pyrites.

The pure aluminium hydrate obtained may be used for making aluminium salts, or it may be treated with hot sulphuric acid and used for sizing paper.

To counteract the inevitable loss of alkali, a sufficient quantity of sodium sulphate or bisulphate and lime, is added to the condensed lye before the bauxite.—S. P. E.

### *Photographic Paper.* Papier Zeit. 1898, 23, [36], 1319.

Paper intended for photographic purposes needs great care in its manufacture, requiring a plant of the best material, where the pulp and water are not allowed to come into contact with iron or other impurity. The paper must be of considerable strength, in order to stand the different treatments it has to undergo in the process.

The paper is coated with gelatin and barytes, and then treated with the sensitised emulsion or albumin. The nitrate of silver contained in the emulsion gradually penetrates the coating, and if the paper contain any particle of iron or dust, a black speck is formed.

"Ferro-prussiate" paper does not require so much care or such exceptional conditions as the ordinary sensitised paper, therefore several other mills supply this paper.

—S. P. E.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

### *The Cactus Alkaloids.—III.* A. Heffter. Ber. 1898, 31, [8], 1193–1199. (See also this Journal, 1895, 381; 1896, 554, 827.)

*Pellotine*, $C_{10}H_{10}O(OCH_3)_2N.CH_3$.—The hydriodide of the base, $C_{13}H_{19}NO_3.HI$, is obtained by passing dry hydriodic acid gas into an ethereal solution of the alkaloid.

*Mezcaline*, $C_7H_5(OCH_3)_3N.CH_3$, is soluble in water with a strong alkaline reaction; the aqueous solution expels ammonia from its salts and precipitates the hydroxides from copper, lead, and zinc salts. The base is obtained in the most pure state by extracting an alkaline solution of the sulphate with chloroform, evaporating off the solvent, and adding anhydrous ether to the gelatinous residue, when the alkaloid separates as a white powder, consisting of very small needles. On heating, it softens at 105° C., and melts between 150° and 160°. The hydrochloride and hydriodide of this base are obtained by double decomposition between the sulphate and barium chloride or iodide. Mezcaline forms a compound with methyl iodide, $C_{11}H_{17}NO_4.CH_3I$, obtained by digestion in methyl alcohol solution. By the oxidation of an aqueous solution of mezcaline sulphate with potassium permanganate, an acid containing nitrogen is formed, melting at 169°, readily soluble in boiling water, alcohol, and ether, and subliming above the melting point. Mezcaline is the constituent of *Anhalonium Lewinii*, to the presence of which the plant owes its remarkable properties, 0·2 grm. of the alkaloid taken internally induces "beautiful and quickly changing colour-visions."

*Anhalonidine*, $C_{12}H_{15}NO_3$, is readily soluble in water; chloroform extracts it from the solution. Recrystallised from benzene it forms small octohedra, which soften at 151° and melt at 154° with discoloration. In aqueous solution the base shows the same alkaline properties as pellotine. It is optically inactive; the earlier statement to the contrary is erroneous. The hydrochloride, $C_{12}H_{15}NO_4.HCl$, forms warty masses of hard transparent prisms, readily soluble in water, with difficulty in alcohol. The sulphate forms thin white prisms uniting in masses; it is very soluble in water, almost insoluble in absolute alcohol. Anhalonidine contains two methoxyl groups, but no methylimido group.

*Anhalonine.*—The hydriodide, $C_{12}H_{15}NO_3.HI$, is formed by the same reaction as the corresponding pellotine compound; it forms yellow needles, readily soluble in water and alcohol. Anhalonine is a secondary base, a nitroso compound is obtained on the addition of sodium nitrite to an acidified solution of the hydrochloride; yellow oily drops separate, which crystallise from alcohol or ether; it is obtained pure by the careful addition of water to the alcoholic solution, the crystals soften at 51°, and melt completely at 59°. By the action of methyl iodide (1 mol.) on anhalonine (1 mol.) methyl alcohol solution, methylanhalonine hydriodide is obtained. Anhalonine contains one methoxyl group and no hydroxyl group.

*Lophophorine*, $C_{13}H_{17}NO_3$, has only been obtained in the form of colourless drops. The hydrochloride is readily soluble in water and hot alcohol; from the latter solvent it crystallises in spherical aggregates of white microscopic needles without water of crystallisation. The aqueous solution of the salt is without action on polarised light. Lophophorine contains one methoxyl group, but no hydroxyl groups. No methylimido group is present; it is isomeric, but not identical, with methylanhalonine.—A. C. W.

*Camphor from Pinene, A New.* [*Pinocamphone.*] O. Wallach and J. A. Smythe. Annalen, **300**, [3], 286—293.

By the reduction of nitrosopinene with zinc and acetic acid, pinylamine, $C_{10}H_{15}NH_2$, an unsaturated base, is obtained. Nitrous acid converts this amine into pinocarveol, $C_{10}H_{15}.OH$, and oxidising agents transform the latter into pinocarvone, $C_{10}H_{14}O$, a ketone isomeric with carvone (Annalen, **268**, 197, **277**, 149). The semicarbazone of this ketone though soluble with difficulty, is not readily purified, the oxime crystallises well, melts at 98°, and is readily reconverted into pinocarvone.

If the reduction of nitrosopinene be conducted in the following manner, a considerable quantity is converted into pinocamphone. Dissolve 5 grms. of nitrosopinene in 40 c.c. of glacial acetic acid, warming gently, add water until separation commences, and then a large excess of zinc dust. When the violent action is over, heat on the water-bath for 3—4 hours, pour off the liquid, steam distil, extract the distillate with ether, shake the ethereal solution with sodium carbonate, dry over potash, distil off the ether and fractionate *in vacuo*. Pinocamphone, $C_{10}H_{16}O$, is obtained as a liquid of terpene-like odour, boiling at 211°—213° C. under ordinary pressure. Its semicarbazone melts at 199°—200° C., its oxime readily crystallises in large transparent plates melting at 86°—87°, and is volatile with steam. Pinocamphone is, like camphor, a completely saturated compound. By reduction with sodium in a wet ethereal solution, pinocamphone is transformed into pinocampheol, $C_{10}H_{17}.OH$, boiling at 218°—219° C., its phenylurethane melts at 98°. Pinocamphonoxime is converted into the nitrile by the action of sulphuric acid.

By the reduction of nitrosopinene bromide a ketone is obtained isomeric with pinocamphone; this is probably bihydrocarvone. In the same reaction a base is obtained, not identical with pinylamine, and apparently differing from bihydrocarvylamine.—A. C. W.

*Caffeïne, Some Derivatives of.* A. Einhorn and E. Baumeister. Ber. 1898, **31**, [8], 1138—1141.

The observation of Gomberg (Amer. Chem. Journ. **17**, 402) is confirmed, that by digestion of a dilute alcoholic solution of chlorocaffeïne with potassium cyanide, the amide of caffeïnecarboxylic acid is directly formed, which by warming with dilute sulphuric acid is converted into caffeïnecarboxylic acid. Maly and Andreasch (Monatsh. für Chem. **4**, 231), have found that caffeïne is converted into caffeïdinecarboxylic acid by warming with alkalis. In accordance with these observations the authors find that from caffeïnecarboxylamide by the action of caustic alkalis, caffeïdinedicarboxylic acid is produced. By the action of strong hydrochloric acid on the sodium salt, caffeïnedicarboxylic acid is obtained. Boiled with water, carbonic acid is evolved, from the concentrated solution left caustic potash separates caffeïdine as an oil which gradually solidifies.

Chlorocaffeïne reacts with secondary bases; the combination is effected by heating with rather more than 2 mols. of the base at 130°—180° in a sealed tube. Piperidylcaffeïne, $C_8H_9O_4O_2.C_5H_{10}N$, crystallises in colorless needles melting at 142°. Diethylaminocaffeïne begins to sinter at 91° and melts at 109°, dibenzylaminocaffeïne melts at 162° C.

—A. C. W.

*Hydrocotarnine, Electrolytic Preparation of.* E. Bandow and R. Wolffenstein. Ber. 1898, **31**, [10], 1577—1578.

In the reduction of cotarnine by zinc and hydrochloric acid, the zinc chloride double salt of the base is formed and prevents further reaction. Electrolytic reduction gives good results. Crude cotarnine (30 grms.), as obtained from the mother-liquors of opianic acid after the decomposition of narcotine by manganese peroxide and sulphuric acid, was dissolved in 170 grms. of sulphuric acid (1 : 5); platinum-foil electrodes were employed, the anode being immersed in dilute sulphuric acid. The current strength at the cathode was 3·5 ampères, the voltage 5·3. The brown cotarnine solution is decolorised in about 4 hours. The reaction is finished when a test gives a pure white precipitate with ammonia. Pure hydrocotarnine is at once obtained, in spite of the use of crude cotarnine. Hydrastinine, $C_{11}H_{13}NO_3$, may be reduced to hydrohydrastinine, $C_{11}H_{13}NO_2$, by the same method with equal success.—A. C. W.

*Gaïacyl.* C. André. J. de Pharm. et de Chim. 1898, **7**, 324—328.

"Gaïacyl" is the calcium salt of guaiacol-mono-sulphonic acid, and has the formula $(C_7H_7O_2SO_3)_2Ca$. It is prepared by treating guaiacol with its own weight of concentrated sulphuric acid, the acid being added gradually to prevent too great a rise in temperature. When all the acid has been added the mixture is allowed to stand for 48 hours, and then diluted with 6—7 times its weight of distilled water, heated to 80° C., neutralised with calcium carbonate, and filtered. The filtrate is evaporated to dryness, and dissolved in alcohol to separate a small amount of insoluble matter. On evaporating the alcoholic solution, "gaïacyl" is obtained as a mauve-coloured powder, very soluble in water and in alcohol, but insoluble in oils. It is said to be useful as a local anæsthetic.—W. P. S.

*Ouabaïn (Strophanthin), Action of Alkalis on.* Arnaud. Comptes Rend. 1898, **126**, [18], 1280—1282.

Ouabaïn, or Strophanthin (this Journal, 1898, 268), is not hydrolysed by alkalis either in aqueous or alcoholic solution, but yields a hydration product containing no trace of reducing sugar. This body is readily obtained by heating 1 part of crystalline ouabaïn with 3 parts of strontium hydroxide in 10 parts of water for 12 hours at the boiling temperature. After saturating the hot product with carbon dioxide, the liquid is filtered, concentrated *in vacuo*, and poured little by little, into 10 times its volume of absolute alcohol. The precipitated strontium salt is dissolved in water, decomposed with the exact amount of sulphuric acid, and the liquid, when filtered and concentrated *in vacuo*, yields the hydration product which the author terms "ouabaïc acid."

This substance is a mono-basic acid capable of decomposing carbonates of the alkalis and alkaline earths. It is amorphous, yellowish-white, and resembles gum in appearance. It is very soluble in water and alcohol, but insoluble in ether. It melts about 235° C., and decomposes with an evolution of gas and odour of caramel. It is lævo-rotatory, and its barium salt was found to have $[\alpha]_{D20} = -46{\cdot}40°$ in aqueous solution. Its salts are very soluble in water, and crystallise with difficulty. Its formula, deduced from the analysis of its salts, is $C_{30}H_{48}O_{13}$. The lead salt is very soluble in water, but subacetate of lead precipitates it and its soluble salts. Dilute mineral acids hydrolyse it, forming rhamnose and a resinous body.

Ouabaïc acid can also be obtained by the action of water alone by heating its dilute aqueous solution in a sealed tube at 180° C., the resulting liquid containing a notable proportion of the acid ($C_{30}H_{46}O_{12} + H_2O = C_{30}H_{48}O_{13}$). But if the solution be more concentrated (15 to 20 per cent.), this treatment soon leads to hydrolysis.

In the cold, alkalis do not change ouabaïn beyond increasing its solubility in water and rotatory power. When fused with potash or soda, ouabaïn decomposes at 220°—240° C., with the liberation of carbon dioxide and formation of a little oxalic acid and resin. Although ouabaïn appears to behave like an anhydride of ouabaïc acid, the author has not succeeded in regenerating it from the acid.—C. A. M.

*Veratrine and some of its Derivatives.* G. B. Frankforter. Contribution from the Chemical Laboratory of the University of Minnesota. Amer. Chem. J. **20**, [5], 358—373.

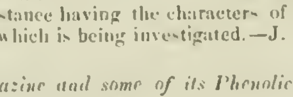
The pure micro-crystalline base, Veratrine, was found to contain one molecule of water, and to correspond to the formula $C_{32}H_{49}NO_9.H_2O$; the melting point was 146°—148° C., and that of the gold salt, $C_{32}H_{49}NO_9HCl.AuCl_3$, 178°—182° C. The presence of a methoxyl group was proved by the Zeisel method, indicating that the constitution of the alkaloid may be represented as $C_{31}H_{46}NO_8(OCH_3)$. Three *iodo compounds* were obtained, containing four, three, and one molecule of iodine respectively. An interesting *chloral compound*, $CCl_3CH:O(C_{32}H_{48}NO_9)O(C_{32}H_{48}NO_9)$, was obtained by the reaction of chloral on the alkaloid. The compound formed a waxy mass, soluble in excess of chloral, insoluble in ether and in chloroform, but very soluble in water and in alcohol. In its general physiological character it resembled veratrine, but had remarkable properties as a local irritant; applied to the moistened skin it produced vesication. It was hygroscopic.

Veratrine was found to combine with methyl iodide, forming the compound $C_{32}H_{49}NO_9CH_3I.1\frac{1}{2}H_2O$. This was a light yellow crystalline powder melting at 210°—212° C. By treating this body with silver oxide, *veratrine methylhydroxide*, $C_{32}H_{49}NO_9.CH_3OH$, was obtained. This appeared to be physiologically inactive, but was very unstable, and readily decomposed on warming. Dilute hydrochloric acid formed with this, *veratrine methylhydroxyhydrochloride* $C_{32}H_{49}NO_9.CH_3OH.HCl$, compounds with ethyl bromide and allyl iodide, having the formulæ $C_{32}H_{49}NO_9.C_2H_5Br$ and $C_{32}H_{49}NO_9.C_3H_5I$, were also preprepared.

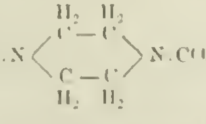
By heating alkaloidal veratrine at various temperatures, a crystalline substance having the characters of veratric acid was obtained, which is being investigated.—J. O. B.

*Dimethylpiperazine and some of its Phenolic Compounds.* [*Lycetol.*] P. Cazeneuve and Moreau. Comptes Rend. 1898, **126**, 1573—1375.

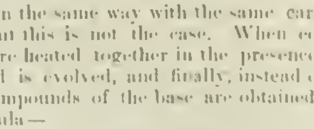
In a former communication (Comptes Rend., Feb. 1898) it was shown that piperazine or hexahydro-γ-diazine reacts with phenolic carbonates to form aromatic diurethanes having the constitution—



It might be expected that dimethylpiperazine supposed to have the formula—



would react in the same way with the same carbonic esters of phenols, but this is not the case. When equimolecular proportions are heated together in the presence of alcohol, carbonic acid is evolved, and finally, instead of urethanes, diphenolic compounds of the base are obtained, having the general formula—



In this way the authors have obtained the diphenate, diguaiacolate and the α and β-dinaphtholates of dimethylpiperazine, and details are given of their preparation and properties. This hydrolysing action of dimethylpiperazine, due to its great chemical energy, suggests that it is not a secondary base containing 2NH groups in the nucleus, or in other words, that it is not the dimethyl derivative of piperazine. The fact that it does not react with alcoholic iodides and the non-formation of urethanes, such as are produced by piperidine, coniine, piperazine, and also as will be shown later by tetrahydroquinoline, are reasons for doubting the presence of NH in a closed chain in this base. Its further study is especially important in view of its recent introduction into therapeutics as "Lycetol," the tartrate of dimethylpiperazine.—T. A. L.

*Rose Oil, The Examination of.* P. N. Raikow. Chem Zeit. 1898, **22**, [52], 523—524.

A reply to Dietze's answer to the author's paper (see this Journal, 1898, 75, 386, and 599). The author had stated his examinations were conducted on filtered oils, because he employed the crude oil produced by rough methods of distillation, and not the technically pure rose oils. The filtration being conducted at 35° C. removed water and dirt and not stearoptene. Dietze states that by "congealing point" he means the commencement of solidification; the limit 15°—20° C. is regarded by the author as too wide. A rose oil of the previous year, of undoubted purity, recently examined, had the density 0·855 at 27° C. (water at 17·5° = 1); saturation point, 20·5°; rotation at 25° in 100 mm. tube, 2° 45′; acid number, 1·1; saponification number, 15·5; ester number, 14·4; and ratio of free acid to ester numbers, 13. These figures do not agree with those given by Dietze (*loc. cit.*).—A. C. W.

*Geraniol and Citronellol, New Method of Separating.* J. Flatau and H. Labbé. Comptes Rend. 1898, **125**, [24], 1725—1727.

The following method is said to give an almost perfect separation. The essence to be examined is saponified with 5 per cent. alcoholic potash. The portion of the product which distils at 120°—140° under 30 mm. pressure, is mixed with an equal weight of phthalic anhydride, and dissolved in an equal volume of benzene (crystallisable); the solution is boiled for an hour, the benzene then evaporated, and from the residue the sodium salts of the geranyl and citronellyl phthalates are prepared. The gelatinous mass so obtained is dissolved in tepid water, the impurities removed by washing the solution with ether, and the phthalates then liberated by hydrochloric acid diluted with an equal volume of water. The mixture is dissolved in petroleum spirit at 20°—25° C., and the solution cooled to about −5°, when the geranyl phthalate crystallises out; the crystallisation may be hastened by adding a crystal of the pure salt. The crystals are separated and washed with petroleum spirit. On evaporating the ligroin solution, citronellyl phthalate separates as a thick golden-yellow oil, which does not crystallise when cooled. The alcohols are obtained from the phthalates by boiling with 5 per cent. alcoholic potash, and are purified by fractional distillation or distilling with steam. Geraniol is a colourless liquid with a rose-like odour, boiling at 228°·2 C. under 750 mm. pressure without the least decomposition, and at 122° C. under 29 mm. (thermometer completely immersed in the vapour), its specific gravity at 0° = 0·8965. Citronellol has a stronger and even more rose-like odour than geraniol, and boils at 221°·5 C. under 755 mm. pressure.

By means of the above method the following results have been obtained. Indian geranium oil contains 63 per cent. of geraniol and 17 of citronellol; essence of citronella, 40 of geraniol and 6 of citronellol; Bourbon essence of geranium, 10 of geraniol and 70 of citronellol; Bulgarian otto of roses, 70 of geraniol and 15 of citronellol; essence of balm-mint, 20 of geraniol and 12 of linalool. *Geranyl phthalate* crystallises from petroleum spirit in rhombic tables, melts at 47° C., is easily soluble in the usual solvents in the cold, and in petroleum spirit at 25°; it begins to separate from the petroleum spirit solution at 10°—12° C., and is completely insoluble at 5°. Its silver salt is a white powder, and melts at 133°·8 C. The *tetrabromo derivative* is obtained by the action of bromine on the phthalate in ethereal solution; it melts at 114°—115° C.

The following salts have also been prepared by heating the alcohols with an excess of the acid in presence of anhydrous cadmium acetate. The yields of the geraniol compounds are good, and of the citronellol compounds almost theoretical. The *acetate of citronellol* boils at 172°—173° C. under 34 mm. pressure, the *valerate* at 194°—196° under 31 mm., the *caproate* at 168°—170° under

33 mm., and the *crotonate* at 138°—140° under 35 mm.; the *valerate of geraniol* boils at 130°—132° C. under 30 mm. All these substances have strong odours resembling those of the alcohols.—E. W. W.

### *Musk-scented Ketone.* A. Baur-Thurgau. Ber. 1898, **31**, [9], 1344—1319.

According to certain French and German patents of 1892–93, "Artificial musk" is formed by the nitration of ketones obtained by the dry distillation of the calcium salts of toluic and valeric acids, from toluene (or xylene) aluminium chloride and valeryl chloride, and by the action of phosphoric and benzoic anhydrides on butyltoluene. According to the author, musk-scented ketones are not obtained by these processes. If, however, the acetyl, butyryl, or valeryl groups be introduced into butyltoluene or butylxylene, ketones are obtained which, on nitration, yield products of a strong musk odour. To a cooled mixture of 1 part of butyltoluene, 10 parts of carbon bisulphide, and 6 parts of aluminium chloride, 6 parts of acetyl chloride are added as rapidly as the energetic reaction permits. After distillation on the water-bath, the residue is poured over ice and worked up in the usual manner. The ketone so obtained boils at 255°—258° C. On nitration at 0° in 100 per cent. nitric acid, it yields a dinitro derivative, $C_{13}H_{16}N_2O_5$, melting at 131° C., with a strong smell of musk. The isomeric symmetrical dinitro derivative melts at 103°, and is quite odourless. The methyl ketone obtained in a similar manner from butylxylene melts at 48° and boils at 265°; its dinitro compound melts at 136°. The methyl ketone of butylxylene melts at 50° C. and boils at 290°—295° C.; its dinitro derivative melts at 128°. The valeryl ketone boils at 185°—190° under 14 mm. pressure; its dinitro compound melts at 151° C. The three dinitro compounds last mentioned all possess the odour of musk.—A. C. W.

### *Spearmint Oil, Russian.* Report of Schimmel and Co., Leipsic, April 1898, 46.

Russian spearmint oil has recently appeared in commerce in considerable quantity. It differs in odour from the American and German varieties, and has a lower specific gravity, 0·885 at 15° C., and a lower optical rotation, −23°; while the ordinary varieties have a specific gravity between 0·920 and 0·980, and a rotation as high as −43°. The Russian oil was found to contain from 5 to 10 per cent. of lævocarvone, from 50 to 60 per cent. of lævogyrate linalool, and 20 per cent. of cineol and lævo-limonene. The observed difference between this oil and German and American spearmint oil is due to the presence of a larger percentage of linalool and less carvone.—J. O. B.

### *Oil from the Leaves of Cæsalpinia Sapan.* Report of Schimmel and Co., Leipsic, April 1898, 53.

The leaves of the sapan tree yield from 0·16 to 0·2 per cent. of an almost colourless essential oil, having the sp. gr. 0·825 at 20° C. It is strongly dextrogyrate, the rotation ranging from 75° to 100° in 200 mm., and the chief fraction boils at 170° C. Its odour is pepper-like, recalling phellandrene, for which body the oil gives a marked reaction. —J. O. B.

### *Sweet Gum Oil [Liquidambar Styraciflua].* Report of Schimmel and Co., Leipsic, April 1898, 53.

The fresh leaves of *Liquidambar styraciflua* have, when bruised, a peculiar terebinthous odour, and when distilled yield 0·085 per cent. of a yellowish-green oil, having the sp. gr. 0·872 and rotation −38·45°; the saponification number is 0·9; the acetylisation number, 25·2. The oil contains terpenes, borneol, and bornyl acetate.—J. O. B.

### *Lophanthus Oil.* Report of Messrs. Schimmel and Co., Leipsic, April 1898.

The oil was supplied by E. Kremers, and distilled by him from *Lophanthus anisatus*, indigenous and abundant in Wisconsin, U.S.A. It has a specific gravity of 0·943 at 20° C. and a rotation of −7° 10′. The pleasant anisic odour of the oil recalls that of *Solidago odora*.—J. O. B.

### *Oil of Ageratum Conyzoides.* Report of Schimmel and Co., Leipsic, April 1898, 53.

This oil, distilled from the fresh plant at Buitenzorg, had a specific gravity of 1·015 at 27·5° C. and a rotation of −5·5 in 200 mm.; it boiled at about 260° C. It probably contains a sesquiterpene.—J. O. B.

### *Croton Oil.* O. Javillier. J. Pharm. Chim. 1898, **7**, [11], 524—527.

The divergent published data for croton oil are attributed to the method of its preparation. It is found that oils obtained by expression and by maceration in ether (sp. gr. 0·758) and by double digestion in alcohol, 95 per cent. at 75° C., differ considerably in their characters, thus: *Solubility in Absolute Alcohol.—Expressed oil:* Entirely soluble in less than an equal volume. As soon as this quantity is added, precipitation commences, and on increasing the volume of alcohol, the oil separates into two distinct layers. *Ether-extracted oil:* Similar to expressed oil. *Alcohol-extracted oil:* Entirely soluble. *Freezing Point.*—Expressed oil and ether-extracted oil congeal at −7° C.; alcohol-extracted oil at −8° C., but begins to cloud at 0° C. *Iodine Number.*—Expressed oil, 100; ether-extracted oil, 108; alcohol-extracted oil, 91·2. *Koettstorfer's Number.*—Expressed oil, 192·0; ether-extracted oil, 194·5; alcohol-extracted oil, 260·6. *Free Acid Number.*—Expressed oil, 27·3: ether-extracted oil, 30·9; alcohol-extracted oil, 260·6.—J. O. B.

### *Dimethylheptenol: A New Unsaturated Tertiary Alcohol.* P. Barbier. Comptes Rend. 1898, **126**, [20], 1423—1426.

On heating 200 grms. of lemonol (geraniol) with 100 grms. of potash dissolved in a little alcohol at 150° C. for eight hours, precipitating with water, washing the oily product with dilute acetic acid and water, drying, and twice fractionating *in vacuo*, a substance is obtained, boiling at 79° C. under 10 mm. pressure. This is dimethylheptenol, $C_9H_{18}O$, a colourless, rather mobile liquid, with an odour recalling that of natural dimethylheptenone. It unites with a molecule of bromine, and heated at 140° C. with acetic anhydride and sodium acetate, forms an ester, boiling at 84°—86° under 11 mm. The products of the oxidation of dimethylheptenol by chromic acid mixture are acetone, natural methylheptenone boiling at 170°—172°, and a small quantity of levulinic acid.

Dimethylheptenol is found in small quantities in essence of linalool; its acetate has been isolated from the lower portions obtained in the preparation of licarhodol acetate. —A. C. W.

### *Peru Balsam, Examination of.* E. Hirschsohn. Rev. intern. falsific. **11**, 59; Chem. Centr. 1898, **1**, [19], 1068.

The author states that pure Peru balsam, on warming with lime water for half an hour, does not give a solid mass, and that with 80 per cent. acetic acid it gives only a faint cloudiness from which no oily drops separate. The petroleum spirit extract of genuine Peru balsam is said to give no greenish-blue or green coloration with copper acetate, and also to remain colourless on addition of hydrochloric acid.—A. S.

### *Theobromine, Identification and Determination of the Purity of.* M. François. J. Pharm. Chim. **7**, [11], 521.

*See under* XXIII., *page* 700.

## PATENTS.

### *Hydrogen Peroxide, Impts. in the Manufacture of, and in Apparatus therefor.* S. Rosenblum, S. Rideal, and The Commercial Ozone Syndicate, Ltd., all of London. Eng. Pat. 12,274, May 18, 1897.

A process for the manufacture of hydrogen peroxide by the action of ozone and water on terpenes and other organic compounds. The claims are for the different methods by which the admixture of the three substances may be effected, for the purification of the product by atomising it in a jet of ozone, for the manufacture of hydrogen peroxide *in situ* for bleaching purposes by the use of the same reaction, and for devices in apparatus for intermingling ozone with a spray of water and the organic liquid. The apparatus claimed and described is represented in central vertical section in Fig. 1. A is a cylindrical casing carried by the supports $A^1$. B is a closed vessel with two sets of stirrers, $C^1$ and $C^2$, rotating in opposite directions. $B^2$ is a pipe for the admission of turpentine and water; $B^1$ is a pipe, ending in a perforated coil, for the admission of ozone. The hollow conical mounting $A^3$ carries a circular dish D, shown in detail in Fig. 2. The space $D^3$, between the dishes D and $D^2$, communicates with the upper portion of B by means of the pipe E, and with the perforations $D^4$ and the tubular spindles $D^5$. The pipe F extends from the bottom of B to the space $D^6$, between $D^2$ and its cover $D^7$, which is perforated with conical holes $D^8$, into which extend the spindles $D^5$. The pipe G effects communication between the chamber A and the vessel B.

Fig. 1.

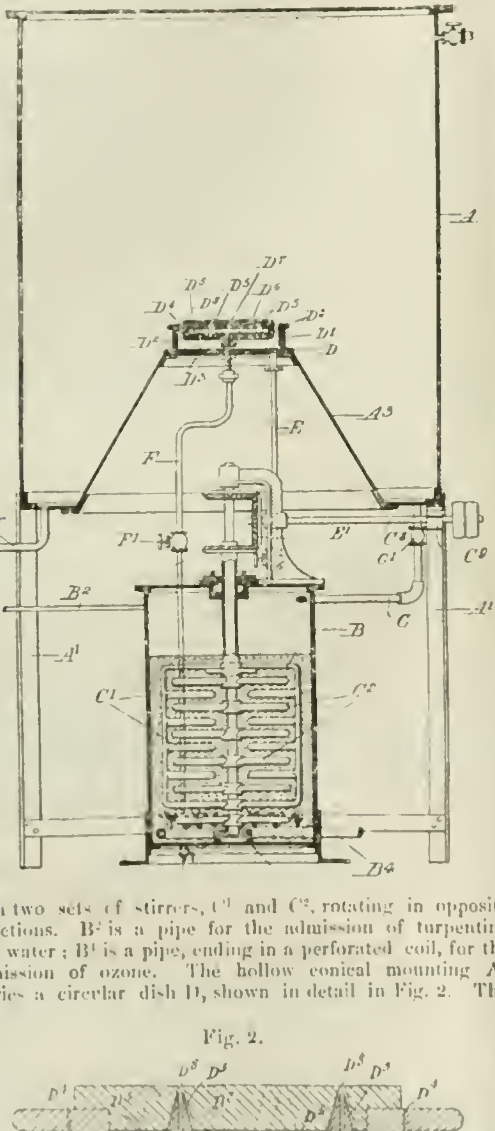
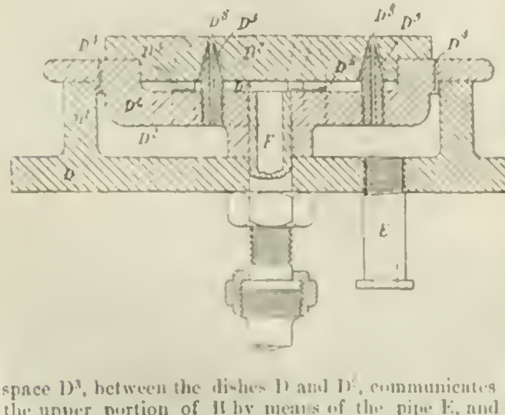


Fig. 2.



The operation is as follows:—An emulsion of turpentine and water is made in B; ozone is admitted under pressure through $B^4$; this ozonises the mixture and forces it through F into the space $D^6$, whilst ozone passes into the space $D^3$ by means of E. This ozone partially passes by $D^4$ into A, and partially escapes through the spindles $D^5$, drawing with it the emulsion through the passages $D^8$, and thus producing a fine spray in the chamber A. The mixture of water, turpentine, and hydrogen peroxide formed, may be withdrawn or returned to B by means of G for re-treatment. A modified apparatus for use with steam or for the production of hydrogen peroxide *in situ*, is also described and claimed.—A. C. W.

*Purine, Derivatives of, containing Sulphur; Impts. in the Manufacture or Preparation of.* J. Y. Johnson, London. From C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 4736, Feb. 25, 1898.

The manufacture of sulphur derivatives of purine (so-called thiopurines) by treating the halogen substitution products of purine with alkaline hydrosulphides, is claimed. Mono-, di-, and trithiopurines have been obtained by this process.—A. C. W.

## XXI.—PHOTOGRAPHY.

*Photographic Sensitisers, Oxamine Colours as.* E. Valenta. Phot. Corr. 1898, **35**, 198; through Chem. Zeit. Rep. 1898, 140.

Several of the oxamine colours of the Farbwerk Friedrichsfeld, P. Remy, Mannheim, make better sensitisers for the red end of the spectrum than the dyes at present employed. They bear additions of ammonia. Further experiments are in progress.—F. H. L.

*Developing Baths, Part played by Diffusion in.* A. Guebhard. Comptes Rend. **126**, [19], 1341—1344.

When a slightly fogged plate is placed in a developing bath and a solid partition arranged to separate two parts of the plate, minute currents occur through any capillary opening in the partition—a phenomenon which the author has called *internal osmose*. If two parts of the plate be covered by a large and a small watch-glass, for example, the direction of the currents is always from the larger to the smaller—from the place where there is relative abundance of unused developer to that where there is less. But the black trace of these currents is always surrounded by a lighter band or wave-front, probably caused by the resistance to the horizontal motion of arriving molecules by those already there, thus causing eddies in a vertical direction. This is certainly not a case of simple diffusion, and probably the figures called *effluves* by Colson are susceptible of the same explanation, and are due not entirely to chemical nor thermic, but rather to mechanical causes. The action of a developed plate on another nearly in contact with it in the bath, like that of coins and similar articles lying on the developing plate, is probably accounted for by the proportionality of the action of the developer to the varying thicknesses of its layer, so long as these thicknesses are very small; and no doubt Colson's *confined development* and *silhouetting* have a similar cause, for they do not occur if the protecting plate be in intimate contact with the gelatin of the developing plate. The *silhouetting* which is often seen at the boundary between a light and a shadow on the plate, is probably due to radiation, for it does not occur with paper, but only with glass.—J. T. D.

*Over-exposed Plates, Impt of.* Mercier. Comptes Rend. **126**, [21], 1500.

The over-exposed plate is plunged for two minutes into a $2\frac{1}{2}$ per cent. solution of tartar emetic, allowed to dry, and then developed with hydroquinone. Even very badly over-exposed plates can be saved in this manner. The coating of tartar emetic may be effected either before or after exposure. Other antimony or arsenic salts of organic acids may be used instead of tartar emetic.

Reducing developers (amidol, pyrogallol, &c.), exposed to the air in 1 per cent. solution, then run over and allowed to dry on the plate in the same manner, permit of very clear images being obtained, though the plate may have been either under- or over-exposed. Amidol in particular, used in this manner, without previous oxidation by air, hastens the development by hydroquinone or pyrogallol, deepening the shadows without diminishing the clearness of the image. None of these substances produce these effects if added to the developer.—J. T. D.

*Photographic Paper.* Papier Zeit. 1898, **23**, [36], 1319.

*See under* XIX., *page* 688.

### PATENT.

*Photographic Solutions, as used for Toning and Fixing Prints, Impts. in.* A. E. Wade, London. Eng. Pat. 19,206, Aug. 19, 1897.

The patentee claims a solution of gold chloride, lead nitrate, potassium nitrate, sodium thiosulphate, flowers of sulphur, and lime in the proper proportions, in water which has been saturated with the smoke of coal or other carbonaceous fuel. —A. C. W.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Trimethyl Carbinol: Combination with Nitrate of Mercury.* G. Denigès. Bull. Soc. Chim. **19**, [9], 1898, 384—387.

The author, in the course of an investigation on the compounds formed from tertiary alcohols and salts of mercury, describes at length one he has obtained from trimethyl carbinol and nitrate of mercury by dissolving 20 grms. of mercuric oxide in 100 c.c. of water and 40 c.c. of pure nitric acid, then pouring the solution into 400 c.c. of water, filtering, and adding 2 c.c. of trimethyl carbinol. This solution is then boiled for some minutes, and, on cooling to 60°—65° C., becomes turbid, and deposits a copious orange precipitate when boiled. The precipitate is then filtered off, washed, and dried on a porous plate over sulphuric acid in the dark, its weight being about eight times that of the tertiary alcohol. It gives, on analysis, numbers corresponding to the formula $C_4H_8Hg_3(NO_3)_2$, and explodes when struck or when heated to about 80° C. It effervesces on treating with hydrochloric acid, giving off dimethylethylene, the residue consisting of a mixture of mercurous and mercuric chlorides. It is not attacked by caustic alkalis in the cold, but on boiling, it yields a white precipitate, which, after washing, dissolves in hydrochloric acid like a mixture of mercurous and mercuric oxides. The method can be employed for the detection of small quantities of trimethyl carbinol and of dimethyl ethylene. In order to test for isobutyl alcohol, the substance is heated with its own weight of sulphuric acid and 60 per cent. of potassium sulphate, the gas evolved being passed into hot mercuric nitrate; or else 2 drops of isobutyl alcohol, 1 drop of sulphuric acid, and 0·05—1 c.c. of water are heated to boiling, and the solution is added to twice its volume of the hot mercuric nitrate solution.—T. A. L.

*Red Phosphorus, Action of Sodammonium in Excess, on.* C. Hugot. Comptes Rend. 1898, **126**, [24], 1719—1722.

The action of phosphorus in excess on sodammonium has already been investigated (Comptes Rend. **121**, 206). By the action of red phosphorus on an excess of sodammonium dissolved in liquefied ammonia, sodamide and a yellow substance, $P_2H_3Na_3$, are formed. The latter, when treated with acids or water, evolves hydrogen phosphide, and when heated in a vacuum at about 100° C., also gives off this gas mixed with a little hydrogen; from 150°—200° the proportion of hydrogen phosphide decreases, and at 200°—300° only hydrogen is obtained. When only a slight excess of sodammonium is used, the products are difficult to isolate but undoubtedly contain Joannis' "phosphidure de sodium" (Comptes Rend. **119**, 557), $PH_2Na$.—E. W. W.

## XXIII.—ANALYTICAL CHEMISTRY.

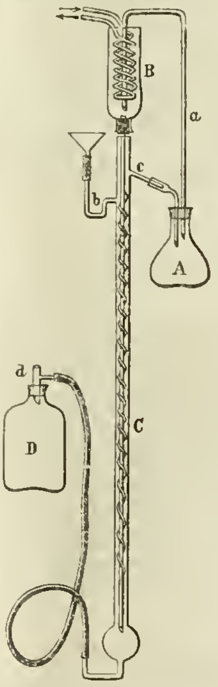
### APPARATUS, ETC.

*Extraction of large Quantities of Liquid by Ether, An Apparatus for the.* H. Malfatti. Zeit. Anal. Chem. 1898, **37**, [6], 374.

The apparatus has been in use for some time past in Loebisch's medico-chemical laboratory.

The flask A, which contains the extracting solvent (*e.g.*, ether), is connected by a fairly wide tube *a* with the condensing vessel B. It is advisable to draw a rubber tube over *a*, in order to prevent this acting as an air-condenser. In B there is a condensing worm. The tube connected to B leads to the bottom of the extraction vessel. This latter consists of a tube C, about 1 cm. in diameter, blown to a bulb at the lower end, then drawn out so as to connect by means of a rubber tube with the vessel D. On to the upper end of C two lateral tubes are fused; the lower of these, *b*, ends in a small funnel and the higher *c* conducts back to A.

As the ether rises in C it meets a stream of the liquid to be extracted, which drops through *b*. The extracted liquid fills the bottom of C and flows over through the ┬-tube *d* into the receiver D, whilst the ether, which condenses in B, falls to the bottom of C, then rises through C, and the ethereal solution returns through *c* to A.



In order that the ether rising in C should be brought into close contact with the liquid to be extracted, a thick spiral of several fat-extracted woollen threads loosely twisted together is wound round the tube leading from B to the lower part of C. The windings of the spiral must not be allowed to touch each other, and it must be renewed after having been used several times. This spiral cannot be used in cases where the liquid to be extracted has a specific action on wool (*e.g.*, colouring matters), and should then be replaced by a spiral of glass thread.

Before starting the extraction some of the liquor to be extracted, or water, must be introduced into C to serve as a trap.

During the extraction, fresh ether must be put into A, as some is lost by being dissolved by the liquid, and is carried to D.

The reflux-tube entering A, from *c* must be narrow and should contain a loose cotton-wool plug wrapped round with platinum wire to prevent the ether vapour from passing into C through *c*.

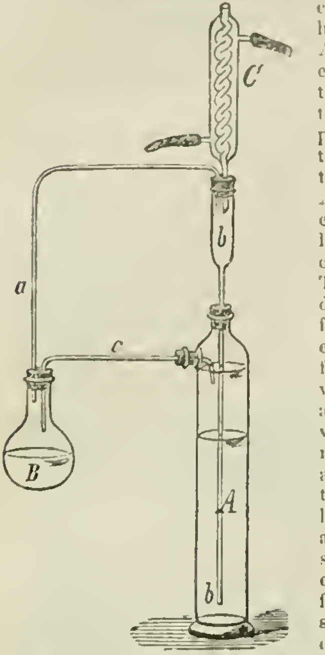
The efficiency of the apparatus is shown by the fact that by a single extraction of the urine collected during a period of 24 hours, after taking 5 mgrms. of strychnine, and with no other preliminary treatment than the addition of caustic potash, at least half of this alkaloid soluble with such difficulty, was recovered by the ether.

The apparatus is recommended for the extraction of urine, as there is a tendency in other extractors to form an emulsion, which can only take place in this one if the liquid be allowed to flow through *b* too fast.—J. McC.

*Extraction of Liquids, An Apparatus for the.* O. Foerster. Chem. Zeit. 1898, **22**, [42], 421—422.

This apparatus, which was designed especially for the extraction of cholesterol, &c. from soap solutions, consists of three parts: a cylinder, A, with a side tubulure, and holding about 1 litre; a boiling flask, B, of about 200—300 c.c. capacity; and a condenser, C. In practice, the soap solution (about 600 c.c.) is shaken with about 300 c.c. of ether in the cylinder, previously disconnected and stoppered

After standing for some time for the layers to separate, the apparatus is fitted together as illustrated, and the flask B, containing ether, is heated on the water-bath. As the evaporated ether condenses in C, it falls to the bottom of A through the funnel *b*, and passing upwards through the soap solution, returns to B through the tube *c*. After a long-continued extraction, an aqueous layer of soap solution collects below the ether. This must be removed day by day to prevent frothing. The ethereal extract is washed free from dissolved soap with water, the ether evaporated, and the residue weighed. By obvious modifications, the apparatus can be adapted to the hot-extraction of liquids, but this is not advisable in the case of soap solutions, since emulsions are readily formed, and so much soap solution is carried over by the ether that the flask B becomes inconveniently full. The author states that with four hours' extraction in the cold in this apparatus, he recovered 0·9997 grm. of cholesterol from 1 grm. dissolved in a solution of a soap of stearic acid.—C. A. M.



## PATENT.

*Taking Samples of Pulverised and Granulated Materials, Improved Apparatus for.* C. Geissler, Stassfurt, Prussia. Eng. Pat. 7021, March 23, 1898.

A series of hollow drums, with longitudinal slots therein, are arranged vertically above one another, and connected by a system of chain gearing driven in any suitable manner. Material is fed into a hopper above, and as the slot of the topmost drum passes the feeding aperture, some of the material passes into the drum, while during the remainder of the revolution material falls into a side shoot. The next lowest drum is fed from the first acting as a hopper, and so on through the series, the overplus material being discharged into the side shoot, while the sample below is collected in a suitable receiver. The slot in each drum is adjustable, and in the arrangement shown and described, the revolutions and sizes of the drums increase regularly from the lowest to the highest.—E. G. C.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Carbon Monoxide, Determination of Traces of, in Air, in the Presence of Hydrocarbons.* A. Gautier. Comptes Rend. **126**, [19], 1299—1305.

The author now determines (see this Journal, 1898, 603) the carbon monoxide in air by absorbing by hot copper the iodine produced when the air is passed over iodine pentoxide at 100° C. The air (10 to 100 litres) is filtered through cotton wool or glass wool, passed over caustic potash solution to absorb carbon dioxide and then over caustic baryta and phosphorus pentoxide; it then passes through two tubes containing respectively iodine pentoxide and finely divided copper which has been reduced from the oxide by hydrogen at a low temperature and allowed to cool in a current of carbon dioxide. These two tubes are connected without india rubber, are thoroughly dried in a current of hot air before being weighed, and are placed in an air-bath at 100—105° C. The increase in weight of the tube containing the copper gives the amount of iodine set free from the iodine pentoxide, whence the amount of carbon monoxide can be calculated. Experiments with known mixtures showed that the method is extremely accurate, and proved that no greater amount of carbon monoxide or of iodine could escape reaction and collection than corresponded with 0·01 mgrm. of iodine or 0·0011 c.c. of carbon monoxide in 10 litres of air, or $\frac{1}{5000000}$ of the total volume of the air. Experiments on the air of Paris by the method gave amounts of carbon monoxide varying from zero to 9 parts per million, and the air of the author's laboratory gave 12 parts per million. The author is making an extended series of similar observations. Certain of the hydrocarbons of the alkylene and acetylene series are more or less completely oxidised by iodine pentoxide under the circumstances of this method. To determine carbon monoxide in presence of these the method is modified as follows:—The iodine pentoxide tube and the copper tube are both weighed after having been thoroughly dried by air at 220° C., and are followed by a tube of phosphorus pentoxide to collect the water formed, and by a system of three tubes containing phosphorus pentoxide, baryta, and phosphorus pentoxide, to collect the carbon dioxide formed. (All the weighings are done by the method of vibrations, and each tube is weighed against a tube of similar size and form to neutralise barometric or hygrometric variations.) At least 200 litres of air are passed through the apparatus, and the loss of weight of the iodine pentoxide, as well as the increases of the copper tube, the water collector, and the carbon dioxide collector are determined. The total oxygen in the water and carbon dioxide is calculated; this is greater than the amount lost by the iodine pentoxide (determined, of course, by the difference between the total loss of that tube and the gain of the copper tube) by the amount contained originally in the carbon monoxide present, which can thus be calculated. —J. T. D.

*Sulphuric Acid, Volumetric Estimation of Combined.* M. Reuter. Chem. Zeit. 1898, **22**, 357.

The author considers that of all the methods proposed for the volumetric estimation of combined sulphuric acid, Andrews' process (Amer. Chem. J. 1880, **2**, 567) is the best; and he finds that, if it be carried out exactly as described below, its accuracy leaves nothing to be desired; it needs but one standard solution (decinormal thiosulphate); and it is far more rapid than any gravimetric method.

3 or 4 grms. of pure precipitated barium chromate are dissolved in 30 c.c. of strong hydrochloric acid, and the whole is diluted to 1 litre. The liquid to be tested, which should contain about 0·07 grm. of $SO_3$ as an alkali sulphate, is mixed at the boiling point with an excess (150 c.c.) of the chromate solution; the acid is neutralised with pure powdered chalk, and the precipitate is removed by filtration. After thorough cooling, the filtrate is acidified with 5 c.c. (not more) of strong HCl, 20 c.c. of a 10 per cent. solution of potassium iodide are added, and the liquid is allowed to rest for five minutes in a covered beaker and in an atmosphere of carbonic acid, until the chromic acid is entirely reduced. Finally, it is diluted to 1 or $1\frac{1}{2}$ litres, and titrated quickly with thiosulphate; three atoms of iodine corresponding to 1 molecule of $SO_4$.—F. H. L.

*Perchlorate [or Total Chlorine] in Sodium Nitrate (Chili Saltpetre), Estimation of.* O. Foerster. Chem. Zeit. 1898, **22**, 357.

10 grms. of the sample suspected of containing perchlorate are mixed with an equal weight of dry sodium carbonate (free from chlorine), and heated in a large covered crucible of porcelain or platinum, for about 10 minutes, until the melt is quite liquid and tranquil. There is no danger of losing chlorine by volatilisation and no necessity to employ a particular temperature; the fused mass does not creep up the walls of the vessel, and the residue is easily soluble. When cold, it is dissolved in excess of nitric acid, and the total chlorine determined in the usual manner. The examples quoted show that the process is perfectly accurate in the presence of perchlorate alone; whilst if the nitrate contain chloride and chlorate as well, the whole of the chlorine is recovered satisfactorily. (See also this Journal, 1898, 275.)—F. H. L.

### *Chlorine, Bromine, and Iodine, A Quantitative Electrolytic Separation of.* H. Speketer. Zeits. f. Elektrochem. **4**, 1898, [23], 539—542.

THE E.M.Fs. necessary for the electrolytic decomposition of the halogen acids are respectively: HCl = 1·31 volt, HBr = 0·94 volt. and HI = 0·52 volt. An attempt to quantitatively separate the halogens by successively increasing the E.M.F. from the lowest required value upwards did not give satisfactory results. Iodine, for example, could not be completely separated from the other halogens, even although the E.M.F. was raised to 1·2 volt, and the iodine which was formed removed by means of carbon bisulphide. The cause of failure is attributed to the diffusion of the free iodine towards the cathode, where hydriodic acid is regenerated.

The quantitative separation may be effected, however, if a platinum cathode and a perfectly pure silver anode are employed. Under these circumstances the E.M.F. of decomposition can be calculated from Nernst's formula. The electrolysis is conducted in a N—$H_2SO_4$ solution, when the iodine is precipitated (as Ag I) on the silver anode below 0·13 volt., the bromine below 0·35 volt., whilst the remaining chlorine is best determined in the ordinary way by Volhard's method. The most convenient source of current is a Gülcher's thermopile which is short-circuited through a convenient resistance, the requisite E.M.F. being obtained by tapping the resistance at suitable points. The vessel in which the electrolysis is conducted is a tall narrow cylinder provided with a platinum cathode and a silver gauze anode made of pure electrolytic silver. During the electrolysis, a current of hydrogen is continuously passed through the electrolyte in order to exclude air or oxygen from the cathode. If this precaution be neglected, bromide and chloride of silver will be precipitated on the anode along with the iodide, for silver in a solution of these halogen acids liberates hydrogen on the cathode, and if this hydrogen be continually removed by the oxygen of the air, the halides of silver will be continuously deposited on the anode even without the application of any electromotive force. A sensitive galvanometer introduced into the circuit indicates when any one halogen is completely removed from the solution. The precipitate on the anode is then washed with water and dried at 120° C. The following test analyses show the accuracy of the method :—

I. *Separation of Iodine from Chlorine. E.M.F.* 0·13 *volt.*

| Iodine used. | Iodine found. | Iodine used. | Iodine found. |
|---|---|---|---|
| Grm. | Grm. | Grm. | Grm. |
| 0·29087 | 0·2892 | 0·1588 | 0·1598 |
| 0·0481 | 0·0489 | 0·2290 | 0·2265 |
| 0·3807 | 0·3773 | 0·15457 | 0·1532 |
| 0·2394 | 0·2386 | | |

II. *Separation of Bromine from Chlorine. E.M.F.* 0·35 *volt.*

| Bromine used. | Bromine found. | Bromine used. | Bromine found. |
|---|---|---|---|
| Grm. | Grm. | Grm. | Grm. |
| 0·19437 | 0·1940 | 0·19625 | 0·1958 |
| 0·1062 | 0·1041 | 0·19047 | 0·1883 |
| 0·27354 | 0·2736 | 0·18994 | 0·1906 |
| 0·2122 | 0·2111 | 0·18651 | 0·1836 |

III. *Separation of Iodine from Bromine. E.M.F.* 0·13 *volt.*

| Iodine used. | Iodine found. | Iodine used. | Iodine found. |
|---|---|---|---|
| Grm. | Grm. | Grm. | Grm. |
| 0·17066 | 0·1685 | 0·1078 | 0·1080 |
| 0·1636 | 0·1610 | 0·1235 | 0·1218 |
| 0·2029 | 0·2036 | 0·1356 | 0·1318 |

—J. S.

### *Gold Assay, A Semi-Electrolytic Process of.* A. Bock. Chem. Zeit. 1898, **22**, [36], 358.

IN the ordinary process of assaying by cupellation and parting, the former operation is accompanied with a loss of gold, while the latter is incompetent to remove the last traces of silver. When the whole assay is experimentally carried out on absolutely pure gold, mixed with absolutely pure silver, the final error is a constant one, and the proper allowance may be made. But in practical work other metals beside silver and lead are liable to be present (although in small amounts); and by their differences in specific gravity they cause the results to vary.

The author finds that if a plate of gold be placed as anode in an electrolytic bath filled with nitric acid of the specific gravity 1·2, opposite a platinum cathode, the liquid rapidly becomes opalescent and opaque. On interrupting the current, the electrolyte clears again, and a precipitate of metallic gold is deposited; but the cathode remains uncoated. The action is therefore mechanical in its nature: the anode is disintegrated by the evolution of oxygen and the current of electricity, without being actually dissolved. If, however, the cell contain water but be faintly acidified with nitric acid, the electrolyte does not become cloudy, the weight of the gold remains unchanged, whilst the silver and all other soluble metals in the anode (except lead) are deposited on the cathode or dissolved in the liquid. Bock's process of gold-assay depends on this phenomenon; and the following example, which shows its accuracy, also indicates the method of carrying it out in practice.

Two pieces of fine gold, weighing 500 mgrms., were each melted with 1,300 mgrms. of silver in previously warmed graphite crucibles. After cooling, the buttons were hammered, rolled, and melted once more to ensure uniformity of composition—the yield in each case was exactly 1,800 mgrms. of alloy. The second buttons were rolled out, parted in the usual manner, then immersed in a bath composed approximately of 1 vol. of nitric acid (sp. gr. 1·2) diluted with 6 vols. of water, and submitted to an electric current, the strength of which was "about that employed in copper analysis." After 10 minutes, the platinum vessel containing the rolls of gold, was rinsed out with water, placed in boiling water for a few minutes, again washed, and the gold dried and ignited in an earthen crucible. The total weight of metal recovered was exactly 1,000 mgrms.

When the legal alloy of 900° fineness is being examined, not more than 900 mgrms. of silver should be added to each 500 mgrms., lest the pieces of gold become too porous and break; but the process is generally as accurate as in the case of fine metal. In melting this alloy, it is well to use nearly new plumbago crucibles, so that the carbonic oxide produced, may prevent oxidation of the copper.

As lead is not dissolved by the above process, when this metal occurs in the pieces of gold, they must be treated in a fresh bath of nitric acid with the direction of the current reversed; but under no circumstances must the change of polarity be made in the one platinum dish.

The new method occupies practically the same time as the old one; it is, taken altogether, a little cheaper, and it forms an easier way of preparing samples of pure gold to act as standards.—F. H. L.

### *Lead in Ores, Analytical Determination of.* F. Moldenbauer. Chem. Zeit. 1898, **22**, [26], 256—257.

IN crucible tests the results of two observers often differ, and it is necessary to have recourse to a more exact method to decide which is correct. For this purpose, the author proposes the following process :—

Of ores containing under 30 per cent. of lead, not less than 2 grms. are taken. The finely-powdered ore is boiled with hydrochloric acid until all action ceases. If much antimony be present, it is advisable to pour the whole into a porcelain dish and evaporate to dryness two or three times with strong hydrochloric acid so as to drive off all the antimony. In the alternative case, nitric acid is added drop by drop, with final evaporation to dryness, to complete the destruction of the sulphur compounds as quickly as possible. The residue is treated with excess of hydrochloric acid and 80 to 100 c.c. of water, heated to boiling, and filtered hot. The filter is washed free from lead chloride with hot water containing hydrochloric acid. The filtered solution is again heated to bring the crystallised lead chloride into solution, saturated with ammonia almost to precipitation point, sulphuretted hydrogen passed through, and then the liquid cooled as quickly as possible. The precipitate contains black lead sulphide and other precipitable metals that may be present. It is filtered and washed with ammonium sulphide, dried partly on the filter-paper and finally in a porcelain crucible, and converted into sulphate with the aid of concentrated nitric acid, heating to dryness over a small flame. The treatment with nitric acid is repeated and it is then boiled for a moment with concentrated sulphuric acid. After cooling, water is added and the whole boiled, to dissolve soluble matters. After cooling again, the soluble matters are filtered off from the pure lead sulphate, which is dried and weighed. After weighing it may, if necessary, be dissolved in sodium potassium tartrate and ammonia, so as to be certain of its purity. According to Merck, on boiling the lead sulphate, bismuth would pass over into the precipitate. It is well to bear this in mind, when, if necessary, the requisite modification may be made in the process.

—L. J. de W.

### *Nickel Assay, New Caledonia.* H. W. Edwards. Eng. and Mining J., May 28, 1898.

The following method, which is used in this island, was invented by Mr. Moore, the official analyst to the Government of the Colony:—Two solutions are prepared: A., 11 grms. of 98 per cent. commercial cyanide of potassium 0·5 grm. of silver nitrate, and 1 litre of distilled water; and B., 50 grms. of citric acid, 38 grms. (approximately) of sodium carbonate, 7·5 grms. of potassium iodide, and 500 c.c. of distilled water; 35 grms. of the sodium carbonate are first added, and then the remainder, decigram by decigram, until neutrality is attained, before adding the potassium iodide. It is important that solution B. be either absolutely neutral or only very slightly alkaline. 2·5 grms. of the ore (after drying at 100° C.) are placed in a 250-c.c. flask, dissolved in 20 c.c. of hydrochloric acid, and the solution made up to 250 c.c. with water, and then well agitated. The insoluble silica, &c., is then filtered off, and to 50 c.c. of the filtrate, 10 c.c. of solution B. are added, then dilute ammonia in slight excess till the characteristic blue coloration is produced, and the solution is cooled. The liquid is then titrated with solution A. added gradually and with stirring. A white cloudy precipitate forms at first, but disappears on the addition of the last drop of solution A. A standard solution of pure nickel is prepared and titrated in the same manner as the ore.

The process takes about 30 minutes, and the results, although usually a little too high, are very concordant. The method is not applicable to ores containing large quantities of iron, manganese, or cobalt, 25 per cent. being the limit for iron and manganese, and 1 per cent. for cobalt.—A. S.

### *Superphosphates, Determination of Phosphoric Acid in.* L. Vignon. Comptes Rend. **126**, [21], 1522–1523.

The usual plan with superphosphates is to exhaust successively with water and with ammonium citrate, and to determine separately the phosphoric acid in each solution. The author has found that when the amount of phosphoric acid soluble in citrate is small, it comes down very slowly, and the precipitation is far from complete, even after many hours.

The following figures were obtained from a bone superphosphate (I.) and two mineral superphosphates (II. and III.)

It is easy to understand that the phosphoric acid in the citrate solution may come down readily and completely when the solutions are mixed and it is in presence of the greater amount in the water solution, and yet precipitate with difficulty when the dilute solution is alone. The author's recommendation is to determine separately the phosphoric acid in the water solution, to determine the total amount in the mixed solutions from a second sample, and to take the phosphoric acid soluble in citrate by difference.

| — | I. | II. | III. |
|---|---|---|---|
| *a.* Solutions separately precipitated, allowed to stand eight hours: | | | |
| Phosphoric acid soluble in water.. | 14·32 | 14·13 | 10·20 |
| " " citrate. | 0·77 | 0·27 | 3·10 |
| Total .................... | 15·09 | 14·40 | 13·30 |
| *b.* Solutions separately precipitated, allowed to stand 15 hours: | | | |
| Phosphoric acid soluble in water.. | 14·32 | 14·13 | 10·20 |
| " " citrate. | 1·08 | 0·45 | 3·18 |
| Total .................... | 15·40 | 14·58 | 13·38 |
| *c.* Solutions mixed and precipitated together: | | | |
| Phosphoric acid, total ............ | 17·79 | 14·94 | 13·91 |
| " " soluble in water (*a.* or *b.*). | 14·32 | 14·13 | 10·20 |
| " " soluble in citrate (difference). | 3·47 | 0·81 | 3·71 |

—J. T. D.

### *Standard Acid Solution by Direct Absorption of Hydrogen Chloride, Preparation of a.* G. T. Moody. Proc. Chem. Soc. 1898, [197], 149–150.

A very rapid and accurate method of preparing a standard acid solution consists in absorbing hydrogen chloride in water, determining the resulting increase in weight, and subsequently diluting to a suitable bulk. The absorption is conveniently carried out in a conical glass flask, having a capacity of about 80 c.c., and closed by a glass stopper. Through the stopper pass two tubes, one of which reaches nearly to the bottom of the flask and serves for delivery of the gas. From 2 to 4 grms. of the gas may be absorbed in 40 c.c. of water in three minutes, and if the necessary apparatus for generating hydrogen chloride be kept fitted up, a standard acid solution can be prepared in less than 15 minutes.

## *ORGANIC CHEMISTRY—QUALITATIVE.*

### *Seal Oil in Cod-Liver Oil, Detection of.* E. Dowzard. Pharm. J. 1898, [1458], 532.

The author considers that the presence of seal oil in cod-liver oil can be detected with certainty by means of Amagat and Jean's oleorefractometer. The following figures were obtained:—

| Cod-Liver Oil. | | | Pale Seal Oil. |
|---|---|---|---|
| English. | Newfoundland. | Norwegian. | |
| + 44 | + 44·5 | + 45·0 | + 32·0 |
| .. | + 44·0 | + 43·5 | + 32·0 |
| .. | + 45·0 | + 44·0 | + 32·5 |
| .. | + 44·0 | + 43·5 | .. |
| .. | + 44·5 | + 45·0 | .. |
| .. | + 44·0 | + 44·0 | .. |
| .. | + 44·5 | .. | .. |

The two samples having the number + 43·5 may be disposed of as probably being impure, and thus the maximum for pure cod-liver oil may be taken as + 45, and the minimum as + 44. The following numbers were obtained with mixtures of the two oils:—

| Cod-Liver Oil. | Seal Oil. | Refractometer Number at 22° C. |
|---|---|---|
| 100 | 0 | + 44·50 |
| 90 | 10 | + 43·25 |
| 80 | 20 | + 42·00 |
| 70 | 30 | + 40·75 |
| 60 | 40 | + 39·50 |
| 50 | 50 | + 38·25 |
| 40 | 60 | + 37·00 |
| 30 | 70 | + 35·75 |
| 20 | 80 | + 34·50 |
| 10 | 90 | + 33·25 |
| 0 | 100 | + 32·00 |

In carrying out the test, the oil must first be agitated with alcohol (sp. gr. 0·800), the mixture heated to 30° C., then allowed to separate, and the oil finally dried at 110° C.

The author considers it would be as well to take +43·5 as the minimum figure, until more pure samples of cod-liver oil have been examined.—A. S.

### *Butter and Lard, Detection of Foreign Fats in.* C. B. Cochran. Rev. Intern. Falsific. **11**, 49; Chem. Centr. 1898, **1**, [19], 1069.

Two c.c. of the melted fat are placed in a burette, 22 c.c. of amyl alcohol added, the mixture warmed to 38° C., then allowed to cool to 16°—17° C. and kept for 3—4 hours at this temperature. For butter and margarin, only 8 c.c. of amyl alcohol are used per 2 c.c. of fat. It is stated that a characteristic precipitate for each kind of fat is formed. This is filtered off, dissolved in ether, the ethereal solution allowed to evaporate in a tube closed with a piece of cotton-wool, and the crystalline residue examined microscopically. Fats rich in stearin yield much larger precipitates than others; with oleostearin, nearly the whole of the tube is filled with precipitate; beef tallow yields a precipitate of 16 c.c., whilst lard gives only a precipitate of 4 c.c.—A. S.

### *Formaldehyde in Articles of Food, Detection of.* E. Rimini. Ann. di Farmacol. 1898, 97—101; Chem. Centr. 1898, **1**, [21], 1152.

The author gives two extremely sensitive colour reactions, which are characteristic for formaldehyde only, and are specially suitable for the detection of the latter in milk. (1.) 15 c.c. of a very dilute formaldehyde solution are treated with 1 c.c. of a dilute solution of phenylhydrazine hydrochloride, then with a few drops of freshly-prepared sodium nitroprusside solution, and finally with concentrated caustic soda solution. A blue colour is formed, which, after a long time changes to red. This reaction is capable of indicating formaldehyde in milk, even in a dilution of 1 in 30,000. (2.) The second reaction is obtained in a similar manner, except that iron chloride is used instead of sodium nitroprusside, followed by concentrated hydrochloric acid, instead of caustic soda. This gives a red colour, which changes, after some time, to orange yellow. —A. S.

### *Phenylhydrazine, Louis Simon's Reaction for, Applicability of.* E. Rimini. Ann. di Farmacol. 1898, 102—103; Chem. Centr. 1898, **1**, [21], 1152.

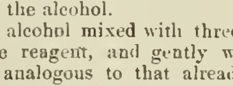
Simon's reaction of phenylhydrazine (this Journal, 1898, 494), with sodium nitroprusside and caustic soda in presence of trimethylamine greatly resembles the reaction obtained by the author by using formaldehyde in place of trimethylamine (see preceding abstract). The latter now shows that preparations of trimethylamine which give Simon's reaction, when acidified and distilled, yield a distillate which also gives the reaction; and also that perfectly pure trimethylamine, prepared from one of its salts, does not give the reaction. The reaction of the phenylhydrazine thus depends not on the presence of trimethylamine, but on that of formaldehyde, which may be present as impurity.—A. S.

### *Colouring Matters in Milk, Detection of Added, especially "Orleans."* J. Froidevaux. Ann. Chim. Anal. Appl. **3**, 110—114; Chem. Centr. 1898, **1**, [19], 1068.

It has been customary for some months in Paris, to add small quantities of yellow colouring matters, in order to veil the bluish colour of mixed and thin samples of milk. Such milk usually shows a normal white colour; if only a glimmer of another colour is apparent, colouring matters should be tested for. "Orleans" gives to the milk a flesh colour; saffron, a faint orange-yellow; turmeric, a faint greenish-yellow; "carrot-juice," an orange-yellow; and Orange III. Poirier, a flesh colour. On adding 1 c.c. of hydrochloric acid to 20 c.c. of the milk, the intensity of the coloration is slightly increased if Orange III. be present, whilst if vegetable colouring matters be present, the liquid acquires a rose colour. If turmeric be present, the sample turns yellowish-brown on addition of ammonia. It is stated that if the milk be coagulated, the coloration may be recognised still better, since the vegetable colouring matters are completely fixed by formation of lakes with casein; "Orleans" giving a flesh red colour; saffron, yellow; turmeric, greenish-yellow; and "carrot juice," a golden yellow. "Orange III." does not alter the white colour of the coagulum, and only gives a yellowish colour to the serum. The following method is recommended for the detection of "Orleans" and saffron:—50 c.c. of the milk are shaken cautiously with an equal volume of Adam's solution, which dissolves all the fat, but only traces of the colouring matter. The lower layer is run off into a small beaker, and a strip of filter paper (or bleached linen) is immersed in the liquid for four days. The strip is then pressed between filter paper and cut into two pieces, which are dipped respectively into concentrated sulphuric acid and into 2 per cent. sulphuric acid. If turmeric be present, the paper, originally coloured a reddish-orange, acquires a blue colour on treatment with concentrated sulphuric acid, and a rose colour with the 2 per cent. acid. If the colouring matter present be saffron, the strip is first coloured yellow, changing to a faint orange-yellow with 2 per cent. sulphuric acid.—A. S.

### *Tertiary Alcohols, A New Reaction of.* G. Denigès. Comptes Rend. 1898, **126**, [18], 1277—1279.

In a former communication (Comptes Rend. **126**, 1145) the author described a method of detecting hydrocarbons of the ethylene series by means of a reagent consisting of: mercuric oxide, 50 grms.; sulphuric acid, 200 c.c.; and water, 1,000 c.c. This test can also be nsed for the detection of all compounds which readily yield those hydrocarbons, and especially tertiary alcohols with which the mercuric sulphate acts both as a dehydrating agent liberating the hydrocarbon, and also as a reagent combining with it when formed. When two or three drops of a tertiary alcohol are warmed with a few c.c. of the mercuric reagent, a yellow precipitate is rapidly produced, the tint varying with the particular alcohol tested. The author describes the behaviour of tertiary butylic and amylic alcohols as representative examples. With the former a yellow compound is obtained, which, when washed, and dried in the dark on a porous tile over sulphuric acid, has a composition corresponding to the formula—



$$\left(SO_4 \begin{matrix} \diagup Hg \diagdown \\ \diagdown Hg \diagup \end{matrix} O\right)_3 . C_4H_3$$

which is also the formula for the substance yielded by dissymetric dimethylethylene, the corresponding olefiant hydrocarbon. It is soluble in hydrochloric acid with effervescence, yielding butylene and mercuric chloride. On boiling the precipitate in its mother liquid for 10 minutes, the colour gradually disappears, and finally mercurous sulphate is produced with oxidation of the butylene residue. The test is very sensitive, and makes possible the detection of 0·0001 grm. of the alcohol.

Tertiary amylic alcohol mixed with three to four times its volume of the reagent, and gently warmed, gives a yellow precipitate analogous to that already described and identical with that given by the corresponding ethenic hydrocarbon. On boiling, this compound is immediately decomposed, giving white crystalline mercurous sulphate, which is rapidly reduced to metallic mercury. The three stages of the reaction can be observed well by heating 2 c.c. of the mercuric reagent to the boiling point and then adding a drop of the alcohol.

There is a characteristic difference between the behaviour of pental and of tertiary amylic alcohol with the reagent. On adding a drop of the former to 2 c.c. of the reagent in the cold, there is an immediate yellow coloration, followed by the gradual formation of a precipitate of which there is an abundant deposit after 10 minutes. Tertiary amylic alcohol tested in this way in the cold scarcely shows any change, though on warming it gives the three stages of the reaction already described. When a drop of the liquid under examination is added to 2 c.c. of the boiling reagent, a yellow precipitate is obtained, which does not alter on standing, in the case of pental, whilst with tertiary amylic alcohol the precipitate becomes white and crystalline, and finally grey.

The other tertiary alcohols give similar compounds with the reagent, always provided they are capable of yielding hydrocarbons of the ethylene series on dehydration. Primary and secondary alcohols do not form these compounds. Isopropylic alcohol, however, which is readily dehydrated with the formation of propylene, gives a precipitate with mercuric sulphate, but with very much less readiness than the tertiary alcohols. The esters of tertiary alcohols also give a yellow precipitate with the reagent, and the test is very characteristic in the case of the tertiary amylic nitrite employed in medicine.—C. A. M.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Paranitraniline Red, Solvents for.* C. Kurz. Rev. Gén. des Mat. Col., 1898, **2**, 188—189.

Paranitraniline Red is readily dissolved by amido compounds and phenols, from which it may be obtained in long, bluish-red, needle-shaped crystals.

The solvent action may be applied:—

I. To render Paranitraniline Red, as commonly produced on cotton, faster to rubbing.

II. To estimate the amount of the dyestuff on the fibre.

For the first of these purposes, the dyed or printed fabric is washed, soured, washed and soaped as usual, and is then boiled for 20—30 minutes in a 0·02 per cent. solution of phenol, after which it is washed and dried.

The determination of the azo dyestuff present on the fibre is accomplished by means of a Soxhlet apparatus. A square decimetre of tissue is taken, dried at 80° C., and weighed. It is then placed in the extraction tube and extracted with aniline (50 c.c.) When the azo compound has been completely removed, the tissue is washed with alcohol, then with very dilute hydrochloric acid or ether, and is then again dried at 80° C. and weighed.

The amount of the red on different kinds of tissues varies per square decimetre from 0·031 grm., in the case of a fine lawn tissue, to 0·108 grm., in that of a heavy flannelette, raised on both sides.

The claret dyestuff from α-naphthylamine and β-naphthol is also soluble in phenol and aniline, and can be determined in a manner similar to that described. The results obtained are, however, less accurate, as a trace of dyestuff always remains unacted upon by the solvent.—E. B.

### *Tartaric Acid, Analysis of Raw Material containing.* Chem. Fabrik vorm. Goldenberg, Geromont, and Co. Zeits. anal. Chem. 1898, **37**, [6], 382. (See this Journal, 1898, 609.)

Details are given of the hydrochloric acid method of determination of total tartaric acid, which differs somewhat from that formerly proposed (Zeits. Anal. Chem. **37**, 312).
—J. McC.

### *Hubl's Method of Iodine Absorption. The Reactions in.* J. J. A. Wijs. Zeits. angew. Chem. 1898, [13], 291—297.

Ephraim (Zeits. angew. Chem. 1895, 254) attributed the additive power of Hubl's solution to the presence of iodine monochloride, and described a method of determining the halogen absorption of fats by means of a solution of that substance. The author states that this formation of iodine chloride takes place in accordance with the equation:—

(1.) $HgCl_2 + 4I = HgI_2 + 2ICl$, though possibly mercury chloriodide $HgClI$ is formed as an intermediate product. That there is an equilibrium in the reaction between the mercuric chloride and the iodine, is shown by the fact that it is possible to prepare a solution giving the same results as that of Hubl by dissolving mercuric iodide in an alcoholic solution of iodine chloride.

The iodine chloride thus formed, reacts with the water in the alcohol in the solution, yielding hypoiodous and hydrochloric acids.

(2.) $ICl + H_2O = HCl + HIO$.

The IO- ion of hypoiodous acid appears to be so unstable that it is only present in consequence of its being continually re-formed as fast as it undergoes decomposition. In Hübl's solution it is slowly transformed into iodic acid and free iodine, and from these, in the presence of hydrochloric acid in alcoholic solution, iodine monochloride is again produced, so that there is a complicated equilibrium.

On adding the potassium iodide and water to the solution before titration, the following changes occur:—

(3.) $ICl + KI = KCl + 2I$.
(4.) $HCl + HIO + KI = KCl + H_2O + 2I$.
(5.) $5HCl + HIO_3 + 5KI = 5KCl + 3H_2O + 6I$.

Theoretically in a blank determination the whole of the original iodine should be found on titration, and the solution should contain no free acid, but in practice this is never the case. Schweitzer and Lungwitz (this Journal, 1895, 130) and Waller (Chem. Zeit. 1895, 1831) have shown that the iodine which does not appear on titration is equivalent to the acid formed, and that the sum of the two equivalents is a constant. The older the solution the less the iodine titre and the greater the amount of acid. The author explains this change which occurs on keeping the solution, by the fact that hypoiodous acid oxidises the alcohol to aldehyde, as in the equation—

$$2HIO + C_2H_6O = 2I + 2H_2O + C_2H_4O,$$

and that this causes a disturbance in the equivalence of the solution, so that the quantity of hypoiodous acid re-formed is less than that decomposed, whilst the amount of hydrochloric acid becomes greater. The free iodine liberated, reacts for the most part with the excess of mercuric chloride in the solution as in equation (1). For this reaction each atom of iodine requires $\frac{1}{4}$ molecule of mercuric chloride, whilst in a solution prepared in accordance with Hübl's directions there is more than $\frac{1}{2}$ a molecule. Ordinarily, each molecule of hypoiodous acid gives on titration, according to (4) 2 atoms of iodine, but when there is aldehyde formation, only 1 atom is liberated, and the titre is correspondingly smaller. On the other hand, for each molecule of reduced hypoiodous acid, a molecule of hydrochloric acid is set free, which is not neutralised as in equation (4), and which, therefore, leaves the liquid acid. This hydrochloric acid the author terms "excess acid." The stability of Hübl's solution is increased by keeping the concentration of hypoiodous acid in the solution as low as possible. This is effected in Welman's method (Pharm. Zeit. 1893, Ap. 8), in which alcohol is entirely, or for the most part, replaced by ether, ethyl acetate, or anhydrous acetic acid. The author also found that a solution prepared with absolute alcohol was more stable than the ordinary solution, in which he is confirmed by Waller (*loc. cit.*). The latter obtained the best results by an addition of strong hydrochloric acid, which acts not by combining with the water present, but by favouring the formation of iodine chloride, and preventing its decomposition as in equation (2).

Of the substances which are formed in the solution, the only ones which take part in the actual addition of halogen to the unsaturated body, are free iodine, iodine chloride, and hypoiodous acid. Of these, the first is so slowly absorbed, that it may be neglected in explaining the reaction. Iodine chloride was regarded by Ephraim (*loc. cit.*), and formerly by the author as being the chief additive body, but the latter has now come to the conclusion that hypoiodous acid must be regarded as the principal agent. He argues that, if iodine chloride were the substance, a solution prepared in accordance with Waller's suggestion (strong HCl), and, therefore, containing more iodine chloride, would act more quickly than Hubl's solution, whereas experiments have shown the contrary to be the case. On the other hand, by shaking an alcoholic solution of iodine with finely-divided mercuric oxide and filtering, the filtrate, according to Koene (Pogg. Ann. **66**, 302), contains mercuric iodide and hypoiodous acid, and with this solution correct iodine values were very rapidly obtained. A number of determinations of the iodine value of earthnut oil by means of Hubl's solution to which various substances had been added, showed that when these substances favoured the formation of hypoiodous acid (*e.g.*, iodine, mercuric chloride and water), the halogen addition took place more rapidly, whilst the converse was the case with substances which tended to retard its formation, such as hydrochloric acid and mercuric iodide.

In determinations of the iodine number there is invariably an increase in the "excess acid." The author considers that, besides the addition of halogen, there is a further reaction with hydrochloric acid, which in the case of oleic acid may be expressed thus—

$$CO_2H.C_{17}H_{33}I.OH + HCl = CO_2H.C_{17}H_{33}ICl + H_2O.$$

But this only occurs without further change in strongly acid solutions, like that of Waller, in which the amount of acid shows hardly any increase after the absorption. When solutions containing less acid are employed, there is a considerable increase in the "excess acid," this being accounted for by a splitting off of the hydrochloric acid, in accordance with the equation—

$$CO_2H.C_{17}H_{33}ICl = CO_2H.C_{17}H_{32}I + HCl.$$

The conclusions which the author has formed on this point are .

1. The separated acid is, *ceteris paribus*, proportional to the amount of absorption.

2. Under the same conditions different fats all yield about the same quantities of acid, proportional to the amount of fat taken. But with substances of different constitution, such as cholesterol, allyl alcohol, &c., different figures are obtained.

3. The greater the acidity of the solution, the smaller the amount of acid separated.

4. The splitting off of the hydrochloric acid is most thorough in liquids which most readily dissolve that acid. Thus it is greater in alcohol containing water than in chloroform, and in methylic alcohol than in ethylic alcohol.

Summarising the main reactions which occur in Hübl's solution, the author gives the following equations :—

1. In the preparation—

$$HgCl_2 + 4I = HgI_2 + 2ICl$$
$$ICl + H_2O = HCl + HIO.$$

2. On keeping the solution—

$$2HIO + C_2H_6O = 2I + 2H_2O + C_2H_4O.$$

3. In the absorption (oleic acid)—

$$CO_2H.C_{17}H_{33} + HIO = CO_2H.C_{17}H_{33}I.OH$$
$$CO_2H.C_{17}H_{33}I.OH + HCl = CO_2H.C_{17}H_{32}ICl + H_2O,$$

and in a part of the fat—

$$CO_2H.C_{17}H_{33}ICl = CO_2H.C_{17}H_{32}I + HCl.$$

—C. A. M.

### *Iodine Absorption, A New Method of Determining.*

J. J. A. Wijs. Ber. 1898, **31**, [6], 750—752.

In a previous communication (see previous abstract) the author gave his reasons for concluding that in the Hübl process the substance chiefly concerned in the addition is hypo-iodous acid, and he now suggests the more direct application of that acid as a means of shortening the time required for a determination. The fact, however, that it so readily decomposes ($5HIO = HIO_3 + 2H_2O + 4I$) prevents it from being employed with any advantage at first hand. It was therefore found best to obtain it by the action of water on iodine chloride ($ICl + H_2O = HCl + HIO$), using a solvent which only contained so much water as would decompose nearly the whole of the latter, and which at the same time would not be oxidised by the hypo-iodous acid when formed. These ends were attained with a solution of iodine chloride in 95 per cent. acetic acid. This was prepared by dissolving 13 grms. of iodine in a litre of the acetic acid, determining the titre of the solution, and passing into it a current of chlorine (free from hydrochloric acid) until the titre was doubled. With a little practice the exact point could be hit by observing the change in colour. The solution thus obtained, was more stable than that of Waller, and was employed in precisely the same manner as Hübl's solution, with the exception that the time required for the absorption was very greatly shortened. In the case of oils with low iodine values, it was complete in 3 or 4 minutes, and with those with higher values not more than 10 minutes were necessary, if too much oil had not been used.

The following are results obtained with various oils, the first figure in each case being the ordinary Hübl value, the second the iodine value by the new method :—Linseed, 180·91, 182·25 ; liver, 160·64, 166·51 ; maize, 124·87, 128·56 ; poppy-seed, 119·36, 119·67 ; sunflower, 117·81, 119·01 ; sesame, 110·35, 111·87 ; cotton-seed, 108·76, 110·07 ; rape, 102·96, 103·33 ; earthnut (arachis), 87·26, 87·25 ; olive, 83·27, 84·45.

In almost every case the numbers obtained were higher than the ordinary Hübl figures, but the author considered them the more correct by reason of the results obtained with purified allyl alcohol. This has a theoretical iodine value of 435. By Hübl's process, Lewkowitsch (*Oils, Fats, and Waxes*, p. 176) obtained results varying from 349—376. Using an excess of 75 per cent. of iodine the author obtained with Hübl's solution an iodine value of 425, and with iodine chloride in acetic acid using the same excess of iodine, his results were :—After 5 minutes 431·1, and after 10 minutes 436·8.—C. A. M.

### *Chicory : Its Analysis, &c.*

A. Ruffin. Ann. Chim. anal. appl. **3**, 114—119 ; Chem. Centr. 1893, **1**, [21], 1147.

It is stated that badly prepared chicory rapidly spoils on keeping, showing strong formation of moulds (*Pencillium Glaucum* and *Aspergillus Glaucus*). The following method is recommended for the technical analysis of chicory :—1. *Matter Soluble in Hot Water.*—15 grms. of the sample, dried at 100° C., are triturated with 150 c.c. of water, added gradually, and the mixture boiled for half an hour with a reflux condenser, frothing being guarded against at the beginning. After cooling, the insoluble matter is washed on a tared filter with boiling water, dried at 100° C., and weighed. 2. *Matter Volatile at* 100° C.—20 grms. of the sample are heated in a current of air at 100° C. to constant weight. 3. *Total Ash.*—10 grms. of the dried chicory are incinerated in a muffle at a red heat at the beginning, and the colour and weight of the ash are noted. 4. *Ash insoluble in Water.*—The ash is treated with boiling water, and the insoluble portion weighed after again incinerating. 5. *Ash insoluble in* HCl.—The residue from No. 4 is mixed with hydrochloric acid to a thin paste, the latter dried at 100° C., taken up with dilute hydrochloric acid (1 in 10), filtered, and the insoluble portion dried and weighed. The following results were obtained from 10 typical samples of commercial chicory :—

| — | Matter soluble in Water at 100° C. | Matter Volatile at 100° C. | Total Ash. | Ash Insoluble in Water. | Ash Insoluble in HCl. |
|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Maximum | 69·90 | 13·00 | 13·76 | 11·25 | 10·12 |
| Minimum | 65·40 | 5·10 | 4·00 | 1·92 | 0·83 |
| Mean ... | 67·25 | 11·40 | 7·22 | 5·80 | 4·93 |

Of 133 samples of commercial chicory taken indiscriminately, four were decayed, 47 gave a normal content of ash, and 82 were adulterated. Added earths caused an increase of ash-content up to 43·2 per cent., whilst other additions of leguminous seeds, acorns, &c., were easily detected under the microscope.—A. S.

### *Sugar, and Liquids Rich in Sugar, Determination of Ash in.*

G. Morpurgo. Chem. Zeit. 1898, **22**, [26], 257.

To facilitate the incineration of sugar, the author adopts the following method, which enables one to obtain in half an hour a pure white ash from 5 grms. of sugar, or from the residue, on evaporating 25 c.c. of a wine containing 20 per cent. of sugar. The residue from the wine is dissolved in a platinum dish with one-third of its volume of hydrogen peroxide, and evaporated carefully over a naked flame to dryness, until the mass becomes brown. It is allowed to cool, and a few more drops of hydrogen peroxide poured over it. It is dried, and the carbonisation is commenced carefully. The carbonised mass is ignited and allowed to glow, without further heating of the dish. In many cases it is only necessary to cause the platinum dish to glow very slightly to convert the mass into a pure white ash. If any dark patches are visible, they are moistened with a few drops of hydrogen peroxide, after cooling, and the mass again heated to redness. The operation is complete as soon as no sparks are visible.

The action of the hydrogen peroxide is twofold, being both mechanical and chemical. During the evaporation with hydrogen peroxide, the bubbles of gas evolved, render the mass porous, so that it burns at a lower temperature, and the combustion is aided by the oxygen. It is necessary to use pure hydrogen peroxide, which leaves no residue on evaporation. The method may also be used in incinerating drugs, where it will be found of great advantage.

—L. J. de W.

*Volatile Acids produced in Fermentation, Formula for the Estimation of, by Duclaux's Method.* F. Bordas and de Raczkowski. J. de Pharm. et de Chim. 1898, **7**, [10], 479—483.

In the case of a single acid the authors have devised the formula—

$$A = F \times n \times N,$$

in which A is the quantity of acid contained in 110 c.c. of the liquid which has been submitted to fractional distillation, F the factor for each acid, $n$ the number of c.c.'s of N-$n$/10 sulphuric acid necessary to neutralise the 20 c.c. of lime water used, and N the total volume of lime water required to neutralise the ten fractions. The values of F for the different acids are: formic, 0·00039; acetic, 0·00037; propionic, 0·00038; butyric, 0·00045; valeric, 0·00051.

In the case of two acids the formulæ are—

$$A' = F' \times n \times N(R - R'')$$
$$A'' = F'' \times n \times N(R' - R)$$

A′, F′, N′, and A″, F″, N″ have for each acid the preceding significations; this applies also to $n$ and N. R, R′, R″ are the values found for the mixture, and those which correspond to each acid. A table is given to show which acids correspond to A′ and A″, and also the value of each of these letters for a mixture of a known nature.—J. L. B.

*Beer, Estimation of Alcohol in.* L. Bousquet. La Bière, 1898, **6**, [5], 65—67.

The author reviews the principal methods which have been proposed from time to time, and awards the preference to those (*e.g.*, Salleron's) wherein the beer is simply distilled and the alcoholic content determined from the distillate by means of the alcoholimeter, the error due to the presence of volatile acids, being removed by neutralisation and re-distillation, and the temperature corrected to a standard (15° C.).

—C. S.

*Theobromine, Identification and Determination of the Purity of.* Maurice François. Journ. Pharm. Chim. **7**, [11], 521—524.

Since theobromine is assuming an important rôle in therapeutics, the author points out that it is desirable to have available, tests for its identification and for the determination of its purity, in particular with regard to its freedom from caffeine. The latter is readily accomplished by the following test, which is based on the relative insolubility of theobromine in strong alcohol at ordinary temperatures. 2·50 grms. of the alkaloid are introduced into a 50 c.c. graduated flask, which is filled to the mark with alcohol 95 per cent., and agitated occasionally for 24 hours at a temperature of 20° C., so as to obtain a saturated solution. 10 c.c. are then filtered off into a tared vessel, the alcohol is evaporated, and the residue dried over sulphuric acid; the weight of this should not exceed 0·005 grm. The relative solubilities of caffeine and theobromine in alcohol of this strength, under these conditions, have been determined. The amount of residue left by 10 c.c. of alcoholic caffeine solution is found to be 0·093 grm., while theobromine gives only 0·004 grm. A mixture containing 10 per cent. of caffeine and 90 per cent. of theobromine will give 0·055 grm. of residue. The crystalline character of the latter is quite distinct from that of pure theobromine. For the identification of the alkaloid the following tests were applied;

(1.) Dissolve 0·10 grm. of the alkaloid in a mixture of 1 c.c. of nitric acid and 2 c.c. of water; add 2 c.c. of silver nitrate solution, 10 per cent.; a precipitate is formed which disappears on warming; on cooling, a mass of crystalline needles is deposited; (2.) 0·10 grm. of the alkaloid is dissolved with heat in 2 c.c. of water and 1 c.c. of hydrochloric acid; 10 c.c. of saturated bromine water is then added and the flask weighed; excess of bromine is driven off by boiling, until an almost colourless solution is obtained, which is made up to its original weight. If to 2 c.c. of this, exactly 1 drop of a 5 per cent. ferrous sulphate solution be added, followed by 2 or 3 drops of solution of ammonia, an intense indigo-blue coloration is obtained. (3.) To 0·10 grm. of the alkaloid add 2 c.c. of water and 1 c.c. of hydrochloric acid and dissolve by the aid of heat; then add 10 c.c. of decinormal iodine solution when a dense black precipitate is formed. Decant the supernatant liquid and add to the precipitate 10 c.c. of 10 per cent. potassium iodide solution and heat to about 80° C. On cooling, long, blackish-green crystals of Joergensen's theobromine tetra-iodide will be formed and will ultimately half fill the tube.—J. O. B.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Potassium Carbonylferrocyanide, A Synthetic Method of Formation of.* J. A. Muller. Comptes Rend. 1898, **126**, [20], 1421—1423.

By the direct action of carbon monoxide upon potassium ferrocyanide, the carbonylferrocyanide is obtained—

$$K_4Fe(CN)_6 + CO + 2H_2O =$$
$$K_3Fe(CN)_5(CO) + NH_3 + KHCO_3.$$

With a 20 per cent. solution the reaction is very slow at the ordinary temperature; at 130° it is more rapid, and is complete in two days. The solution of potassium carbonylferrocyanide is not precipitated by lead nitrate or acetate. The copper salt, obtained by precipitation, has the formula $Cu_3Fe_2(CN)_{10}(CO)_2$.—A. C. W.

*Acidity [Apparent and Real] of some Acids, On the Influence of Temperature on the Determination of.* P. Degener. Zeits. für Spiritusind. 1898, 171.

Classen (Zeits. d. Ver. f. Rübenzucker-Ind. 1894, 692) has observed that asparagine has a neutralising power, varying with the temperature at which it is titrated. The author finds that the apparent acidity of a solution of asparagine increases with the temperature, but the amount is dependent on the indicator which is used. Aspartic acid behaves similarly. Sulphurous acid behaves in the opposite manner neutralising more alkali in the cold; this the author explains by assuming for it a formula $S(OH)_4$ in the cold, and $SO(OH)_2$ when hot. 100 c.c. of $\frac{1}{10}$N lactic acid neutralise 100 c.c. of $\frac{1}{10}$N alkali at 0° C. and 112 c.c. at 100° C. This is explained by assuming that one of the hydroxyl groups acquires, when heated, neutralising power. Butyric acid behaves similarly. Oxalic acid behaves normally. Glutaric acid only neutralises $\frac{3}{4}$ as much alkali as it should. Succinic, malic, tartaric and citric acids neutralise more alkali than theory requires; this is explained by assuming the presence of a small amount of anhydride.

The author sums up his experiments as follows:—

1. Some acids and other bodies possess less apparent acidity in the cold than when hot. Complex molecular groups which are present in the cold are broken up when heated.

2. Other acids which have been neutralised when cold become alkaline when heated; this is caused by the existence of ortho-salts, which are decomposed when heated.

3. Other acids contain hydroxyl groups which attain acid properties when heated.

4. Other acids possess the same apparent acidity hot or cold; or other multibasic acids have their true acidity disguised by that of the different salts which may be formed.

5. Other acids form anhydrides, which are only hydrated by heating, and consequently do not show their full neutralising power until this is effected.—A. L. S.

*Copper-alkali-Glycerin Compounds.* F. Bullnheimer. Ber. 1898, **31**, [10], 1453—1457.

Tartaric acid, glycerin, and other compounds prevent the precipitation of copper by alkalis. The soluble compounds formed have not hitherto been isolated. A copper sodium glycerate may be obtained by the following methods:—Allow 5 grms. of copper hydroxide, 15 c.c. of water, 2·5—3 grms. of glycerin, and 3 grms. of caustic soda (from sodium) to stand in a closed vessel until the alkali has dissolved, add 50 c.c. of 96 per cent. alcohol, filter, and then alcohol to the filtrate until a decided cloudiness appears. After 6—10 hours fine blue needles, united to cubical or warty masses appear. These are dried *in vacuo* without desiccating agents. Or to a solution of copper acetate in 150 c.c. of alcohol, 50 c.c. of water, and 6 grms. of glycerin, saturated at 60° C., add the solution of 5 grms. of sodium in 100 c.c. of 90 per cent. alcohol, filter whilst warm and let stand. The crystals obtained, possess the composition represented by $(C_3H_5O_3CuNa)_2 + C_2H_5.OH + 9H_2O$. The compound is readily soluble in water, absorbs carbonic acid from the air with formation of copper carbonate, and is not decomposed by acids in the cold. Kept at 100° the compound loses $C_2H_5.OH + 6H_2O$, but cannot be obtained anhydrous. Another compound, $C_3H_5O_3CuNa + 3H_2O$, is obtained by similar methods when copper nitrate or copper hydroxide and sodium nitrate are employed; this always contains about 4 per cent. of sodium nitrate. A copper lithium glycerate, $C_3H_5O_3CuLi + 6H_2O$, was isolated. The potassium compounds have not been obtained pure.

—A. C. W.

### *Fluorescence Phenomena in the Coumarin Series.*
H. Kunz-Krause. Ber. **31**, [8], 1189—1193.

When chrysatropic acid (4.hydroxy.5.methoxycoumarin) which in aqueous or alcoholic solution shows a fine blue fluorescence, is treated in a solution in absolute alcohol with metallic sodium, hydrogen is given off, and a voluminous precipitate of a sodium compound is formed. This is very hygroscopic and dissolves in water to a deep yellow solution, having a blue fluorescence. On investigating other coumarin derivatives, it was found that more particularly umbelliferon, daphnetin, and aesculetin, give similar sodium compounds. From his results, the author concludes that fluorescence and the reaction with metallic sodium of the coumarin molecule, is brought about or is increased by the introduction of side chains into the benzene ring whilst the number of the hydroxyl or methoxyl groups has no effect on the fluorescence. According to R. Meyer (Ber. **31**, 513; this Journal 1898, 418) fluorescence is dependent upon the presence of a closed chain fluorophore, but it appears more probable that in solution the lactone ring no longer exists, and that to this is due the fluorescence. The behaviour of coumarin supports this view since its solution only fluoresces after some time, and moreover *o*-coumaric acid, which does not contain the lactone ring, shows a fluorescence although in this case it is green. Further evidence is afforded by analyses of the sodium salts, since, in certain preparations, not only is the lactone ring opened by the action of sodium, but the HC:CH group is also reduced to NaHC.CHNa. The organic bases, aniline, pyridine, quinoline, do not react like alkalis. For example, chrysatropic acid dissolves to a colourless solution in aniline, whilst the solution in pyridine is yellow but non-fluorescent. On the other hand, these bases give well crystallised products with different coumarins. Other compounds, belonging to this group which exhibit fluorescent phenomena, are the condensation products obtained from coumarin with phenols.

—T. A. L.

### *Carbon Monoxide in Blood, The Amount Normally Contained.* M. Nicloux. Comptes Rend. **126**, [21], 1526—1528.

Both Desgrez and the author, and St. Martin (this Journal 1898, 603), have recognised the presence of carbon monoxide as a normal constituent of the blood of animals living in Paris. The author has tried to decide whether this gas is slowly fixed by the haemoglobin of the blood from the air, or is generated in the organism, by examining the blood of a country dog. The amount of carbon monoxide found was as great as that in town animals, but the author does not regard the experiment as conclusive, and is pursuing his investigations.—J. T. D.

### *Carbon Monoxide in the Blood, Influence of Asphyxia on the Quantity of; Production of Carbon Monoxide in the Organism.* M. Nicloux. Comptes Rend. 1898, **126**, [22], 1595—1598.

The blood contains a combustible gas (N. Gréhant). This gas contains hydrogen, "formene" (N. Gréhant), and carbon monoxide (L.-G. de St. Martin). In order to test this hypothesis, advanced in a former communication (Comptes Rend., May 23, 1898), the author takes advantage of the fact that under asphyxia the quantity of carbon monoxide in the blood is diminished. He finds that dogs asphyxiated just short of death afterwards regain in the course of an hour the amount of carbon monoxide originally present in the blood. He proves that the gas cannot be derived from the atmosphere, and concludes that it is a normal product of the organism itself. Details of the experiments are given.—T. A. L.

## New Books.

Traité d'Analyse des Substances Minérales. Par Adolphe Carnot, Membre de l'Institut, Inspecteur-général des Mines, Professeur et Directeur des Laboratoires à l'École supérieure des Mines. Tome Premier. Méthodes Générales d'Analyse, Qualitative et Quantitative. Vve. Ch. Dunod, Editeur, Quai des Grands-Augustins 49. 1898. Price 35 frs. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C.

An important work treating in detail of the analysis of mineral substances, this, the first volume, being devoted to "General Analytical Methods, Qualitative and Quantitative." This first volume contains an introduction, subject-matter filling 978 pages, and illustrated with 357 engravings, having also a table of contents, and an alphabetical index.

The text is subdivided into 14 chapters, treating of the following leading subjects:—I. Blowpipe Tests. II. Flame Reactions. III. Spectroscopic Work. IV. Micro-chemical Tests. V. Qualitative Analysis by the Wet Methods. VI. Principal Characteristics of Acids and Bases. VII. Reagents. VIII. Preliminary Tests. IX. Processes according to the Dry Methods. X. Processes according to the Wet Methods. XI. Electrolysis:—(*a.*) Production of the Electric Current. (*b.*) Electrodes. (*c.*) Metallic Deposits, &c. XII. Volumetric Methods of Analysis. XIII. Colorimetric Tests. XIV. Gas Analysis.

Notes on Volumetric Analysis. By Arthur Thornton, M.A., Senior Science Master, Bradford Grammar School; and Marchant Pearson, B.A., Assistant Science Master, Bradford Grammar School. Longmans, Green, and Co., 39, Paternoster Row, London, New York, and Bombay. 1898. Price 2*s.*

8vo volume containing preface, table of contents, 77 pages of subject matter, and an alphabetical index. There are four illustrations in the text, which is subdivided as follows:—I. Inductory. II. Apparatus. III. Conditions of Experiment. IV. Neutralisation. V. Oxidation. VI. Iodometry. VII. Precipitation Methods.

The Extra Pharmacopœia, Revised in accordance with the "British Pharmacopœia," 1898. By William Martindale, F.L.S., F.C.S., Member of Council of the Pharmaceutical Society, and late Examiner. Serotherapy, Organotherapy, Medical References, and a Therapeutic Index By W. Wynn Westcott, M.B. Ninth Edition. H. K. Lewis, 136, Gower Street, London, W.C. 1898. Price 10*s.* 6*d.*

This is the ninth edition of the work above-mentioned, the eighth edition of which was noticed in this Journal, 1895, 525. The eighth edition contained 584 pages of subject-matter, whilst this, the ninth edition, numbers 626 pages. It is stated that "The principal changes effected in the work have been due to its revision with a view to bring it into accord with the recent official Pharmacopœia." "The new synthetic compounds, Eucaine, Holocaine, and Orthoform, having local anæsthetic properties allied to Cocaine, have been studied. Compounds of Silver, the Citrate and Lactate, Argentol, Argonin, and Protargol have been introduced to medical practice, and are noticed. The compounds of Glycerophosphoric Acid have been employed as nervine tonics. New Bismuth compounds, Chlorodyne equivalents, Iodoform Succedanea, Antipyretics, and Antiseptics for internal as well as external use, have been largely employed; the last group includes Clinosol, Creosote, and Guaiacol compounds." "The controversies on Hyoscine and Scopolamine, and on Pilocarpine and Pilocarpidine, are referred to, as well as Euphthalmine—a synthetic compound for producing mydriasis, and Urotropine, a solvent for Uric acid, designed to assist in its elimination." "New monographs have been introduced on Aloes, Calcium compounds, Chelidonium, Digitalis, compounds of Mercury and of Iron, Mustard, Opium, Orange, Rose, Mescal Buttons, and other drugs." "The revolution in the official strength of Alcohol is explained by two tables, which may be found of practical utility for the preparation of the several degrees of dilution." "The Therapeutic Index has been extended, and now includes a classified list of antidotes to various poisons." The work is of the size known as medium 24mo.

Jahrbuch der Chemie. Bericht über die wichtigsten Fortschritte der reinen und angewandten Chemie, unter Mitwirkung von H. Beckurts, C. A. Bischoff, E. F. Dürre, J. M. Eder, P. Friedländer, C. Häussermann, F. W. Küster, J. Lewkowitsch, M. Märcker, F. Röhmann, and K. Seubert. Herausgegeben von Richard Meyer. VII. Jahrgang. 1897. Friedrich Vieweg und Sohn, Braunschweig. 1898.

8vo volume, bound in cloth, containing a report for the year 1897 of the progress of pure and applied chemistry during that period. The subject-matter fills 494 pages. It is preceded by a table of contents and is followed by alphabetical indexes of subjects and authors. The various branches of pure and applied chemistry treated of, are as follows:—I. Physical Chemistry (F. W. Küster). II. Inorganic Chemistry (Karl Seubert). III. Organic Chemistry (C. A. Bischoff). IV. Physiological Chemistry (Fr. Röhmann). V. Pharmaceutical Chemistry (H. Beckurts). VI. Chemistry of Foodstuffs (H. Beckurts). VII. Agricultural Chemistry (M. Maercker and W. Naumann). VIII. Metallurgy (E. F. Durre). IX. Fuels and Explosives. Inorganic Chemical Technology (C. Haussermann). X. Technology of Carbohydrates and Fermentation (M. Maercker, W. Naumann, and L. Bühring). XI. Technology of Fats and Petroleums (J. Lewkowitsch). XII. Chemistry of Coal Tar and Dyestuffs (Richard Meyer). XIII. Chemical Technology of Textile Fibres (P. Friedländer). XIV. Photography (J. M. Eder and E. Valenta).

# Trade Report.

## *PRIZES.*

### Prize for Matches without White Phosphorus.

*Eng. and Mining J., June* 25, 1898, 758.

United States Consul Roosevelt at Brussels furnishes further details concerning the offer of the Belgian Government of a prize of 50,000 francs for the invention of a match paste containing no white phosphorus. The competition is open until January 1st, 1899, and the conditions which paste and matches submitted to competition must fulfil are as follows: The paste must offer to the action of shocks and friction such a resistance that dangerous explosions may not be apprehended during the process of manufacture. It must not contain any matter which by its emanations or otherwise may be dangerous during the process of manufacture to the health of the workmen. The matches must ignite on any surface (even on cloth). Submitted to alternatives of humidity and dryness, of heat and cold, in the average limits, they must maintain their capacity to easily ignite and not be subject to spontaneous decomposition. The matches, when struck, must not throw off any substance of a nature to burn persons or provoke fires. They must not discharge toxic fumes. Preference will be given to matches the paste of which does not contain any poisonous ingredient. These matches must also not be susceptible to friction, so that under ordinary conditions they will not easily ignite in the consumer's pocket, and that they may be transported and stored without danger.

### Prize for Essay on "The Utility of Sulphate of Ammonia in Agriculture."

The Sulphate of Ammonia Committee offer the sum of 500 guineas to be given for the best essay on the following subject: "The Utility of Sulphate of Ammonia in Agriculture." The subject to be treated from a practical and scientific point of view.

The Committee has secured the services of Mr. William C. Little, Member of the late Royal Commissions on Agriculture, and Dr. Voelcker, Consulting Chemist to the Royal Agricultural Society of England, to act as judges of the essays sent in. In the event of any difference arising, Mr. Samuel Rowlandson, a member of the Council of the Royal Agricultural Society of England, will act as referee.

The essays must be in the English language, and written on one side of the paper only; they must further bear a distinguishing motto or *nom de plume*, but not the names of the authors.

Each author, when sending in his essay, must at the same time write his name and address on a slip, which he shall enclose in a separate envelope, seal the letter, and write outside it the motto or *nom de plume* he adopts for his essay. The essay and the envelope containing the slip, to be sent in, under one cover, to the undersigned.

The envelopes containing the names of the authors will be opened in the presence of the Committee after the judges have made their award.

All essays must be in the hands of the undersigned not later than November 15th, 1898. The selected essay will become the property of the Committee, who reserve to themselves the right to make such use of it as they may think fit.

The Committee also reserves to itself the right to purchase, for a sum not exceeding 50 guineas, any of the other essays.

W. G. Blagden (*Chairman*),
4, Fenchurch Avenue, London, E.C.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

### U.S. Drawback on Cyanide of Potassium.

*Eng. and Mining J., July* 2, 1898, 11.

The Treasury Department has issued the following regulations for the allowance of drawback of duty on cyanide of potassium manufactured from imported yellow prussiate of potash:—"On the exportation of cyanide of potassium, manufactured by the Roessler and Hasslacher Company, of New York City, in the manufacture of which no other than imported yellow prussiate of potash is used, a drawback will be allowed equal in amount to the duty paid on the imported material so used, less the legal deduction of 1 per cent. The entry under which the merchandise is to be inspected and laden must show the marks and numbers of the shipping packages, with the contents thereof, and the gross, tare, and net weight of each package separately. The drawback entry must show the net weight of the exported merchandise, and the manufacturer's declaration thereon must state, in addition to the usual averments, that the said merchandise was manufactured after the formula set forth in their sworn statement, filed with the collector of customs at New York. Samples may be taken, or sworn samples accepted, as ordered by the collector to be submitted to the appraiser for such expert examination as may be required; and in the liquidation of the entries the quantity of yellow prussiate of potash which may be taken as the basis of allowance of drawback shall not exceed 120 lb. for every 100 lb. of cyanide of potassium exported, after official verification of weight by a United States weigher."

### Duty on Coal Tar and Products.

*Eng. and Mining J., June 25, 1898, 758.*

A decision was handed down on June 11th by the Board on Classification of the United States General Appraisers on the protest of Samuel Cabot against the collector at Boston on an importation of merchandise invoiced as "crude tar acids," "tar oil," "creosote oil," and "coal tar product creosote." The merchandise in question is an oily substance of a dark brown colour and disagreeable odour, the product of the distillation of coal tar, and is of the same general character as the merchandise which was the subject of the board's decision on June 23rd, 1896, and resembling that which was the subject of the board's decision of July 23rd, 1896, which were reversed by the United States Circuit Court for the Eastern District of Pennsylvania and the United States Circuit Court of Appeals for the Second Circuit. Following these decisions of the courts the board sustained the claim of the protestants that the merchandise is entitled to free admission under the provisions of paragraph 443, Act of August 28th, 1894, and reversed the assessment of duty at 25 per cent. *ad valorem* under the provisions of paragraph 60 of the same Act.

### Samples of Perfumed Spirits by Parcel Post.

*Chem. and Druggist, July 9, 1898, 65.*

Under a general order, the Board of Customs have hitherto permitted samples of tea and tobacco to be sent by parcel post from Customs bonded warehouses to places abroad. This permission is, by an order dated July 4, to be extended to perfumed spirits manufactured in a bonded warehouse, provided "no single parcel of perfumed spirits shall contain less than 125 galls." Free goods may be included in the same parcel, the usual Customs conditions as to re-importation being observed.

## Germany.

### *Duty on Incandescent Mantles.*

One entirely new article is inserted on the Customs list, namely, mantles for incandescent lights (*Glühstrümfe*).

The following is a full translation of this new addition :—

(No. 12) (*Glühstrümpfe*) mantles for incandescent lights (glow substances for lighting purposes, steeped in solution of salts of certain metals, thorium, cerium, &c.) :—

| —— | No. in Tariff. | Duty per 100 Kilos. |
|---|---|---|
| | | Marks. |
| Mantles, not burnt-out (same as cotton-hosiery). | 2d3 | 120* |
| By "Treaty" tariff | .. | 95† |
| Mantles, not burnt-out, combined with parts of lamps out of non-precious metals. | 20c3 | 120* |
| Mantles, burnt-out | .. | Free. |
| Mantles, burnt-out, combined with parts of lamps, to be treated as goods out of the component metals, in combination with other metals. | | |

*Note.*—The combination of asbestos mantles with an asbestos thread for hanging purposes, is not included above; nor are burnt-out mantles, steeped in gelatin, collodion, gum shellac, &c., for the purposes of transport, included.

### *Denaturalisation of Oils at the Custom House.*

Her Majesty's Ambassador at Berlin, transmits translation of an extract from the *Central Blatt*, having reference to a decree of the Bundesrath, as follows :—

"The Bundesrath, in its sitting on May 20th, decreed that Note 4 to the article "Oils," on pages 315 to 316 of the official list of goods in the Custom House Tariff (*Waarenverzeichnisszum Zolltarif*), was to take the following new form :—

'The denaturalisation of cotton-seed and castor oils is to be effected by the addition of 1 kilo. of turpentine oil, 100 grms. of oil of rosemary, 250 grms. of lavender, pink, bergamot, orange, or lemon oil, 1 kilo. of ordinary strong-smelling petroleum, 3 kilos. of caustic lye of the specific gravity of 1·29, or 3 kilos. of lye of soda (*natronlauge*) of the specific gravity of 1·34 to 100 kilos., gross, of cotton-seed, tree, or castor oils.

"That of earth-nut (*arachis*) oil, coming from most-favoured-nation states, is to be effected by the addition of 1 kilo. of concentrated sulphuric acid or of 3 kilos. of lye of soda (*natronlauge*) of the specific gravity of 1·34 to 100 kilos., gross, of earth-nut oil.

"The means of denaturalisation is by well stirring and mixing with the oil. The caustic lye is to be added hot.

"The denaturalisation will, ordinarily, take place in the business offices of the person requesting it.

"By special permission, the denaturalisation can also be effected by other ingredients."

## France.

### *Chemical Tariff Changes since 1892.*

*Chem. and Druggist, July 16, 1898, 102.*

The French Customs are responsible for occasional changes of tariff, which are only announced by circular, so that British traders have difficulty in learning of them. The British Chamber of Commerce in Paris have just collected all such orders, and have published a circular, in which 61 changes, which have been made since the promulgation of of the French tariff in 1892, are recorded. The following are the items which interest our subscribers, the rates being per 100 kilos. unless where otherwise stated :—

| Articles. | Duty under Old Classification. | Duty under New Classification. |
|---|---|---|
| Iodised antiseptic tissues. | Duty according to the nature of the tissue. | Ditto, *plus* duty on iodine contained, at the rate of 4 frs. per kilo. |
| Unrefined vaseline | 28 frs. | 9 frs. |
| Precipitated phosphate of lime. | 5 per cent. *ad val.* ... | Free. |
| Essences of Indian geranium, palma rosa, and andropogon. | 500 frs. | 50 frs. |
| Acetic ether | 30 frs., *plus* denaturalisation tax at the rate of 1·25 litre of alcohol per kilo. | 30 frs., *plus* denaturalisation tax at the rate of 0·73 litre of alcohol. |
| Pyrazolon and antipyrin. | 5 per cent. *ad val.* ... | 253 frs. for pyrazolon and 5 per cent. *ad val., plus* 99 frs. for antipyrin. |
| Acetic acid, crystallised. | 5 per cent. *ad val.* ... | 69·75 frs. |
| Salol | 15 frs. | 15 frs., *plus* duty on alcohol, at the rate of 60 frs. per 100 kilos. of salol. |
| Antifebrin | 138 frs. | 25 frs. |
| Santonin | 5 per cent. *ad val., plus* the alcohol duty. | 5 per cent. *ad val.* |
| "Bromide, classed as alcoholic chemical product, to be classed as brom." | 5 per cent. *ad val., plus* the alcohol duty. | 50 frs. |
| Theobromine | 5 per cent. *ad val., plus* the alcohol duty on 30 kilos. of alcohol per kilo. of theobromine. | 5 per cent. *ad val., plus* the alcohol duty on 25 kilos. of alcohol per kilo. of theobromine. |
| Permanganate of potash. | 5 per cent. *ad val.* ... | 10 frs. |

*Oxalic Acid.*—It is notified that the legal tare on oxalic acid imported in wooden cases or barrels is fixed at 9 per cent.

*Acetic acid,* when denaturalised in bond, is to be passed free of the internal tax only when leave has been obtained from the Directeur des Contributions Indirectes.

"*Bromide*" is, we presume, ethyl bromide.

We note further that during January–May this year Great Britain exported to France chemical products amounting in value to 13,060,000 frs. (nearly 1½ million francs less than in the same period last year), and France exported to us 6,038,000 frs. worth of the same, or nearly half a million more than in 1897.

### United States.

#### *Customs Decisions.*

So-called *dead oil*, or *tar oil*, which is not known commercially or scientifically as distilled oil, is entitled to free entry as a preparation of coal tar under paragraph 443, Act of 1894.

*Carbolineum*, or *tar oil*, is free of duty as a product of coal tar under paragraph 443, Act of 1894.

All *indigo* is to be admitted free of duty, whether derived from Bengal plants or coal-tar products.

*Ores* containing less than 10 per cent. of iron and from 40 to 50 per cent. of manganese, or over, are commercially known as manganese ores, and free of duty.

*Ground sulphide of antimony ore* is free under paragraph 476, Act of 1897. The mere rough-grinding of the ore, for convenience of transportation, is no bar to free importation under the said paragraph.

### Export Duty on Spanish Iron Ore.

#### *Bd. of Trade J., July* 1898.

A telegram from Her Majesty's Ambassador at Madrid has been received at the Foreign Office, in which it is stated that the Spanish export duty on iron ore has been fixed at 25 centimos per 1,000 kilos., this rate taking effect from the 2nd instant.

## *DIPLOMATIC AND CONSULAR REPORTS.*

### Report on the Trade of Germany for 1897.

#### *Dipl. and Cons. Reps.*, [2130], 4—29.

*Leather.*—About the normal quantity of leather was imported from England during the first half of 1897, but in the second half the amount was greatly diminished, because, owing to a scarcity of the raw material, the price of leather was higher in England than in Germany. In consequence of the high prices ruling in England, a large quantity of sole-leather, leather waste, &c. was exported thither from Germany. It is stated that the trade in leather between England and Germany is no longer as important as it was, owing, on the one hand, to the high import duty (17*s*. 9*d*. and 35*s*. 6*d*. per 220½ lb.), and on the other, to the fact that, in consequence of the great improvement in technical details in German tanneries during the past 10 or 15 years, coarse black leather can be produced in Germany no dearer than in England. Sole-leather and prepared horse-skins are now exported to England. The export trade to England of the finer kinds of leather, as "kips," calf-skin, &c., has greatly diminished. The export trade from England to Germany of different kinds of tanned sheep-skins from English and Australian tanneries, and of buffalo- and cow-hides from East Indian tanneries, has been about the same during 1897 as in 1896. The decree issued by the German Government on Feb. 9, 1897, forbidding the import of all skins and hide from the East Indies, has been rescinded.

*Copper.*—The copper industry of Germany has shown a very large increase during the past year, but, owing to the increased home demand, no export trade has been undertaken. The import of raw copper from England has decreased, the greater part required by the increased consumption being supplied by North America.

It is stated that a competent German critic considers it is no longer possible for the London market to retain its former powerful position as the ruler of prices, as the English production gets smaller and smaller, and is not sufficient to supply the existing demand, the quantity represented by pending contracts being, in fact, frequently larger than the whole reserves. The total reserves were only 23,079 tons on February 15, 1898, as against 25,098 tons at the end of December 1897.

*Indigo.*—A union has recently been formed by three German firms to command cheaper prices and to act independently of the "Institute of Commission Agents in Bombay." The lower prices observed at the last indigo auction in London were probably due to the large quantities of artificial indigo manufactured by a German firm.

*Refined Camphor.*—The prices of this article have been kept down by speculators, who hold a considerable stock. It is stated that the stock of 6,000 piculs held by the London Speculation Syndicate are being kept out of the market.

*Mercury.*—It is believed the reduced demand for mercury for ordinary purposes will be more than compensated for by the proposed use of this metal for electrical purposes.

*Pharmaceutical Preparations.*—A large increase in German exports have taken place since the conclusion of the commercial treaties, and at the same time, an increased production has resulted in the lowering of prices. It is stated that a depressing influence is exerted on pharmaceutical trade by the stringency of the existing laws, and by the fact that the line dividing quack remedies ("Geheimmittel") from legitimate medicines is not strictly defined.

*Acetic Acid.*—The price of this article has been kept low by over-production. The amount of calcium acetate and citrate imported was 7,547 tons in 1895, 7,814 tons in 1896, and 9,867 tons in 1897. Of the latter amount, 1,061 tons came from Belgium, 591 tons from Great Britain, and 7,594 tons (calcium acetate only) from the United States. The export of vinegar and acetic acid in barrels amounted to 3,061 tons in 1897, 681 tons being sent to Great Britain, and the greater part of the remainder to Belgium and Switzerland.

*Borax.*—In consequence of the competition of the English import trade in borax and boracic acid, the Union of German Manufacturers ordered a general reduction in price of 10*s*. per 100 kilos. (220½ lb.), with a further reduction of 3*s*. per 100 kilos. for borax at the beginning of the year. The amount of borax and boracic acid imported in the free German market was 2,934 tons in 1896, and 2,472 tons in 1897, 1,805 tons (principally borax) of the latter amount coming from Great Britain.

*Ammonium Sulphate.*—The depression in the market for sulphate of ammonia lasted until September 1897, being probably due to the over-production of coke consequent on the increased iron trade. The use of ammonium sulphate in Germany is steadily increasing, and the home production is not nearly sufficient to meet the demand. 33,113 tons of ammonia were imported into Germany in 1897, 24,023 tons coming from Great Britain.

*Antipyrin.*—The market for this article will probably undergo a great change shortly, as the English patent rights in the "Hochst Farbwerke" expired in February 1898, and the German patent will expire in July.

*Chloride of Lime* (*Bleaching Powder*).—The large amount of chloride of lime produced by electrical processes has now caused the export of this article from Germany to be greater than the import. Nine-tenths of the amount imported comes from Great Britain, whilst Belgium and the United States each take one-third of the quantity exported. It is stated that a proposal has been made to stop the works producing this article by the cheap Leblanc process, in order that the production of the other factories—at which electrical processes are employed—may be regulated.

*Caffeine.*—A reduction of 25 per cent. in the price of caffeine and similar substances has been effected by the manipulations of the English manufacturers.

*Sodium Carbonate.*—The import of soda from England has gradually decreased, whilst the export from Germany to foreign countries has decreased.

*Phosphorus.*—It is stated that the English monopoly of the phosphorus trade is threatened by the increased production in France and Russia, and probably within a short time also Germany, where many new phosphorus factories are being projected. 260 tons were imported to Germany in 1897, to meet a consumption of about 200 tons, and 71 tons were exported from Germany to Austria.

*Paraffin.*—The export of German candles to England has been reduced to a minimum, in consequence of American competition in the paraffin trade.

*Imports from Great Britain to Germany during* 1896 *and* 1897.

| — | 1896. Without any "Improvement Traffic." | 1897. With a Portion of "Improvement Traffic." |
|---|---|---|
| 1. Drugs, druggists' wares, colours:— | Tons. | Tons. |
| Lacquer varnish | 460 | 442 |
| Caustic soda | 425 | 783 |
| Ammonia (carbonate of) | 1,639 | 1,382 |
| „ (sulphate of) | 21,985 | 23,637 |
| Anthracene | 4,635 | 5,176 |
| Borax, boracic acid | 2,111 | 1,805 |
| Chromate of potash | 508 | 445 |
| Raw glycerin | 2,672 | 4,519 |
| Indigo | 403 | 167 |
| Carbolic acid | 3,077 | 3,272 |
| Sulphate of soda | 367 | 1,040 |
| Coal-tar oil (light) | 7,975 | 6,832 |
| Superphosphate | 15,550 | 27,959 |
| 2. Earths, ores, precious metals:— | | |
| Natural colouring earths | 2,417 | 2,319 |
| Phosphate of lime (natural) | .. | 4,211 |
| Kaolin, felspar, fireclay | 72,517 | 77,944 |
| "Strontian" | 14,900 | 7,609 |
| Lead ore | 2,437 | 1,993 |
| Iron ore | 7,923 | 10,395 |
| Thomas's slag (ground) | 14,779 | 20,182 |
| | Ounces. | Ounces. |
| Gold (coined) | 16,788 | 308,664 |
| „ (unwrought) | 1,103,736 | 804,496 |
| Silver | 2,916,708 | 3,512,604 |
| | Tons. | Tons. |
| 3. India-rubber and gutta-percha | 2,837 | 2,932 |
| 4. Copper and copper wares:— | | |
| Raw copper | 7,358 | 8,521 |
| 5. Leather and leather goods:— | | |
| Leather (not particularised) | 1,177 | 1,318 |
| Sheep and goat skins | 1,853 | 2,142 |
| 6. Oils (not particularised) and fat:— | | |
| Cotton-seed oil (in barrels) | 658 | 1,547 |
| „ (officially denaturised). | 12,836 | 9,364 |
| Palm and cocoanut oil | 1,967 | 2,449 |
| Fat | 6,773 | 6,703 |
| 7. Tar, pitch, resin, asphalt:— | | |
| Pitch (excepting asphalt) | 32,785 | 35,766 |
| Tar | 7,191 | 9,852 |
| 8. Zinc and zinc goods:— | | |
| Raw zinc | 4,155 | 2,810 |
| 9. Tin and tinware: | | |
| Tin (raw and broken) | 5,754 | 5,029 |

*Exports from Germany to Great Britain during* 1896 *and* 1897.

| — | 1896. Without any "Improvement Traffic." | 1897. With a Portion of "Improvement Traffic." |
|---|---|---|
| 1. Drugs, druggists' wares, colours:— | | |
| Ultramarine | 1,670 | 1,594 |
| Alizarin | 2,382 | 1,971 |
| Aniline and other coal-tar stuffs | 3,544 | 3,517 |
| White-lead | 11,020 | 9,532 |
| Chlorate of potash | 11,452 | 9,473 |
| Glycerin (purified) | 581 | 647 |
| Red-lead | 1,910 | 1,858 |
| Nitrate of potash | 4,249 | 2,749 |
| Tartaric acid | 712 | 819 |
| Protoxide of zinc | 5,947 | 5,624 |
| 2. Earths, ores, precious metals:— | | |
| Potash salts ("Abraumsalze") | 34,584 | 44,166 |
| | Ounces. | Ounces. |
| Gold (coined) | 10,164 | 6,104 |
| „ (unwrought) | 1,332 | 384 |
| Silver | 553,522 | 859,667 |
| 3. Leather and leather goods:— | Tons. | Tons. |
| Glove leather, cordovan, morocco | 1,198 | 1,098 |
| Fine leather goods | 1,046 | 1,127 |
| 4. Oil (not particularised) and fat:— | | |
| Rape-seed oil (in barrels) | 6,615 | 2,118 |
| Oilcake | 41,447 | 53,700 |
| 5. Earthenware:— | | |
| Coloured china goods | 4,641 | 4,848 |
| 6. Zinc and zinc goods:— | | |
| Raw zinc | 21,492 | 16,014 |
| Zinc wire, rolled zinc | 6,496 | 6,466 |

—A. S.

### ALBUMIN MANUFACTURE AT HANKOW.

*Foreign Office Annual Series, No.* 2126, *June* 1898.

A new branch of export trade was started in Hankow last year—the manufacture of albumin. Three firms, one Austrian and two German, have already taken it up, and others are about to do so. The competition is likely to become keen, with the result that profits will be small, which is perhaps the reason why no British firm has seen its way to compete for the trade.

Several kinds of albumin are produced, the best being employed in photography, the medium quality is used instead of eggs in various articles of diet, whilst the inferior kinds are notably employed in the printing of cottons. The essence of the yolk of the eggs is also extracted, and is used for polishing leather, &c. At present, apparently, no use whatever is made of the shells.

As the eggs employed in the manufacture must be absolutely fresh, the season is restricted to two periods, of about two months each, every year. One firm alone uses between 30,000 and 40,000 eggs a day during the season. In spite of this large demand, no rise in the price of eggs seems to be anticipated.

### PAINT INDUSTRY OF JAPAN.

*U.S. Cons. Reps., June* 1898, 307.

The *Japan Herald* of March 2, 1898, states that the import of paint is increasing year by year to keep pace with other industries. The following figures show the quantities imported and re-exported during the last ten years:—

| Year. | Import. | | | Re-export. | | |
|---|---|---|---|---|---|---|
| | Quantity. | Value.* | | Quantity. | Value.* | |
| | Lb. | Yen. | Dols. | Lb. | Yen. | Dols. |
| 1888 | 1,205,313 | 109,641 | 82,560 | 3,143 | 244 | 184 |
| 1890 | 1,186,697 | 108,549 | 99,539 | 6,441 | 611 | 560 |
| 1892 | 1,011,338 | 97,184 | 68,612 | 4,863 | 431 | 304 |
| 1894 | 1,221,357 | 148,100 | 75,975 | 8,757 | 1,230 | 631 |
| 1896 | 2,463,921 | 272,146 | 144,237 | 24,660 | 3,909 | 2,072 |
| 1897 | 3,085,082 | 369,435 | 187,673 | 25,989 | 3,864 | 1,963 |

* Reduced to United States currency according to the average given for each year by the director of the mint. The gold standard, on which basis the yen is valued at 49·8 cents, was adopted in Japan, October 1, 1897.

The re-export was to Korea, China, and Asiatic Russia, but was very insignificant, the consumption at home covering nearly the whole. After 1895, the quantity imported, doubled—a fact which may be partly due to the increased repairs of vessels, necessitated by the war with China, but also to general increase of demand. The countries which import paint are England, Germany, Belgium, France, and the United States, the British product forming nearly 80 to 90 per cent. of the entire import. In Japan the industry is so far insignificant.

### THE ENGLISH LEATHER TRADE IN GERMANY.

*Foreign Office Annual Series, No.* 2122, *June* 1898.

Complaints have been made lately by German business men, who do a large and regular trade with England, of the lack of willingness on the part of English manufacturers to meet their wishes for even small changes which may be necessary to increase sales, even at the instance of old customers. The German purchaser simply cannot understand such ways, and complains bitterly of this want of obligingness, which, he says, frequently makes business connection with English firms unpleasant. The German business man, in small or great matters, meets the desires of his business friends; he makes the shape or colour, or the packing of goods altogether in accordance with the wishes of his customers, even when he is convinced that other shapes, colours, or packing would be better. But he never tries to impose his own judgment on buyers; he is satisfied if he can meet the wishes of his customers and can retain their patronage. With such business principles he is contented, prospers, and is becoming more and more a formidable competitor for other countries.

An important leather manufacturer in a neighbouring industrial city has for years tried to obtain from his English purveyor a certain shade of colour, for which he would have had excellent use. All requests and letters met with a negative answer, even when applying personally in England. To-day a great French firm is trying to obtain the contract for this very supply. It is asserted in this special instance that the manufacture of the desired shade of colour would have been an easy matter; the German manufacturer even was ready to supply the colour himself, and was willing to take the quantity manufactured, no matter how it might have turned out. This German merchant holds that such an example is typical of English business men. Whatever he puts on the market is good and solid, but one must be content with it. Similar complaints are frequently made as to the packing. Fine leathers become less valuable if they are folded, because the folded parts cannot be used; yet years of petitioning and trouble were necessary before he could induce the British manufacturer to forward this leather rolled up. Many such examples, it is to be regretted, could be given; but the fact must be constantly emphasised that nowadays it is of the greatest importance in international competition to meet the desires of customers to the greatest possible extent.

### Matches in Tangier.

*Foreign Office Annual Series, No. 2131, 1898.*

Of the 1,674*l.* worth of matches imported, 1,176*l.* came from France, and only 150*l.* from England. The matches most in use amongst the natives are the ordinary cheap French thin match, made up in the small paper boxes, each holding about 50 or 60 matches. These are sold retail here at five boxes for 10 c., say at about 1*d.* the half-dozen boxes.

### Frankfort.

*Foreign Office Annual Series, No. 2122, June 1898.*

Table A.

*Showing Quantity of Imports from England during the Years 1896–87.*

| Articles. | Quantity in 100 Kilos. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1896. | 1895. | 1894. | 1893. | 1892. | 1891. | 1890. | 1889. | 1888. | 1887. |
| Drugs and dyestuffs... | 873,133 | 828,110 | 984,838 | 951,022 | 850,727 | 991,137 | 1,205,885 | 1,082,945 | 827,461 | 668,754 |
| Hides and skins ...... | 76,111 | 68,653 | 76,356 | 78,170 | 63,529 | 65,995 | 66,240 | 110,053 | 57,648 | 52,537 |
| Caoutchouc, gutta-percha. | 33,151 | 26,002 | 27,840 | 24,628 | 24,718 | 22,946 | 22,532 | 28,500 | 21,472 | 16,139 |
| Copper and copper goods. | 102,157 | 97,907 | 96,318 | 279,818 | 254,364 | 255,415 | 266,262 | 226,597 | 34,433 | 74,942 |
| Leather and leather goods. | 36,428 | 35,321 | 30,296 | 32,298 | 31,734 | 37,462 | 41,024 | 44,894 | 42,667 | 34,931 |
| Oils, fats.............. | 382,521 | 405,740 | 431,310 | 476,109 | 505,243 | 494,690 | 505,801 | 555,280 | 371,214 | 372,445 |
| Coal, coke, and peat... | 43,665,225 | 40,130,683 | 37,310,047 | 36,217,996 | 35,528,453 | 40,667,520 | 32,563,973 | 34,619,647 | 19,447,427 | 15,868,353 |
| Tar, pitch, asphaltum . | 477,544 | 392,578 | 313,595 | 322,173 | 197,789 | 155,400 | 190,977 | 157,146 | 157,976 | 116,924 |
| Tin and tinware ...... | 58,721 | 45,983 | 55,075 | 55,183 | 39,597 | 41,579 | 39,337 | 48,116 | 38,906 | 29,910 |

Table B.

*Showing Value of Imports from England during the Years 1896–87.*

| Articles. | Value in 1,000 Marks. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1896. | 1895. | 1894. | 1893. | 1892. | 1891. | 1890. | 1889. | 1888. | 1887. |
| Drugs and dyestuffs... | 40,386 | 37,956 | 38,997 | 46,633 | 46,828 | 43,706 | 52,637 | 55,691 | 43,631 | 38,068 |
| Hides and skins ...... | 19,990 | 19,959 | 19,878 | 19,950 | 14,008 | 17,073 | 17,644 | 24,452 | 15,070 | 16,024 |
| Caoutchouc, gutta-percha. | 15,391 | 11,874 | 12,128 | 12,300 | 14,527 | 14,798 | 18,199 | 20,068 | 15,267 | 12,686 |
| Copper and copper goods. | 12,052 | 10,505 | 10,089 | 29,709 | 29,127 | 31,763 | 33,832 | 24,067 | 5,916 | 7,751 |
| Leather and leather goods. | 12,643 | 12,382 | 9,751 | 10,788 | 10,622 | 12,539 | 13,918 | 14,679 | 13,318 | 11,636 |
| Oil and fats........... | 13,335 | 15,131 | 18,249 | 18,726 | 17,707 | 19,707 | 20,349 | 21,234 | 14,778 | 14,507 |
| Coal .................. | 48,455 | 52,312 | 49,330 | 49,063 | 50,031 | 65,543 | 52,643 | 52,493 | 22,328 | 17,502 |
| Tar, pitch............. | 9,808 | 9,634 | 7,752 | 7,744 | 5,654 | 4,283 | 4,588 | 4,011 | 3,814 | 2,830 |
| Tin and tinware ...... | 7,307 | 5,915 | 7,682 | 9,401 | 7,536 | 7,709 | 7,483 | 9,255 | 8,765 | 1,814 |

Table C.

*Showing Quantity of Exports to England during the Years 1896–87.*

| Articles. | Quantity in 100 Kilos. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1896. | 1895. | 1894. | 1893. | 1892. | 1891. | 1890. | 1889. | 1888. | 1887. |
| Drugs and dyes ....... | 929,645 | 887,480 | 829,784 | 761,190 | 633,423 | 757,047 | 724,591 | 765,801 | 610,109 | 668,472 |
| Glass ................ | 313,057 | 344,311 | 349,870 | 350,575 | 271,364 | 257,596 | 164,921 | 124,525 | 95,196 | 82,107 |
| Copper................ | 44,055 | 36,251 | 34,655 | 34,070 | 26,485 | 27,385 | 31,340 | 22,982 | 16,161 | 11,596 |
| Leather ............... | 33,951 | 38,800 | 32,641 | 32,265 | 32,075 | 33,796 | 32,359 | 29,037 | 8,524 | 16,520 |
| Paper................ | 610,545 | 624,647 | 590,818 | 526,185 | 516,044 | 457,531 | 371,131 | 394,373 | 264,115 | 275,153 |
| Zinc ................. | 293,371 | 295,708 | 305,026 | 300,782 | 261,224 | 305,729 | 291,207 | 365,082 | 278,893 | 317,861 |

### TABLE D.

*Showing Value of Exports to England during the Years* 1896–87.

| Articles. | Value in 1,000 Marks. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1896. | 1895. | 1894. | 1893. | 1892. | 1891. | 1890. | 1889. | 1888. | 1887. |
| Drugs and dyes | 56,807 | 52,895 | 48,157 | 47,798 | 46,214 | 47,972 | 47,612 | 44,785 | 33,351 | 31,091 |
| Glass | 8,136 | 8,842 | 8,902 | 8,749 | 7,792 | 7,831 | 6,467 | 4,859 | 3,874 | 3,229 |
| Copper | 8,302 | 6,990 | 6,641 | 6,065 | 5,836 | 6,309 | 6,946 | 5,711 | 4,880 | 3,546 |
| Leather | 38,395 | 64,664 | 36,822 | 39,052 | 39,139 | 41,761 | 40,132 | 36,023 | 23,735 | 22,558 |
| Paper | 29,335 | 32,968 | 31,204 | 29,704 | 29,845 | 28,377 | 26,332 | 22,446 | 14,444 | 16,162 |
| Zinc | 10,402 | 9,005 | 9,332 | 11,026 | 11,845 | 11,889 | 13,686 | 15,402 | 10,478 | 9,748 |

*General Imports and Exports in Values for the Year* 1897, *as compared with those of the Years* 1896 *and* 1895, *calculated for* 1897 *according to the Prices fixed for* 1896.

| Articles. | Value in Thousand Marks. | | | | | |
|---|---|---|---|---|---|---|
| | Imports. | | | Exports. | | |
| | 1897. | 1896. | 1895. | 1897. | 1896. | 1895 |
| Rags and refuse of every description | 70,453 | 63,243 | 40,600 | 12,315 | 10,542 | 12,305 |
| Lead and leaden goods | 8,592 | 8,143 | 6,487 | 12,787 | 11,668 | 11,641 |
| Drugs and dyes | 244,075 | 233,685 | 232,913 | 326,730 | 316,788 | 298,809 |
| Ores, fine metals, asbestos, &c. | 329,099 | 372,711 | 233,341 | 218,974 | 285,303 | 152,883 |
| Glass and glassware | 11,358 | 10,958 | 9,659 | 43,733 | 42,793 | 46,956 |
| Hides | 181,622 | 155,483 | 186,378 | 84,365 | 71,620 | 81,825 |
| Hops | 6,872 | 7,829 | 6,612 | 19,623 | 19,556 | 27,291 |
| Caoutchouc, gutta-percha, and goods thereof | 46,403 | 45,104 | 35,736 | 35,119 | 31,251 | 27,432 |
| Copper and copper goods | 87,001 | 71,823 | 55,537 | 85,051 | 88,718 | 72,960 |
| Leather and leather goods | 58,359 | 53,084 | 54,808 | 150,446 | 140,398 | 165,807 |
| Candles | 121 | 102 | 115 | 199 | 658 | 581 |
| Oils, not otherwise mentioned, and fats | 155,398 | 128,868 | 136,518 | 29,366 | 28,987 | 26,394 |
| Paper and cardboard goods | 13,607 | 12,630 | 10,754 | 97,277 | 100,706 | 164,354 |
| Petroleum | 81,402 | 74,791 | 76,321 | 1,939 | 1,266 | 1,151 |
| Soap and perfumery | 1,941 | 1,831 | 2,628 | 13,213 | 13,693 | 10,734 |
| Stones and earthenware | 36,983 | 33,194 | 31,756 | 23,860 | 25,359 | 21,860 |
| Coal, brown coal, coke, and peat | 124,783 | 114,181 | 110,742 | 169,293 | 161,681 | 145,769 |
| Tar, pitch, resin, and asphaltum | 39,567 | 38,785 | 33,353 | 9,576 | 10,100 | 8,312 |
| Animals, and animal products not otherwise mentioned. | 134,349 | 116,594 | 127,745 | 8,016 | 7,647 | 9,191 |
| Oilcloth | 904 | 973 | 1,235 | 1,232 | 1,310 | 1,140 |
| Zinc and zinc goods | 6,901 | 5,789 | 5,294 | 27,274 | 29,150 | 25,038 |
| Tin and tinware | 15,715 | 17,481 | 13,927 | 4,982 | 5,169 | 4,923 |
| Total | 4,832,891 | 4,577,951 | 4,246,111 | 3,808,131 | 3,753,822 | 3,424,076 |
| Increase, 1896 | | 274,940 | | | 54,309 | |
| " 1895 | | 586,780 | | | 384,055 | |

*General Imports and Exports in Quantities for the Year* 1897, *arranged according to the Branches of Goods in the German Customs Tariff.*

| Articles. | Quantity. | |
|---|---|---|
| | Imports. | Exports. |
| | Met. Centners. | Met. Centners. |
| Rags, refuse of every description | 9,150,719 | 1,294,912 |
| Lead and leaden goods | 358,910 | 379,344 |
| Drugs and dyes | 10,834,330 | 6,693,268 |
| Ores, fine metals, asbestos, &c. | 57,454,687 | 54,188,859 |
| Glass and glassware | 152,623 | 1,150,231 |
| Hides | 1,295,881 | 450,624 |
| Hops | 26,739 | 99,106 |
| Caoutchouc, gutta-percha, and goods thereof. | 100,154 | 66,711 |
| Copper and copper goods | 805,903 | 483,941 |
| Leather and leather goods | 117,090 | 163,067 |
| Candles | 1,340 | 6,240 |
| Oils, not otherwise mentioned, fats | 6,714,356 | 1,574,649 |
| Paper and cardboard goods | 394,769 | 1,851,594 |
| Petroleum | 10,359,507 | 130,019 |
| Soap and perfumery | 13,348 | 72,194 |
| Stones and earthenware | 9,706,913 | 5,959,692 |
| Coal, brown coal, coke, peat | 147,595,699 | 148,330,751 |
| Tar, pitch, resin, asphaltum | 2,426,297 | 779,370 |
| Animals and animal products, not otherwise mentioned. | 1,569,057 | 90,165 |
| Oilcloth | 4,936 | 6,953 |
| Zinc and zinc goods | 200,666 | 706,825 |
| Tin and tin wares | 124,800 | 21,594 |
| Total, 1897 | 401,630,423 | 280,076,094 |
| " 1896 | 364,102,570 | 257,198,756 |
| Increase, 1897 | 37,527,853 | 22,877,338 |

### CEMENT AND CANDLES AT TANGIER.

*Foreign Office Annual Series, No.* 2131, 1898.

Of 562*l.* worth of Portland cement, 332 barrels were English, whilst 612 came from France and 59 from Germany. French cement, though recognised as inferior, is frequently preferred to English on account of its being cheaper. 5,545 bags of gypsum were brought from France.

Though the quantity of candles imported was greater that the preceding year a fall in prices makes their total value smaller. As usual they were almost entirely of English make. Belgian manufacturers are making great efforts to place their candles on this market. English makers should therefore be careful not to allow themselves to be ousted from this market, as in ordinary years the total comsumption of candles throughout Morocco probably amounts to about 30,000 cwt.

### FIREBRICKS AT WARSAW.

*Foreign Office Annual Series, No.* 2135, 1898.

There is a great scarcity of firebricks in this district, although refractory clay is to be found in abundance. As yet there is only one factory for their manufacture in Poland, the output of which cannot meet the demand on account of the growth of metallurgical and chemical industries. Nearly all the firebricks used come from Germany. There would be a good opening for an English firm in this line, as English bricks are deemed superior to German. Those on the market are sold at a high price, and to compete with the German article the price would have to be lower.

## MINERAL PRODUCTION OF CANADA, 1897.

*U.S. Cons. Reps., June* 1898, 197.

*Summary of the Mineral Production of Canada in* 1897.

| Product. | | Quantity.* | Value.* |
|---|---|---|---|
| | | | Dols. |
| METALLIC. | | | |
| Copper (fine, in ore, &c.)† | lb. | 13,300,802 | 1,501,660 |
| Gold | .. | .. | 6,190,000 |
| Iron ore | tons. | 71,451 | 178,716 |
| Lead (fine, in ore, &c.)‡ | lb. | 39,018,219 | 1,396,853 |
| Mercury | " | 688 | 324 |
| Nickel (fine, in ore, &c.)§ | " | 3,997,647 | 1,399,176 |
| Platinum | .. | .. | 6,600 |
| Silver (fine, in ore, &c.) | oz. | 5,558,446 | 3,322,905 |
| Total metallic | | .. | 13,996,234 |
| NON-METALLIC. | | | |
| Asbestos and asbestic | tons | 25,262 | 324,700 |
| Chromite | " | 2,627 | 32,474 |
| Coal | " | 3,876,201 | 7,286,257 |
| Coke* | " | 78,811 | 209,920 |
| Felspar | " | 1,275 | 3,506 |
| Fireclay | " | 1,923 | 5,759 |
| Grindstones** | " | .. | 40,000 |
| Gypsum | " | 239,691 | 244,531 |
| Limestone for flux** | " | .. | 10,000 |
| Mica** | .. | .. | 75,000 |
| Mineral pigments:— | | | |
| Baryta | tons | 571 | 3,060 |
| Ochres | " | 3,905 | 23,560 |
| Mineral water** | galls. | .. | 140,000 |
| Moulding sand | tons | 5,485 | 10,931 |
| Natural gas†† | .. | .. | 325,873 |
| Petroleum‡‡ | barrels | 709,857 | 1,011,546 |
| Phosphate (apatite) | tons | 908 | 3,984 |
| Pyrites | " | 38,910 | 116,730 |
| Salt** | " | .. | 190,000 |
| Sundry minerals, partly estimated (including actinolite, graphite, manganese, soapstone, and tripolite). | .. | .. | 10,000 |
| Cement:— | | | |
| Natural | barrels | 85,450 | 65,893 |
| Portland | " | 119,763 | 209,380 |
| Pottery** | | .. | 125,000 |
| Terra cotta | | .. | 155,595 |
| Total in 1897 | | .. | 23,789,173 |
| 1896 | | .. | 22,590,825 |
| 1890 | | .. | 16,763,353 |
| 1886 | | .. | 10,221,255 |

* Quantity or value of product marketed. The ton used is that of 2,000 lb.

† Copper contents of ore, matte, &c., at 11·29 cents per lb.

‡ Lead contents of ores, &c., at 3·58 cents per lb.

§ Nickel contents of ore, matte, &c., at 35 cents per lb.

Silver contents of ore at 59·79 cents per oz.

* Oven coke, all the production of Nova Scotia and British Columbia.

** Partly estimated.

†† Gross return from sale of gas.

‡‡ Calculated from inspection returns at 100 galls. crude to 42 refined oil and computed at 1·42½ dols. per barrel of 35 imperial gallons. The barrel of refined oil is assumed to contain 42 imperial gallons.

The following table shows the principal changes in production and values for the calendar year 1897, as compared with the figures given in the revised summary for 1896:—

| Product. | Quantity. | | Value. | |
|---|---|---|---|---|
| | Increase. | Decrease. | Increase. | Decrease. |
| METALLIC. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Copper | 11·6 | .. | 46·9 | .. |
| Gold | .. | .. | 122·6 | .. |
| Iron ore | .. | 22·3 | .. | 6·7 |
| Lead | 61·2 | .. | 93·7 | .. |
| Nickel | 17·7 | .. | 17·7 | .. |
| Silver | 73·4 | .. | 54·6 | .. |
| NON-METALLIC. | | | | |
| Asbestos and asbestic | 106·2 | .. | .. | 24·5 |
| Coal | 3·5 | .. | 0·8 | .. |
| Gypsum | 15·8 | .. | 37·3 | .. |
| Natural gas | .. | .. | 17·9 | .. |
| Petroleum | .. | 2·3 | .. | 12·5 |
| Cement | 37·6 | .. | 36·5 | .. |

## EXPORTS OF CHEMICALS, MEDICAL PREPARATIONS, &C., IN 1,000*l*.

*Foreign Office Annual Series, No.* 2138, 1898.

| Year. | Great Britain. | German Empire. | Increase or Decrease for England. | Increase or Decrease for Germany. |
|---|---|---|---|---|
| | £ | £ | £ | £ |
| 1895 | 8,288 | 14,940 | .. | .. |
| 1896 | 8,242 | 15,839 | − 46 | + 899 |
| 1897 | 8,674 | 15,992 | + 432 | + 153 |

## TOTAL EXPORTS OF AMAZONIAN RUBBER, INCLUDING THAT FROM THE STATES OF PARÁ AND AMAZONAS AND THE REPUBLICS OF BOLIVIA AND PERU, DURING THE YEAR 1896–97.

*Foreign Office Annual Series, No.* 2140, 1898.

| —— | Quantity. | Total. |
|---|---|---|
| | Tons. | Tons. |
| Europe— | | |
| Great Britain | 10,331 | |
| France | 2,036 | |
| Italy | 1 | |
| | | 12,368 |
| United States | .. | 9,848 |
| Total | .. | 22,216 |
| Exported during 1895–96 | .. | 21,084 |
| Increase in 1896–97 | .. | 1,132 |

| Sources of Rubber Supply. | Quantity. |
|---|---|
| | Tons. |
| From River Purús | 3,500 |
| " Madeira | 2,200 |
| " Jurua | 2,100 |
| " Solimões | 1,000 |
| " Negro | 700 |
| " Javary and Port of Iquitos | 1,500 |
| From Peru and Bolivia (caucho) | 2,000 |
| " Pará | 9,000 |
| Annual production (about) | 22,000 |

## EXPORTS OF COPPER FROM HUELVA IN 1897.

*Foreign Office Annual Series, No.* 2143, 1898.

This province has only two articles of exportation really important—minerals and wine.

Copper ore appears to have increased, as during the year shipments to England amounted to 162,092 tons, against 141,229 tons in 1896. To France 3,991 tons were sent in 1897, against 114 tons in 1896. Belgium has had 10,681 tons to 4,852 in 1896. The United States, 141,705 tons in 1897 to 126,308 tons in 1896.

On the other hand, Holland only had 121,749 tons in 1897, and 161,099 tons in 1896; Germany, 107,143 tons in 1896 to 89,494 tons in 1897; and Norway, which received nothing this year, had 1,380 tons in 1896, whilst Denmark has had 1,493 tons in the present year against none in 1896.

A decrease is also remarkable in the exportation of precipitate of copper. To the 33,633 tons exported, almost all to Great Britain, in 1896, this year only 32,613 tons have been shipped to England, and 174 tons to France.

The exportation of sulphate of copper in 1896 was limited to 152 tons shipped to France, and 75 tons to Portugal.

In the present year 18 tons were shipped to Great Britain, 1,969 tons to France, 109 tons to Austria, and 261 tons to Portugal.

## CHEMICALS AT WARSAW.

*Foreign Office Annual Series, No.* 2135, 1898.

Poland is not advantageously situated for the development of chemical industry, there being a complete absence of raw materials, which come mostly from Hamburg; but on the other hand, labour is cheap, coal abundant, and the market good, and growing continually, as new districts of the Empire are being thrown open year after year, so that the dividends paid by the different works are very good.

Within the last few years three new branches of the chemical industry were started in Poland, namely, artificial manures, organic dyes, and electro-chemistry.

In 1893 some Belgians, together with some Polish capitalists, started works on a large scale at Lowicz for the production of superphosphates. Up to that time all artificial manure came from Germany, "Thomas slag" being the most popular. Thanks to cleverly worked advertising and the gratis distribution of superphosphates amongst the peasants, this Thomas slag is gradually taking a second place, and finds difficulties in competing with superphosphates. The price of Thomas slag is 7·5 c. per 1 per cent. of soluble phosphoric acid.

The growth of the organic dye industry will, in all probability, be very slow, owing to the absence of raw materials, the coal in this district not being adapted to coking, whilst gas is only known at Warsaw and some four or five provincial towns, so that the sources from which the materials can be obtained are very limited; whilst, on account of the comparatively heavy duty which has to be paid on these materials if imported, it is in many cases more advantageous to import the dyes as such. There are here, however, some large works started by a German firm, who, being the pioneers in this branch, are said to be doing very well.

Electro-chemistry is quite new in Poland. Water-power is practically unknown, and consequently electrolytic plants have to be worked by steam, and although salt (required for the electrolytic process) has to be brought from the Don, it is still an industry which pays well, chiefly owing to the fact that the duty imposed on articles obtained by electrolysis is very heavy. The first works of this kind, which are now in course of construction, have been started by a German firm, part of the capital being subscribed by some Polish capitalists.

### GERMAN MINING INDUSTRY IN 1896.

*Foreign Office Annual Series, No.* 2122, *June* 1898.

According to the official statistics of mines, salt mills, and foundries in the German Empire and Luxemburg, the quantities and values of the main products of the mining industry in 1896 as against 1895 compares as follows:—

*Mineral Production of Germany.*

| Substance. | Measure. | Quantity. 1896. | Quantity. 1895. | Value in Thousand Marks. 1896. | Value in Thousand Marks. 1895. |
|---|---|---|---|---|---|
| I. MINING PRODUCTS. | | | | | |
| Coal | Tons | 85,690,233 | 79,169,276 | 592,976 | 538,895 |
| Brown coal | " | 26,780,873 | 24,788,363 | 60,883 | 58,011 |
| Rock salt | " | 758,867 | 686,940 | 3,249 | 3,108 |
| Kainit | " | 877,885 | 680,174 | 13,299 | 9,609 |
| Other kali salts | " | 902,707 | 841,748 | 11,857 | 11,106 |
| Iron ore | " | 14,162,335 | 12,349,600 | 51,399 | 41,076 |
| Zinc ore | " | 729,942 | 706,423 | 17,023 | 10,577 |
| Lead ore | " | 157,504 | 161,614 | 12,996 | 12,940 |
| Copper ore | " | 717,346 | 633,354 | 16,959 | 15,380 |
| Silver and gold ore | " | 11,320 | 20,845 | 1,712 | 1,708 |
| Pyrites | " | 129,168 | 127,036 | 975 | 976 |
| II. SALTS BY DRAINAGE. | | | | | |
| Common salt (chloride of sodium) | " | 547,486 | 525,396 | 14,650 | 14,253 |
| Chlorate of potassium | " | 174,515 | 154,427 | 22,874 | 19,685 |
| Glauber salt | " | 71,958 | 71,411 | 1,796 | 1,627 |
| Sulphate of potash | " | 19,682 | 19,452 | 3,254 | 3,220 |
| Sulphate of alumina | " | 34,370 | 30,836 | 2,380 | 2,156 |
| III. FOUNDRY PRODUCTS. | | | | | |
| Zinc (in blocks) | " | 153,100 | 150,286 | 47,108 | 41,637 |
| Lead " | " | 113,793 | 111,058 | 25,032 | 22,278 |
| Copper " | " | 29,319 | 25,777 | 29,174 | 23,276 |
| Silver (pure) | Kilos. | 428,429 | 391,979 | 38,872 | 34,403 |
| Gold " | " | 2,487 | 3,547 | 6,916 | 9,878 |
| Sulphuric acid | Tons | 590,887 | 537,928 | 15,124 | 14,855 |
| Sulphate of copper | " | 6,046 | 4,638 | 1,866 | 1,366 |

### ZINC IN POLAND.

*Foreign Office Annual Series, No.* 2135, *June* 1898.

The smelting of zinc was begun in the kingdom of Poland in 1816, and gradually increased until 1837, since when the amount of zinc cast annually has been about 4,000 tons with very slight variations.

Zinc ore is found in the district of Olkusz in the government of Kielce, and in the district of Bendzin in the government of Piotrokoff; there are 28 mines in the first and 32 mines in the second, but of these only four belonging to the "Sosnowice Mining Company" and two belonging to the Crown are worked.

The amount of ore obtained in 1895 by the first company was 21,700 tons, and by the second 40,000 tons, the cost of ore being 8 c. per poud (10*s*. per ton), and the amount of zinc extracted therefrom about 9 per cent., whereas in Silesia 18 per cent. is obtained.

This is explained by the fact that whereas in Silesia and in Europe in general 40 per cent. of sulphide of zinc (mock ore) is added to the ore when it is smelted, which enables a greater quantity of zinc to be extracted from even the poorest ores, this is not done in Russia. This process reduces the price of zinc by 10 c. per poud (12*s*. per ton).

The decline of the zinc industry is chiefly caused by the competition of the works in Silesia, to prevent which the owners of the works in Poland tried to obtain the imposition of a heavy duty on imported zinc; but not having obtained this the Sosnowiec Company has endeavoured to obtain the free importation of foreign sulphide of zinc, by the employment of which it would be possible to obtain the same percentage of zinc as is now obtained in Silesia, besides which it is proposed that both companies should together exploit the mines of sulphide of zinc in the Dombrowa district and so render themselves independent of foreign help.

### SULPHUR IN POLAND.

*Foreign Office Annual Series, No.* 2135, *June* 1898.

Natural sulphur is to be found in the Empire of Russia in six places: in the governments of Astrachan, Kazan, and Samara, and in Daghestan, in Transcaspia, and in the government of Kielce in this consular district.

In the first three places the beds of sulphur have neither been examined geologically nor from a commercial point of view, and remain untouched. In Daghestan there are several rich and large beds of sulphur, which are not worked for want of proper appliances, with the exception of that at Khiontska, from which last year 450 tons were extracted, 120 men being employed. In Transcaspia there are very rich beds, which have been properly surveyed and reported upon scientifically, but not as to their commercial value.

Unique in Russia for their wealth and extent are the sulphur beds at Czarkowsky, in the government of Kielce, besides which, of all the beds already mentioned, they occupy the most favourable position both geologically and commercially.

They lie not far from the Austrian frontier on the bank of the River Nieder, about five versts from its junction with the Vistula, in the district of Pincholf, in the government of Kielce.

These beds were already known in the thirteenth century. Sulphur lies here in beds of a thickness of from 20 to 35 ft., containing 20 to 25 per cent. of pure sulphur, increasing to 50 per cent. as a greater depth is obtained.

These beds were first worked for a short time at the beginning of this century, when this territory still belonged to Austria; they were then abandoned till 1871, when Count Poslowski established works here for extraction of sulphur from the ore by means of overheated steam, but the first year showed that this was a losing undertaking, as the heating by imported coal came too expensive, besides the great cost of necessary materials.

An attempt to dissolve sulphur ore in $CS_2$ for the extraction of sulphur also proved a failure.

It being evident, however, that the attempts to derive profit from the sulphur beds failed only from the want of proper means, some Moscow merchants have already started works for the extraction of sulphur by a process invented by a Mr. Patcanoff.

This method, which is very simple and cheap, is roughly as follows:—The ore is placed in a hermetically closed apparatus filled with water, which is heated by the aid of steam; the sulphur, when dissolved, falls through a kind of filter or sieve into a receptacle, where it solidifies into little balls or prisms; the useless part of the ore remains in the upper part of the apparatus and is considered waste.

There are at the works eight such machines made at Warsaw, and one machine is being put up now for distilling sulphur.

The amount of sulphur imported into Russia is as follows:—

| Year. | Quantity. | Value. |
|---|---|---|
| | Tons. | £ |
| 1891 | 13,048 | 64,000 |
| 1892 | 14,813 | 72,000 |
| 1893 | 21,016 | 89,900 |
| 1894 | 20,540 | 92,500 |
| 1895 | 18,000 | 49,300 |
| 1896 | 18,000 | 48,500 |

Now, however, that Mr. Patcanoff's method has proved to be a practical success, endeavours are being made to get the duty on imported sulphur raised to 20 c. gold per poud, when, the supply of sulphur ore being practically unlimited, it is hoped that Russian sulphur will be able to compete successfully with the imported article.

### SOAP IN ZANZIBAR.

*Foreign Office Annual Series, No. 2129, 1898.*

Common mottled soap, cut into bars of a certain length, commands a ready sale; it is made both in Great Britain and in Germany, but the product of the latter country is in far greater request because it is more malleable and is not so liable to flake off or break when pressed with the finger. English soap has also been known to arrive in a half melted condition, or under weight, or cut into wrong sizes.

## *GENERAL TRADE NOTES.*

### THE WORLD'S COPPER PRODUCTION, 1891-97.*

*Eng. and Mining J., June 25, 1898, 756.*

| Countries. | 1894. | | 1895. | | 1896. | | 1897. | |
|---|---|---|---|---|---|---|---|---|
| | Tons of 2,240 Lb. | Metric Tons. | Tons of 2,240 Lb. | Metric Tons. | Tons of 2,240 Lb. | Metric Tons. | Tons of 2,240 Lb. | Metric Tons. |
| Australasia | 9,000 | 9,144 | 10,000 | 10,160 | 11,000 | 11,176 | 17,000 | 17,272 |
| Austria-Hungary | 2,120 | 2,154 | 1,310 | 1,331 | 1,285 | 1,306 | 1,645 | 1,671 |
| Bolivia | 2,300 | 2,337 | 2,250 | 2,286 | 2,000 | 2,032 | 2,200 | 2,235 |
| Canada | 3,786 | 3,847 | 3,924 | 3,987 | 4,190 | 4,225 | 5,905 | 5,999 |
| Cape of Good Hope: | | | | | | | | |
| Cape Company | 5,150 | 5,233 | 5,350 | 5,436 | 5,470 | 5,558 | 5,290 | 5,375 |
| Namaqua | 1,500 | 1,524 | 1,730 | 1,758 | 1,980 | 2,012 | 2,150 | 2,215 |
| Chile | 21,340 | 21,681 | 22,075 | 22,428 | 23,500 | 23,876 | 21,900 | 22,250 |
| Germany: | | | | | | | | |
| Mansfeld | 14,963 | 15,202 | 18,600 | 18,898 | 18,265 | 18,557 | 17,960 | 18,247 |
| Other mines | 2,210 | 2,245 | 1,695 | 1,722 | 1,800 | 1,829 | 2,185 | 2,220 |
| Italy | 2,500 | 2,540 | 2,500 | 2,540 | 3,400 | 3,454 | 3,400 | 3,454 |
| Japan | 20,050 | 20,371 | 18,430 | 18,725 | 21,000 | 21,336 | 23,000 | 23,368 |
| Mexico: | | | | | | | | |
| Boleo | 10,371 | 10,537 | 10,600 | 10,769 | 9,940 | 10,099 | 10,170 | 10,334 |
| Other mines | 1,100 | 1,122 | 1,170 | 1,189 | 4,032 | 4,098 | 4,868 | 4,945 |
| Newfoundland | 1,900 | 1,930 | 1,800 | 1,829 | 1,800 | 1,829 | 1,800 | 1,829 |
| Norway | 1,785 | 1,813 | 2,685 | 2,728 | 2,500 | 2,540 | 3,450 | 3,505 |
| Russia | 5,000 | 5,080 | 5,000 | 5,080 | 5,000 | 5,080 | 5,000 | 5,080 |
| Spain and Portugal: | | | | | | | | |
| Rio Tinto | 32,689 | 33,215 | 32,985 | 33,513 | 34,501 | 35,055 | 33,900 | 34,442 |
| Tharsis | 11,000 | 11,177 | 12,380 | 12,638 | 11,040 | 11,217 | 12,000 | 12,192 |
| Mason and Barry | 4,200 | 4,267 | 4,100 | 4,166 | 3,381 | 3,435 | 4,300 | 4,369 |
| Sevilla | 1,170 | 1,188 | 1,050 | 1,069 | 1,025 | 1,041 | 810 | 823 |
| Other mines | 4,805 | 4,882 | 4,300 | 4,369 | 3,400 | 3,455 | 3,050 | 3,099 |
| Sweden | 500 | 508 | 515 | 523 | 500 | 508 | 554 | 564 |
| United Kingdom | 100 | 106 | 400 | 406 | 500 | 508 | 550 | 559 |
| United States | 161,510 | 164,194 | 172,524 | 175,294 | 208,760 | 212,112 | 227,763 | 231,421 |
| Venezuela | 2,500 | 2,540 | .. | .. | Nil | Nil | Nil | .. |
| All other countries | 670 | 680 | 635 | 645 | 840 | 853 | 1,200 | 1,219 |
| Totals | 324,850 | 330,132 | 334,268 | 339,699 | 381,100 | 387,207 | 412,050 | 418,677 |

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* The figures in this table are taken from the annual metal circular of Henry R. Merton and Co., except where returns have been received by *The Mineral Industry* direct from producers.

### Salt in the Isle of Man.

*Chem. Trade J., July* 9, 1898, 23.

The Liverpool syndicate which has been conducting borings in the Isle of Man to find, if possible, the continuation of the seam of coal which dips under the sea at Whitehaven, has failed in its object, but it is nevertheless fortunate in having come across immense beds of rock salt, which are so promising that plans have been drawn up for the constitution of a port, estimates for carrying-steamers have been obtained, and the old shipyard at Ramsay has been purchased with the intention of establishing a great salt industry.

### Rubber Plant in British Central Africa.

*Board of Trade J., July* 1898, 9.

The Director of the Royal Gardens at Kew identifies a rubber plant, found in abundance throughout the West Nyassa district, British Central Africa, as *Landolphia florida*, one of the most important of the so-called "vines" producing rubber in Tropical Africa.

According to a report on the sample received, the rubber is of very good quality, and the sample has been properly prepared. Its value to-day is 3*s.* to 3*s.* 1*d.* per lb., and it is stated that any quantity of this rubber, provided it is as clean as the sample in question, would find a ready market in London for some years to come at a minimum price of 2*s.* 6*d.* to 2*s.* 9*d.* per lb.

### Borax in Peru.

*Eng. and Mining J., July* 9, 1898, 42.

The borax industry in Peru appears to be assuming some importance. In 1896, 7,350 tons, valued at 36,970*l.*, were exported, while in 1897, 12,464 tons, valued at 62,420*l.*, were shipped from Mollendo. The deposits are found about 20 miles in the interior from Arequipa. This product has greatly increased and improved trade in that city, owing to the number of mules and donkeys employed in the carriage down from the deposits to the railway station at Arequipa, and to the impetus given to agriculture on the adjacent lands by the cultivation of alfalfa (lucerne) for the maintenance of these animals, the value of alfalfa having increased fourfold in as many years ; this is now one of the greatest interests that the department of Arequipa has to depend on, and apparently the supply of borax is inexhaustible.

### Patents in Austria.

*Eng. and Mining J., July* 9, 1898, 34.

It is stated officially that an important amendment of the patent law of Austria will come into force in January next, says the *Engineer*. Patents will be granted for 15 years for industrial inventions, excluding articles of food, medicines, disinfectants, or chemical products not produced by a new technical process. Prior publication in another country will prevent a valid patent being obtained in Austria. The Patent Office has the power to grant compulsory licenses if the inventor declines to allow his invention to be used, but the onerous and unsatisfactory provisions as to working patents which at present exist in Austria will be practically done away with. Annual renewal fees will be charged on an ascending scale.

### Acetylene Black.

*Eng. and Mining J., July* 9, 1898, 35.

The soot of the acetylene flames represents a valuable product, especially for the colour industry. When acetylene gas is burned with a smoking flame, three to four times as large a quantity of soot is obtained as from the same quantity of mineral oil. The acetylene soot is very light and exhibits an absolutely black colour without any tinge of brown. Besides, all tarry admixtures and other substances contained in lamp black, &c., are absent from the soot. It is very bulky, and will be specially adapted for India ink, as well as for colours used in printing and lithography. A recent French patent issued to E. Hubon gives three methods for the production of the colour. According to the first the acetylene gas is kept in a steel cylinder under two atmospheres pressure, and is then ignited through an electric spark or a glowing platinum wire by way of explosion. The final pressure of the resulting hydrogen does not exceed 12 atmospheres, so that the danger of an explosion is excluded if steel cylinders of corresponding strength are employed. This method has the advantage that the theoretic yield of carbon is obtained from the acetylene, the product being of the greatest purity.

### The Compulsory Licensing of Patents: An Important Precedent.

*Chem. Trade J., July* 16, 1898, 39.

The Board of Trade has made the following order in the matter of the petition of Messrs. Levinstein, Limited, and Ivan Levinstein to the Board of Trade (hereinafter called the petitioners) for an order for compulsory licences under certain foreign patents :—

Whereas, by section 22 of the Patents, Designs, and Trade Marks Act, 1883, it is enacted : "If, on the petition of any person interested, it is proved to the Board of Trade that by reason of the default of a patentee to grant licences on reasonable terms—

"(*a*) The patent is not being worked in the United Kingdom, or (*b*) the reasonable requirements of the public with respect to the invention cannot be supplied, or (*c*) any person is prevented from working or using to the best advantage an invention of which he is possessed,"

The Board may order the patentee to grant licences on such terms as to the amount of royalties, security for payment, or otherwise, as the Board, having regard to the nature of the invention and the circumstances of the case, may deem just; and any such order may be enforced by mandamus.

And whereas, on the 11th June 1889, a patent numbered 9642 was granted to Oliver Imray, of 28, Southampton Buildings, London, Patent Agent, for an invention communicated to him by The Farbwerke vormals Meister, Lucius, and Brüning, of Hoechst-am-Maine, in the German Empire (herein-after called the patentees), relating to the production of oxysulphonic acids of naphthalin.

And whereas, on the 24th May 1892, the said patent was duly assigned by the said Oliver Imray to the patentees, which said assignment was, on the 27th May 1892, duly registered at the Patent Office.

And whereas, on the 26th September 1889, another patent, numbered 15,176, was also granted to the said Oliver Imray, for an invention, in like manner communicated to him by the patentees, relating to improvements in the production of amido-naphthol-monosulphonic acids and the manufacture of their diazo compounds and azo colouring matters therefrom.

And whereas, on the 24th May 1892, the said patent was duly assigned by the said Oliver Imray to the patentees, which said assignment was, on the 27th May 1892, duly registered at the patent office.

And whereas the petitioners presented a petition to the Board of Trade under the said section for an order that the patentees, under the said Letters Patent, No. 9642, of the 11th June 1889, and No. 15,176, of the 26th September 1889, should grant to the petitioners a licence under, or in respect of, the inventions described and claimed in and by the specifications of the said respective Letters Patent.

And whereas, on consideration of the said petition and of the matters therein mentioned, it has been proved to the Board of Trade that the said petitioners are persons interested in the matter of the said petition, and that by reason of the default of the patentees to grant licences on reasonable terms, the said patents are not being worked in the United Kingdom, and that by reason of the default aforesaid the said petitioners are prevented from working and using to the best advantage certain inventions of which they are possessed.

Now, therefore, the Board of Trade, in exercise of the power conferred upon them by section 22 of the Patents, Designs, and Trade Marks Act, 1883, and of all other powers enabling them in this behalf, do hereby order as follows:—

1. That a licence, to take effect from the date of this order, and in the form set forth in the schedule hereto, be forthwith granted by the patentees to the petitioners to make, use, exercise, and vend, within the United Kingdom, the inventions described and claimed in and by the specifications of the said Letters Patent, No. 9642 of 1889 and No. 15,176 of 1889, for the unexpired residues of the respective terms of the said Letters Patent, at a royalty of one halfpenny for each pound weight, avoirdupois, of products made by the licensees under the said Letters Patent or either of them, but so that in each year a minimum royalty of 250*l.* per annum shall be paid by the licensees, and the patentees shall forthwith deposit such licence duly executed by them with the Board of Trade.

2. The said petitioners, before the 6th day of August 1898, shall execute and deposit with the Board of Trade a counterpart licence in the form aforesaid, and in default of their doing so this order shall be of no effect.

(Signed) COURTENAY BOYLE,
Secretary,
Dated this 6th day of July 1898. Board of Trade.

### REPORT OF THE PETROLEUM COMMITTEE.

*Pharm. J.*, July 16, 1898, 72.

After an inquiry extending over four sessions, the Select Committee on Petroleum has now concluded its labours, and Mr. Jesse Collings, Chairman of the Committee has presented its report to the House of Commons. The following is a summary of the recommendations agreed to:—

1. That the present law affecting petroleum spirit, not being adequate for public safety, should be amended in the manner specified in the report.
2. To adopt a flash point of 100° (Abel close test) as the dividing line between petroleum oil and petroleum spirit.
3. Legislation to control the storage, transport, and sale of petroleum generally and admixtures of the same with other substances, certain heavy oils being exempted.
4. To provide for an efficient system of testing.
5. To provide for adequate supervision and administration by local authorities.
6. That official inquiries be made into the causes of accidents arising from the storage, transport, or use of inflammable liquids.
7. Statutory powers to enable the Secretary of State to issue orders affecting the manufacture and sale of lamps.
8. To spread information among the public as to the nature of petroleum and the management of petroleum lamps.

The Committee added to these recommendations the following conclusions:—

"That to place such legislative restrictions on petroleum oil below 100° (Abel close test) as are placed on petroleum spirit would have the effect of preventing the use of such oil for domestic or trade purposes; that the effect of such legislation would be to materially raise the cost of petroleum to the consumer; that it is in accordance with the evidence that if immunity from accidents is to be secured, it will be necessary to prevent the use of oil below 100°; that the number of lamp accidents has not increased out of proportion to the vast increase in the number of lamps used; and that if freedom from accidents is to be secured, ordinary care must be exercised in the use of petroleum, whether of 73° or 100° flash point."

### SPIRIT FOR CHEMICAL MANUFACTURERS.

*Chem. and Druggist, July* 23, 1898, 110.

At the annual meeting of the London Chamber of Commerce, held at Salter's Hall on Monday, July 18th, the Chemical Trade Section reported *inter alia* what had been done in regard to the spirit concessions. The Joint Committee appointed in 1896 to consider the law relating to spirits and the regulations thereunder as affecting chemical manufacturers had held several meetings. After considerable discussion, the committee made a number of recommendations, which it was agreed should be laid before the Inland Revenue authorities at Somerset House. A conference was accordingly arranged between the committee and the Chief Inspector of the Excise Department and Dr. Thorpe (the principal of the laboratory). The difficulties under which chemical manufacturers in this country laboured, as compared with the advantages enjoyed by their German and American competitors, were explained by members of the committee. The Chief Inspector pointed out the obstacles in the way of adopting the recommendations of the committee. He intimated that the Inland Revenue authorities would do everything in their power to encourage the chemical trade of this country, and suggested that the committee might gather information which would enable him to take steps to meet their views. Further meetings of the committee had been held, and they were still collecting information and statistics. Information, however, had been received to the effect that, in consequence of the representations made to the Inland Revenue authorities by the Joint Committee, certain concessions had been granted to chemical manufacturers, which would place them on more equal terms with their foreign competitors.

## *BOARD OF TRADE RETURNS.*

### SUMMARY OF IMPORTS.

| Articles. | Month ending 30th June. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,881,128 | 1,994,529 |
| Chemicals and dyestuffs | 401,507 | 479,388 |
| Oils | 620,226 | 735,273 |
| Raw materials for non-textile industries. | 5,025,197 | 4,487,625 |
| Total value of all Imports | 36,321,869 | 39,032,305 |

### SUMMARY OF EXPORTS.

| Articles. | Month ending 30th June. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 2,793,784 | 2,820,557 |
| Chemicals and medicines | 628,031 | 678,061 |
| Miscellaneous articles | 2,751,040 | 2,801,203 |
| Total value of all exports | 19,089,907 | 19,413,696 |

### IMPORTS OF METALS FOR MONTH ENDING 30TH JUNE.

| Articles. | | Quantities. | | Values. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| Copper:— | | | | £ | £ |
| Ore | Tons | 8,694 | 5,788 | 46,507 | 38,732 |
| Regulus | " | 7,567 | 6,696 | 176,758 | 183,741 |
| Unwrought | " | 5,561 | 7,078 | 274,157 | 369,634 |
| Iron:— | | | | | |
| Pig | " | 15,873 | 8,904 | 51,945 | 37,835 |
| Ore | " | 562,834 | 484,261 | 124,869 | 360,371 |
| Bolt, bar, &c. | " | 6,761 | 9,008 | 51,662 | 72,968 |
| Steel, unwrought | " | 4,344 | 3,703 | 28,300 | 22,558 |
| Lead, pig and sheet | " | 12,620 | 15,581 | 149,972 | 209,699 |
| Pyrites | " | 47,356 | 51,141 | 80,450 | 80,852 |
| Quicksilver | Lb. | 911,160 | 931,500 | 89,481 | 91,390 |
| Silver ore | Value £ | .. | .. | 86,463 | 90,849 |
| Tin | Cwt. | 47,768 | 30,937 | 145,357 | 102,237 |
| Zinc | Tons | 6,130 | 6,524 | 111,368 | 111,457 |
| Other articles | Value £ | .. | .. | 163,598 | 213,206 |
| Total value of metals | | .. | .. | 1,881,128 | 1,994,529 |

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898 |
| | | | £ | £ |
| Alkali ............ Cwt. | 28,325 | 29,657 | 10,253 | 16,454 |
| Bark (tanners', &c.) „ | 36,168 | 51,453 | 12,394 | 16,482 |
| Borax ............ „ | .. | 34 | .. | 22 |
| Brimstone ........ „ | 44,100 | 54,010 | 9,537 | 11,746 |
| Chemicals ...... Value £ | .. | .. | 120,123 | 132,694 |
| Cochineal ......... Cwt. | 466 | 673 | 2,951 | 3,931 |
| Cutch and gambier Tons | 1,802 | 1,584 | 30,281 | 27,804 |
| Dyes:— | | | | |
| Alizarin ....... Value £ | .. | .. | 16,391 | 20,087 |
| Anilin and other „ | .. | .. | 32,544 | 39,157 |
| Indigo ............ Cwt. | 1,708 | 1,844 | 26,674 | 29,551 |
| Nitrate of potash . „ | 38,331 | 23,727 | 26,369 | 17,909 |
| Valonia .......... Tons | 1,426 | 2,862 | 14,818 | 31,422 |
| Other articles ... Value £ | .. | .. | 99,122 | 132,129 |
| Total value of chemicals | .. | .. | 401,507 | 479,388 |

### IMPORTS OF OILS FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut ......... Cwt. | 15,096 | 24,751 | 16,918 | 27,870 |
| Olive .............. Tuns | 1,910 | 2,550 | 59,444 | 84,069 |
| Palm .............. Cwt. | 126,324 | 97,355 | 125,341 | 103,414 |
| Petroleum ........ Gall. | 14,431,962 | 15,943,236 | 261,917 | 266,005 |
| Seed .............. Tons | 1,622 | 2 552 | 33,197 | 50,261 |
| Train, &c. ......... Tuns | 1,141 | 2,244 | 19,318 | 41,457 |
| Turpentine ....... Cwt. | 29,082 | 67,159 | 29,894 | 73,181 |
| Other articles .. Value £ | .. | .. | 74,197 | 89,016 |
| Total value of oils ... | .. | .. | 620,226 | 735,273 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,847 | 4,054 | 3,186 | 7,834 |
| Bristles .......... Lb. | 255,375 | 369,160 | 34,021 | 44,544 |
| Caoutchouc ...... Cwt. | 25,286 | 36,290 | 264,635 | 423,032 |
| Gum:— | | | | |
| Arabic ......... „ | 6,864 | 5,693 | 10,363 | 13,230 |
| Lac, &c. ....... „ | 17,566 | 12,085 | 73,697 | 40,987 |
| Gutta-percha .... „ | 3,471 | 6,885 | 34,777 | 72,325 |
| Hides, raw:— | | | | |
| Dry ............ „ | 51,034 | 35,517 | 141,838 | 101,222 |
| Wet ........... „ | 60,888 | 62,820 | 125,794 | 133,483 |
| Ivory ............ „ | 1,002 | 1,126 | 39,704 | 45,860 |
| Manure:— | | | | |
| Guano ......... Tons | 1,075 | 140 | 3,650 | 476 |
| Bones ......... „ | 4,932 | 1,714 | 12,164 | 6,885 |
| Nitrate of soda ... „ | 8,948 | 5,700 | 67,398 | 42,452 |
| Phosphate of lime „ | 28,051 | 24,964 | 42,578 | 37,489 |
| Paraffin .......... Cwt. | 48,847 | 56,470 | 44,095 | 50,394 |
| Linen rags ....... Tons | 1,984 | 2,292 | 19,890 | 20,279 |
| Esparto .......... „ | 18,268 | 15,512 | 70,671 | 57,501 |
| Pulp of wood:— | | | | |
| Chemical ...... „ | 37,388 | 16,421 | 182,172 | 116,938 |
| Mechanical .... „ | | 15,334 | | 43,363 |
| Rosin ............ Cwt. | 192,984 | 181,232 | 46,909 | 41,563 |
| Tallow and stearin „ | 246,330 | 228,493 | 229,678 | 236,245 |
| Tar .............. Barrels | 2,588 | 2,165 | 1,110 | 1,228 |
| Wood:— | | | | |
| Hewn ......... Loads | 238,732 | 191,568 | 498,908 | 443,925 |
| Sawn .......... „ | 898,272 | 635,687 | 2,077,011 | 1,537,370 |
| Staves ......... „ | 10,915 | 12,743 | 37,525 | 49,046 |
| Mahogany ..... Tons | 3,730 | 4,541 | 35,212 | 41,879 |
| Other articles .... Value £ | .. | .. | 928,211 | 878,075 |
| Total value ....... | .. | .. | 5,025,197 | 4,487,625 |

Besides the above, drugs to the value of 74,412*l*. were imported, as against 68,670*l*. in June 1897.

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass ............ Cwt. | 10,622 | 7,947 | 43,750 | 36,646 |
| Copper:— | | | | |
| Unwrought .... „ | 29,753 | 45,916 | 76,667 | 124,146 |
| Wrought ...... „ | 23,442 | 19,355 | 71,656 | 60,874 |
| Mixed metal ... „ | 13,126 | 15,485 | 31,584 | 38,400 |
| Hardware and cutlery. Value £ | .. | .. | 173,796 | 119,970 |
| Implements ..... „ | .. | .. | 100,125 | 114,695 |
| Iron and steel ... Tons | 317,309 | 275,034 | 2,029,863 | 1,898,265 |
| Lead ............ „ | 3,260 | 3,494 | 42,431 | 50,989 |
| Plated wares ... Value £ | .. | .. | 28,322 | 32,889 |
| Telegraph wires . „ | .. | .. | 88,340 | 145,281 |
| Tin ............ Cwt. | 8,609 | 11,999 | 27,901 | 41,428 |
| Zinc ............ „ | 15,773 | 16,524 | 11,686 | 14,365 |
| Other articles .. Value £ | .. | .. | 68,163 | 94,953 |
| Total value ....... | .. | .. | 2,793,784 | 2,820,557 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali ........... Cwt. | 440,186 | 278,797 | 112,532 | 75,478 |
| Bleaching materials „ | 95,958 | 109,045 | 29,998 | 29,513 |
| Sulphate of copper Tons | 3,606 | 4,124 | 59,474 | 66,045 |
| Chemical manures „ | 18,902 | 31,965 | 103,849 | 165,191 |
| Medicines ....... Value £ | .. | .. | 92,015 | 97,739 |
| Other articles ... „ | .. | .. | 230,163 | 244,095 |
| Total value .......... | .. | .. | 628,031 | 678,061 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH JUNE.

| Articles. | Quantities. | | Values. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder ...... Lb. | 936,200 | 595,000 | 21,308 | 16,879 |
| Military stores .. Value £ | .. | .. | 188,219 | 158,317 |
| Candles .......... Lb. | 1,784,300 | 1,820,700 | 24,045 | 25,719 |
| Caoutchouc ..... Value £ | .. | .. | 86,147 | 105,293 |
| Cement .......... Tons | 32,138 | 31,996 | 52,633 | 60,511 |
| Products of coal Value £ | .. | .. | 133,323 | 134,369 |
| Earthenware ... „ | .. | .. | 169,365 | 150,958 |
| Stoneware ...... „ | .. | .. | 12,414 | 12,129 |
| Glass:— | | | | |
| Plate .......... Sq. Ft. | 94,706 | 123,791 | 5,559 | 7,286 |
| Flint .......... Cwt. | 6 149 | 7,099 | 13,305 | 16,355 |
| Bottles ........ „ | 65,333 | 68,077 | 31,168 | 33,121 |
| Other kinds .... „ | 17,405 | 18,461 | 15,135 | 16,580 |
| Leather:— | | | | |
| Unwrought .... „ | 11,575 | 15,958 | 106,026 | 137,407 |
| Wrought ..... Value £ | .. | .. | 34,503 | 30,801 |
| Seed oil .......... Tons | 4,216 | 3,289 | 70,674 | 58,267 |
| Floorcloth ..... Sq. Yds. | 1,899,300 | 1,826 600 | 77,879 | 78,972 |
| Painters' materials Val. £ | .. | .. | 129,656 | 151,535 |
| Paper ........... Cwt. | 78,449 | 83,522 | 122,009 | 122,488 |
| Rags ............. Tons | 4,288 | 5,081 | 21,479 | 25,825 |
| Soap ............. Cwt. | 61,008 | 83,068 | 62,111 | 87,476 |
| Total value .......... | .. | .. | 2,751,040 | 2,801,203 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### Applications.

13,305. G. F. Redfern.—From H. Schaffstaedt, Germany. A method of and apparatus for condensing. June 14.

13,406A. C. Hoy. Improvements in and relating to bottles for storing unstable salts or any other unstable substances or liquids. Filed 15th July. Date claimed 16th June 1898.

13,463 T. W. Bloxam. Improvements in apparatus for supplying liquids in proportion to each other. June 16.

13,618. S. S. Bromhead.—From Möller and Pfeifer, Germany. Apparatus for drying. Complete Specification. June 13.

13,619. S. S. Bromhead.—From Möller and Pfeifer, Germany. Improvements in cell cylinders for drying apparatus. Complete Specification. June 18.

13,698. G. de Roussy de Sales. Improvements in apparatus for mixing gases. June 20.

14,131. C. D. Abel.—From Strassfurter Chemische Fabrik vormals Vorster and Grüneberg Actien-Gesellschaft, Germany. Improved means for preventing back-draught in pyrites furnaces. June 25.

14,228. W. Hinchliffe. Improvements in apparatus for drying, purifying, separating oil or grease from and condensing steam, and for heating and purifying water for boiler-feed and for other manufacturing purposes. June 28.

14,400. T. H. Bristow and Co., Ltd., and W. R. Renshaw. Improvements in filtering apparatus. June 29.

14,409. G. S. de Ritter. Improvements in machines for the more rapid and effectual drying of materials. June 30.

14,488. J. T. Waine. Improvements in apparatus for distilling water. June 30.

14,718. J. B. Bourseau. Improvements in metallic capsules for storing liquids or gases under pressure. Complete Specification. July 4.

14,783. G. W. Simpson. Improvements in maximum thermometers. July 5.

15,151. M. Otto. *See* Class XVII.

15,152. M. Otto. Improvements in apparatus for the preparation of sterilised ice in blocks or in bottles and the sterilisation in a cold condition and at the ordinary pressure of liquids generally. Complete Specification. July 9.

15,173. A. Grounds. Cylindrical vessels used for containing gases, liquids, &c. under pressure. July 11.

15,331. H. W. Abbott. Improved process and apparatus for refrigerating and cooling air. Complete Specification. July 12.

15,344. J. G. Gordon. Improvements in melting furnaces. July 12.

15,409. J. Wilson and A. Masson. Improvements in apparatus suitable for treating or softening water. July 13.

15,489. C. Erith. Improvements in and relating to dryers for granular pulverulent and pasty materials and the like. July 14.

15,651. R. G. Nash. Improved means applicable for use in the sterilization of liquids or substances. July 16.

15,652. V. E. J. Durafort. Improvements in means or apparatus for charging liquids with gases. July 16.

### Complete Specifications Accepted.

1897.

14,468. A. J. Boult.—F. Würtenberger, Italy. Regenerative furnaces. June 22.

15,107. S. C. Davidson. Apparatus for applying aqueous vapour, or moist air, or gases to substances; especially applicable to softening, limping, or withering tea leaf, moistening or "ordering" tobacco leaf, or for similar treatment or other analogous substances. June 22.

18,311. L. A. Chevalet and W. Boby. Apparatus for heating and purifying water. July 6.

18,819. W. R. Renshaw. Apparatus suitable for cooling liquids and condensing. July 20.

18,950. P. Boimare. Smokeless furnaces. June 29.

19,196. E. Stephenson. Condensers. July 6.

1898.

7178. K. Abraham. Filters. June 29.

9606. I. Fokchaner. Filters. June 29.

10,132. P. A. Newton.—From The Baker and Adamson Chemical Company, United States. Apparatus for distillation of acids and other corrosive substances. June 22.

11,364. P. Reuss. Centrifugal machines. June 22.

## II.—FUEL, GAS, AND LIGHT.

### Applications.

7281. A. S. Rosinski and Madame V. E. Ducroix *née* M. M. Pierguin. A new composition of matter for automatically lighting gas and for other purposes. Filed June 15. Date claimed, March 25, 1898.

13,164. P. Naef and R. S. Garton. Process and apparatus for producing coke-gas and by-products. June 13.

13,165. P. Naef and R. S. Garton. Process and apparatus for producing coke-gas and by-products. June 13.

13,185. W. Donaldson. Improvements in methods of vaporising inflammable oils and storing the vapour or gas produced at constant pressure, for use in heating, lighting, and gas motors. June 13.

13,232. B. H. Thwaite and H. Allen. Improved means for effecting the slow action of water upon calcic carbide, and making the same inoffensively odorous. June 14.

13,263. E. Rischgitz. Improvements relating to the treatment of peat. June 14.

13,387. C. D. Abel.—From P. Lipcke, Germany. Improvements in apparatus for generating acetylene. Complete Specification. June 15.

13,504. A. Koch. Process for manufacturing durable incandescent illuminating bodies. Complete Specification. June 17.

13,598. E. K. Hutton and W. Hutton. An automatic acetylene gas generator. June 18.

13,636. Sir C. S. Forbes, Bart. Improvements in or applicable to acetylene generators. June 18.

13,712. V. Toile. New or improved means for economising coal. June 20.

13,830. A. J. Boult.—From O. Payan, France. Improvements in or relating to apparatus for producing acetylene gas. Complete Specification. June 21.

13,875. E. J. Pennington. Improvements in or relating to the manufacture of gas. June 22.

13,881. L. von J. Jarocki and Kiezerowski. *See* Class XII.

13,987. W. V. Ebert. Improvements in lamps for generating and burning acetylene gas. June 23.

13,990. G. Wiltshire. Improvements in apparatus for producing acetylene gas. June 24.

14,037. G. Zschocke. Improvements in gas scrubbers. June 24.

14,010. J. J. Shedlock. Improvements in or relating to gas-producing apparatus. June 24

14,049. K. E. Rosenthal. Improvements in or connected with generators for acetylene gas. Complete Specification. June 24.

14,050. J. Billwiller and K. E. Rosenthal. Improvements in generators for the production of acetylene gas. Complete Specification. June 24.

14,119. W. T. McGarry. Improvements in the treatment or carbonising of peat or other decayed vegetable matter, and apparatus therefor. June 25.

14,127. H. J. Dowsing. Improvements in apparatus for utilising radiant heat. June 25.

14,139. Sir C. S. Forbes, Bart. Improvements in apparatus connected with the manufacture of acetylene gas. June 25.

14,184. A. E. Stamm. Improved mass for impregnating incandescent mantles. Complete Specification. June 27.

14,197. W. P. Thompson.—From H. Sez, France. Improvements in apparatus for producing acetylene. Complete Specification. June 27.

14,210. A. J. Boult.—From Suret et Cie., France. Improvements in or relating to apparatus for use in the production of acetylene gas. June 27.

14,215. E. Philpott. Improvements in the manufacture and production of candles. June 27.

14,240. J. W. Dougal. Improved manufacture of charcoal. June 28.

14,285. W. P. Thompson.—From R. M. Snyder, United States. Improvements in or relating to apparatus for generating and burning gas from liquid hydrocarbons. Complete Specification. June 28.

14,295. Sir C. S. Forbes, Bart. Improved means and apparatus for enriching coal-gas or the like. June 28.

14,307. H. Böhm. Improvements in and relating to means for producing ozone. June 28.

14,372. W. Beth. An improved process for extracting coal-dust from flue gases. Complete Specification. June 29.

14,386. E. N. Trump. Improvements in means or apparatus for receiving and transporting the discharged contents of coke ovens and the like. June 29.

14,432. The British Acetylene Gas Generator Company, Ltd., and H. K. Spence. Improvements in acetylene gas generators. Complete Specification. June 30.

14,438. S. J. Ingram. Apparatus for charging coal-gas and other retorts. June 30.

14,450. E. Böhm. Improvements in and relating to means for producing ozone. June 30.

14,463. P. G. de Schodt. Improvements in incandescent gas lighting. Complete Specification. June 30.

14,557. J. Ashworth. Improvements in treating or enriching oils or spirits for illuminating and heating purposes. July 1.

14,567. M. Davoren, J. Belton, and J. Davoren. Improvements in and in connection with the manufacture of illuminating gas. July 1.

14,590. F. C. Askham. Means for the generation and temporary storage in a compressed state of gas or vapour, caused by the chemical change produced in various substances, gases, or liquids, by their combustion or explosion. July 2.

14,616. M. Fournier. Improvements in or relating to apparatus for the production of acetylene gas. Complete Specification. July 2.

14,707. Sir C. S. Forbes, Bart. Improvements in acetylene generators. July 4.

14,713. H. H. Lake.—From La Compagnie Continentale d'incandescence et de chauffage, Belgium. Apparatus for generating acetylene gas. Complete Specification. July 4.

14,729. G. Gastaldi. Improvements in apparatus for generating acetylene. Complete Specification. July 4.

14,858. J. E. Scarth. Improvements in or relating to apparatus for the generation of acetylene gas. July 6.

14,895. W. H. Akester. Improvements in the production of ethylene. July 6.

14,919. D. J. V. Praag. An invention to neutralise the smell of calcium carbide and acetylene gas. July 7.

14,972. H. E. A. Cousin. Improvements in apparatus for the production of acetylene gas. Complete Specification. July 7.

15,014. H. Sutcliffe. Improvements in acetylene gas generators. July 8.

15,020. E. Henriquez. Improvements in acetylene generating apparatus. Complete Specification. July 8.

15,138. W. H. Akester. Improvements in the production of ethylene. July 9.

15,179. E. Deemouly. Improvements in generating acetylene gas. Filed July 11. Date applied for March 29, 1898, being date of application in France.

15,207. J. Mactear. Improvements in obtaining cyanogen compounds. July 11.

15,212. G. Dillberg. Improvements in the treatment of calcium carbide whereby the conversion of same into acetylene gas may be retarded without the aid of any mechanical devices. Complete Specification. July 11.

15,213. G. Dillberg. Improvements in protective coverings for compressed cakes composed of granulated calcium carbide, or an admixture of calcium carbide with other ingredients. Complete Specification. July 11.

15,214. G. Dillberg. Improvements in means for using calcium carbide for the purpose of generating acetylene gas. Complete Specification. July 11.

15,241. J. Boss. A new or improved apparatus for generating and utilising acetylene gas. July 12.

15,367. J. A. Armitage and E. Ramsden. The safety acetylene generator. July 13.

15,394. P. Myers. Improvements in or relating to acetylene or similiar generators. July 13.

15,396. D. M. Baumgarten. Acetylene gas generators. Complete Specification. July 13.

15,423. M. Bohne. Improvements in acetylene gas generators. July 13.

### Complete Specifications Accepted.

1897.

17,482. N. Reggiani and A. Chrisini. Acetylene gas generators. July 20.

17,644. H. H. Lake. From Fellner and Ziegler, Germany. Apparatus for purifying gas by washing or scrubbing. June 29.

17,749. V. B. Lewes. Apparatus for the manufacture of illuminating gas. July 20.

17,793. J. G. A. Kitchen. Acetylene generators. July 6.

18,355. J. C. Bull.—From Jossé and Defays, France. Manufacture of acetylene gas. June 29.

18,907. A. Denich. Portable apparatus for producing acetylene gas. July 6.

18,971. J. R. Wigham. Manufacturing acetylene gas from calcium carbide. June 22.

18,989. H. Maxim and W. H. Graham. Method and apparatus for the production of calcium carbide; applicable also to other purposes. July 20.

20,142. F. A. Keifier. Apparatus for the automatic supply of carbide of calcium in the production of acetylene gas. July 6.

20,184. W. Dancer. Production of gas for illuminating and heating purposes, and of certain by-products. July 20.

20,319. E. Saizenberg. Incandescent gas lighting. June 29.

25,318. J. W. Grainger. Manufacture of fire-lighters. July 20.

30,145. A. J. Boult.—From The Firm Chemische Fabrik von Max Jasper, Germany. Manufacture of incandescent bodies for illuminating purposes. July 20.

1898.

8030. A. E. R. Söhnel and A. R. Zehner. Acetylene gas apparatus. July 20.

8555. E. Tooth. Illuminating and heating gas. July 20.

9545. A. Williamson. Acetylene gas generators. June 22.

10,305. J. H. Schülke. Acetylene gas generator. June 29.

10,763. J. Sartig. Treating calcium carbide for use in acetylene generators. June 22.

12,356. W. H. A. Sieverts. Method of and apparatus for forming incandescent bodies for gas-lighting. July 20.

## IV.—COLOURING MATTERS AND DYES.

### Applications.

13,167. A. Bang.—From Dahl and Co. A black cotton dyestuff, and process for the production of the same. June 13.

13,201. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. A new manufacture of azo-dyes. June 13.

13,453. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture of aromatic compounds by means of hydrocyanic acid and certain derivatives thereof. June 16.

13,561. A. G. Green, A. Mayenberg, and The Clayton Aniline Company, Ltd. Improvements in the manufacture and production of colouring matters. June 17.

13,564. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of oxy-anthra-quinoni-sulpho acids. June 17.

13,724. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of acetylleuco-methylene blue and of acetylleuco-ethylene blue. June 20.

13,797. H. R. Vidal. Improvements in the manufacture of colouring matters. Filed June 21. Date applied for, Nov. 27, 1897, being date of application in France.

14,251. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of secondary disazo dyes from nitro-amidophenolsulpho acids. June 28.

14,252. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of azo-colouring matters, and materials for use therefor. June 28.

14,253. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of azo-colouring matters, and materials for use therefor. June 28.

14,364. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. New process for producing carbonic acid ethers, and intermediate products therefor. June 29.

14,465. A. Ehrenberg. A process for the production of methylmorphine (codein) and ethylmorphine. June 30.

14,468. P. C. Bonn. Improvements in dyes and dyeing. June 30.

14,533. R. Holliday and Sons, Ltd., and A. Dickson. Improvements in the manufacture of bases of the diphenylmethane group, and colouring matters therefrom. July 1.

14,627. S. M. Angel. Improved dye for textile fabrics, feathers, and the like. July 2.

14,961. J. Imray.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Improvements in the manufacture of colouring matters of the safranine series. July 7.

14,962. J. Imray.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of sulpho-acids of aposafranines. July 7.

### Complete Specifications Accepted.

1897.

12,326. S. Herzberg. Vegetable blue, and process for manufacturing the same. March 9.

15,587. H. Koechlin. Treating paranitraniline reds and analogous colours, when applied on vegetable fibres or silks, to render them fast to light. June 22.

16,496. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Manufacture and production of new colouring matters from animal fibre. June 22.

17,018. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of dyestuffs for cotton. June 29.

18,017. M. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement Gilliard P. Monnet et Cartier, France. Manufacture of dyestuffs. July 6.

18,376. I. Levinstein and Levinstein, Ltd. Manufacture or production of new colouring matters. July 6.

19,622. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture of anthraquinone dyestuffs. July 6.

19,634. O. Imray.—From H. Schneider, Germany. Manufacture of new amido base and new direct dyeing polyazo dyestuffs therefrom. July 6.

1898.

7659. O. Imray.—From The Society of Chemical Industry, Basle. Manufacture of new trisazo colouring matters capable of dyeing unmordanted cotton and of yielding blue shades by development on the fibre. June 29.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

14,472. S. Pitt. From L. Cassella and Co., Germany. Improvements in the manufacture of textile fabrics having two coloured and crape effects. June 30.

14,503. G. D. Wild. Improvements in drying textile fabrics by means of hot or cold air or gases. July 1.

15,491. J. E. G. Méran. Method and apparatus for burning and carbonizing wools and woollen fabrics. July 14.

### Complete Specifications Accepted.

1897.

14,868. J. M. MacDonald and H. H. Boyle. Treatment of rhea grass and fibrous plants, and apparatus therefor. June 22.

1898.

10,592. H. Ferguson. Process and machinery for breaking and scutching fibrous plants and stems, such as brown rhea, ramie, hemp, jute, and the like. June 29.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### Applications.

13,495. D. Horsburgh. Improvements in and relating to vats for dyeing or impregnating fabrics. June 17.

13,503. J. B. Koenig. An improved dyeing soap or paste. June 17.

13,662. E. Rolffs. An improved process for the production of genuine dark blue dyes upon mixed chromium iron mordants. June 20.

13,725. W. F. Toifel. Improved process of and apparatus for producing designs upon paper-woven fabrics and the like by means of template printing. June 20.

14,421. W. E. Aykroyd, W. H. Aykroyd, and H. E. Aykroyd. Apparatus for the better and more even dyeing of yarn and similar fibrous substances, and the means of lubricating said apparatus. June 30.

14,429. T. Bentley. Improvements in hank scouring and dyeing apparatus. June 30.

14,758. C. H. Taylor.—Partly communicated by J. Santoux, France. Improvements in the process of means for producing striped effects on textile and other fabrics. July 5.

14,917. J. Nelson. Improvements in machinery or apparatus for dyeing, bleaching, and mercerising cloth. July 7.

14,960. L. J. P. Dumons. Improvements in dyeing textile materials, and apparatus for that purpose. July 7.

15,169. M. S. Sharp. A process for fixing the finish on cotton piece goods. Complete Specification. July 11.

15,370. A. J. King and F. H. Gornall. Improvements in dyeing and printing textile fabrics, warps, and paper. July 13.

15,556. J. Mendess. Improvements in compounds for cleansing and dyeing purposes. July 15.

15,575. F. I. Horrocks. Improvements in dyeing fabrics or yarns made of cotton or other vegetable materials, and also in producing figures on such fabrics when required. July 15.

### Complete Specifications Accepted.

1897.

15,587. H. Koechlin. *See* Class IV.

19,260. W. Hadfield, J. J. Sumner, and H. Hadfield. Method and means for bleaching textile fabrics. July 6.

1898.

8108. F. J. Oakes. Process of mordanting fibres or fabrics. June 29.

10,205. R. Brandts. Apparatus for dyeing and similarly treating textile rovings. June 29.

## VII.—ACIDS, ALKALIS, AND SALTS.

### Applications.

13,202. G. Bischof. Improvements in apparatus for reducing litharge, and other lead oxides to sub-oxide. June 13.

13,406. C. Hoy. Improvements in the manufacture and storage of carbonate of iron. June 16.

13,496. A. F. Watson. An apparatus whereby solid carbonic anhydride can be converted into the liquid form and thus stored. June 17.

13,521. C. F. Wülfing. Improvements in the manufacture of ferrous and ferric compounds. June 17.

13,645. W. R. Smith. An improved process and means for the production of carbide of calcium. June 18.

14,030. The Aluminium Company, Ltd., and A. G. Haddock. Improvements in bleaching compounds. June 24.

14,038. W. Schmeisser. A process for the manufacture of fluoro compounds. Complete Specification. June 24.

14,199. T. Cryer, trading as Deanhead Chemical Company. Improvements in the manufacture of ammonia and ammonium chloride and certain by-products. June 27.

14,269. C. E. Acker. Improvements in the manufacture of caustic alkalies from alloys of lead with the alkaline metals. Complete Specification. June 28.

14,453. J. E. T. Woods and W. A. Byrom. Improvements in and connected with the manufacture of calcium carbide. Complete Specification, June 30.

14,728. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning. Improvements in the manufacture of sulphur trioxide (sulphuric anhydride). July 4.

14,780. J. E. T. Woods and W. A. Byrom. Improvements in the manufacture of carbide of magnesium. Complete Specification. July 5.

14,795. O, Imray. From G. E. Junius, France. Improvements in obtaining baryta and pure iron by treatment of barium sulphate or carbonate with iron oxide. July 5.

14,804. W. Saward.—From E. L. C. Schiff, Holland. Improvements in and relating to the production of carbonic acid gas. July 5.

14,911. J. T. Hewitt and F. G. Pope. Improvements in obtaining alumina. July 7.

15,289. D. Williams. A process for the manufacture of concentrated hydrochloric acid. July 12.

15,502. J. Hargreaves. Improvements in the manufacture of alkalies and in means applicable for use therein. July 14.

15,557. C. C. Moore. Improvements in the treatment or purification of brine, and other saline solutions. July 15.

### Complete Specifications Accepted.

1897.

26,326. D. Lance and R. L. E. de Bourgade. Process for the economic production of cyanides, their derivatives, and other nitrogen compounds. July 6.

1898.

8570. W. P. Thompson.—From H. Harland and S. D. Crenshaw, United States. Roasting ovens or furnaces for the desulphurisation of volcanic sulphur, sulphurous ores, and ores containing a greater or less percentage of sulphur. July 20.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### Applications.

13,260. F. Schuman. Improvements in apparatus for the manufacture of wire-glass. Complete Specification. June 14.

13,310. F. O'Niell. A machine for the manufacture of glassware. Complete Specification. June 14.

13,386. C. D. Abel.—From La Société Anonyme des Faïenceries de Creil et Monteneau and E. G. Faugeron, France. Improvements in continuous kilns or ovens with travelling hearths for firing ceramic goods. June 15.

13,389. R. T. Hughes. Improvements in apparatus for the manufacture of perforated bricks and other articles. June 15.

13,706. J. M. Humphreys. Improvements in the manufacture of glassware. Complete Specification. June 20.

13,927. R. Hoyle. Improvements connected with the drying of china clay and other substances of like nature. June 23.

### Complete Specifications Accepted.

1897.

15,217. E. Gobbe. Continuous kiln for baking pottery or burning cements. June 23.

20,399. W. Boulton. Filter presses for treating potters' clay and other substances. July 20.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

13,346. F. Pulda. Improvements in fireproof floors or ceilings. Complete Specification. June 15.

13,954. W. L. H. Roberts. Improvements in or connected with the manufacture of Portland cement and apparatus employed therefor. June 23.

14,180. S. J. Payne, F. S. Taylor, and J. J. Edwards. Improvements in the manufacture of building bricks, blocks, and the like. June 27.

14,187. G. Leicht. Improvements in presses suitable for moulding artificial stones and for analogous purposes. June 27.

14,378. A. W. Perriman. Improvements in and in apparatus for the production of artificial marble and stone. June 29.

14,391. W. Antony. An improved process for and method of producing slabs for building and other purposes. Complete Specification. June 29.

15,339. T. E. Morris. A new or improved article of manufacture that can be used as a substitute for slate or other purposes. July 12.

15,656. C. Tschurl and C. Petraschek. Improvements in process and in apparatus for preserving wood. July 16.

### Complete Specifications Accepted.

#### 1897.

14,970. J. M. Danielli. Imitation of coloured marbles. June 29.

17,419. N. T. Ashton and J. Crompton. Manufacture of hydraulic cement. June 22.

21,146. W. J. Pearce. Vitreous decoration of walls, panels, and other surfaces. July 20.

#### 1898.

681. W. A. Oakley. Artificial stone and method of manufacturing same. June 22.

1209. H. Rous. Fireproof composition suitable for building purposes. June 29.

1513. C. Schoenfelder. Manufacture of artificial stone, and furnaces for burning the same. July 20.

8816. J. Feketeházy. Fireproof floors and ceilings for buildings. June 29.

9181. H. L. Cunnah and H. A. Hughes. Fireproof and other floors and ceilings. June 29.

10,046. G. Lilienthal. Construction of fireproof floors. July 6.

## X.—METALLURGY, MINING, Etc.

### Applications.

13,177. E. Banner. Improvements in the manufacture of iron. June 13.

13,332. W. J. Clapp. Improvements in furnaces and apparatus for and in the recovery and purifying of iron metals, slag, and cinder tap ores, and other kinds of waste products, and for other purposes. Complete Specification. June 15.

13,391. F. George. A method of and means for casting or moulding aluminium. June 15.

13,800. J. C. Mewburn.—From The Chemische Fabrik von Heyden, Gesellschaft mit beschränkter Haftung, Germany. Improvements in the manufacture of mercury. June 21.

13,871. G. Attwood. Improvements in the treatment of ores. June 22.

14,137. S. O. Cowper-Coles. Improvements in the treatment of ores for obtaining metals and other products therefrom. June 25.

14,235. W. Robertson and G. Low. An aluminium alloy. June 28.

14,523. H. R. Angel. Improved furnaces for treating sulphides containing zinc and other ores. July 1.

14,795. O. Imray.—From G. E. Junius, France. *See* Class VII.

15,065. H. C. Bull. Improvements in the extraction of gold from sea-water by chemical means and apparatus to be employed in connection therewith. July 8.

15,191. A. S. Bower. Improvements in means or apparatus for use in protecting iron and steel by producing magnetic oxide thereupon. July 11.

15,198. J. E. T. Woods and W. A. Byrom. Improvements in the manufacture of steel. July 11.

15,236. S. O. Cowper-Coles. Improvements in the extraction of tin from its ores. July 12.

15,397. T. Gwynne. Improvements in and relating to apparatus for preparing sheet iron and steel in the manufacture of tin and terne plates or for galvanising. July 13.

15,645. F. Ellershausen. Improvements in the treatment of refractory sulphide ores. July 16.

### Complete Specifications Accepted.

#### 1897.

15,674. C. Wetherwax. Process of treating gold ores. June 29.

22,256. The General Gold Extracting Company, Ltd.—From L. Peletan, France. Apparatus for the treatment of ores or the like containing gold or silver, or both gold and silver, for the obtainment of the precious metal therefrom. July 6.

#### 1898.

938. E. Martin. Manufacture of a compound metal of brass and silver. June 22.

8523. B. J. B. Mills.—From T. A. Edison, United States. Process and apparatus for concentrating magnetic iron ores and bricking the same for market. July 20.

9717. F. W. Minck. Smelting furnaces for metallic alloys. July 6.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

13,209. W. H. Hyatt. Bromo-electric process for the extraction of fine or refractory gold or other metals. June 13.

13,261. H. Jobben. Improvements in and relating to the outer cells or containing vessels of electric batteries. June 14.

13,393. J. R. Wylde, J. W. Kynaston, and The United Alkali Company, Ltd. Improvements in electrolytic apparatus especially intended for use in the electrolytic decomposition of solutions of alkaline chlorides. June 15.

13,680. S. W. Hart and E. J. Clark. Improvements in secondary electric batteries or accumulators. June 20.

13,861. W. E. Heys.—From E. Dumoulin, France. Improvements in and connected with the electro-deposition of copper and other metals on rotary cathodes. June 22.

13,960. H. Dämm. Electrode for accumulators. Complete Specification. June 23.

14,112. A. Peters. Improvements in or connected with primary batteries. Complete Specification. June 25.

14,132. W. L. Durban and J. Gore. Improvements in and relating to electrical furnaces more particularly applicable for the production of calcium carbide. June 25.

14,204. M. M. Bair. Improvements in electric batteries. June 27.

14,211. A. J. Ward. Improvements in or relating to excitant solutions for electric batteries or the like. June 27.

14,316. A. E. J. Ball. Improvements in and relating to dry and other galvanic batteries. June 29.

14,380. E. Giglio. Improvements in electric batteries. June 29.

14,455. T. A. B. Carver. Improvements in secondary batteries. June 30.

14,599. F. Greenfield. Improvements in anodes employed in connection with the electro-deposition of metals. July 2.

14,714. D. Martini. Improvements in vacuum dielectrics, electrodes, and apparatus for the production of ozone. July 4.

14,726. W. Rowbotham. Improvements in primary batteries. July 4.

14,809. W. H. Smith and W. Willis. Improvements relating to electric accumulators or storage batteries, and to the manufacture of plates or electrodes therefor. July 5.

14,839. E. Andreoli. Electro-deposition of metals by high tension currents. July 6.

14,868. P. Stiens. Improvements in primary batteries. July 6.

15,028. H. J. Haddan.—From C. Francke, Germany. Improvements in the manufacture of electrodes. Complete Specification. July 8.

15,155. E. Bailey and W. T. Hey. An improved method in the production of white-lead by the electric arc, and the introduction of compounds during volatilisation of the lead in a closed chamber. July 11.

15,222. H. H. Lake.—From J. Burns, France. Improvements in electric accumulators. Complete Specification. July 11.

15,304. J. B. Lee. Improvements in and relating to primary batteries for generating electricity. July 12.

15,346. J. J. Courtenay.—From C. A. Faure, France. Improvements in secondary batteries or electric accumulators. July 12.

15,430. W. P. Thompson.—From E. Langguth, Germany. Improved diaphragm for electrolytic apparatus. Complete Specification. July 13.

15,463. F. J. Gillibrand. Improvements in porous cells or porous division-plates for primary batteries, electrolytic cells, and the like. July 14.

15,573. I. Landauer. An improved process for the manufacture of metal paper by electro-metallurgy. July 15.

15,647. H. Bettermann. Improvements in electrically-ignited fuses for blasting purposes. Complete Specification. July 16.

### Complete Specifications Accepted.

1897.

7958. E. A. Jahncke. Accumulator plates or electrodes for secondary batteries. July 6.

18,029. C. Marschner. Process for the manufacture of electrode plates for storage batteries. June 22.

18,176. A. Heil. Electrical accumulators or storage batteries. July 6.

20,486. W. E. Heys.—From The Electrical Copper Company, Ltd., France. The electrode position of copper and other metals on rotary cathodes. June 22.

20,845. A. Müller. Accumulators or secondary batteries, and means for transporting the same. July 20.

25,889. F. C. Geary and J. H. Rowbottom. Cell or box suitable for accumulator or other electric batteries. July 20.

1898.

5186. R. Haddan.—From H. Dolter, France. Secondary batteries. June 29.

9778. M. Bernstein, proprietor of the firm of J. F. Wallmann and Co. Plates for electric accumulators. June 22.

10,094. A. C. Croftan. Making plates for electric battery purposes. June 22.

12,323. E. Edwards.—From P. A. Emanuel, United States. Electric batteries. July 20.

## XII.—FATS, OILS, and SOAP.

### Applications.

13,786. J. C. W. Stanley. Improvements in or relating to the extraction of oil from cocoa and other nuts. June 21.

13,881. L. von J. Jarocki and T. Kiczerowski. Process for purifying Caucasian petroleum. Complete Specification. June 22.

14,334. J. Lewkowitsch. Improvements in the manufacture and preparation of drying oils. June 29.

14,569. E. Andreoli. Improvements in deodorising and decolorising fats and oils. July 1.

14,570. E. Andreoli. Improvements in the treatment of fatty matters for deodorising and decolorising the same. July 1.

15,131. J. Smith and Sons, Limited, and W. Leach. Improvements in the treatment of the wash liquors from wool and other animal fibre to recover the grease and potash therefrom. July 9.

### Complete Specifications Accepted.

1897.

16,382. F. Becker and O. Wolff. Manufacture of soaps or cleansing compositions. July 20.

20,082. J. McKee. Method of and compound for treating lighting oils. July 6.

1898.

7561. P. Pessé. *See* Class XVI. June 22.

7651. W. S. Thomson. Soap, and process of manufacture therefor. June 22.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

13,342. H. A. Harvey. Improvements in preservative and anti-fouling compositions for ships bottoms and other submerged structures. June 15.

13,994. H. H. Bigland and J. Cunningham. An improved enamel paint. June 24.

14,647. R. Matthews. Improvements in the production of zinc oxide. July 2.

14,746. M. Zingler. Improvement in treating decayed or inferior or other india-rubber as described. July 5.

14,863. R. Seabrook. A new or improved anti-fouling composition for coating the bottoms of ships and other vessels. Complete Specification. July 6.

15,140. H. J. Haddan.—From G. Zonca and Co., Germany. An improved proceeding for producing a varnish and colour mordant. July 9.

15,155. E. Bailey and W. T. Hey. *See* Class XI.

15,364. C. W. Bonell. New or improved composition for bleaching and rendering harness leather and similar materials impervious to moisture. July 13.

15,524. G. Dobson. Improvements in or relating to preservative mixtures or compounds. July 15.

15,569. O. Zürcher. Improvements in processes for extracting caoutchouc. Complete Specification. July 15.

### Complete Specifications Accepted.

1897.

13,825. H. Steinem and K. S. Murray. Apparatus for the manufacture or production of carbonic acid gas. May 25.

19,727. R. J. Friswell and Brooke, Simpson, and Spiller, Limited. Obtaining guttapercha. July 6.

19,728. R. J. Friswell and Brooke, Simpson, and Spiller, Limited. Obtaining guttapercha. July 6.

24,178. C. E. Lee, C. F. Lawton, A. L. Lawton, and A. W. Lawton. Manufacture of paint. July 6.

1898.

11,264. G. F. Newman. A composition to be used for rendering garments and other articles waterproof. June 22.

12,184. G. Wegelin. Method of and means for the manufacture of lamp black and other carbon of great density. July 6.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

14,170. J. Eaton. Improvements in the method of treating light leather or split skins, and in apparatus for producing the said improvements. June 27.

14,220. G. D. Burton. Process of tanning hides or skins of animals, &c. Complete Specification. June 27.

14,221. G. D. Burton. Unhairing all kinds of animal skins. Complete Specification. June 27.

14,398. F. de Rechter and G. de Rechter. Improvements relating to the treatment of skins for use in furriery and tanning operations. Complete Specification. June 29.

14,724. G. Feith. Process of rapid tanning. July 4.

15,137. J. Noad. An improved method and means to be employed in the tanning of hides, kips, and skins. July 9.

### Complete Specifications Accepted.

1897.

15,352. A. Mario. Improvements in tanning. June 22.

16,801. C. Michel. Process and apparatus for the rapid tanning of skins and hides. June 22.

20,134. H. Sefton-Jones, of the firm of W. P. Thompson and Co.—From E. Bardon, France. Tanning apparatus. July 20.

20,273. J. Hall. Machinery for treating skins, hides, leather, and other like substances. June 29.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

13,690. F. Silberstein and L. Lilienfeld. Improvements in and connected with the manufacture of soluble products from proteids. June 20.

13,718. C. D. Abel.—From The Actien Gesellschaft für Anilin Frbrikation, Germany. Manufacture of soluble albuminous preparations. June 20.

### Complete Specifications Accepted.

1898.

7561. P. Pessé. Decoloration and purification of liquids. June 22.

11,753. A. J. Boult.—From C. M. Higgins, United States. Adhesive compounds. July 6.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

13,412. E. A. Hamlyn. Improvements in the method of and apparatus for mixing yeast with wort and other liquids. June 16.

13,862. W. H. Akester. Improvements in the production of alcohol. June 22.

13,880. G. G. Cave and H. S. Stoneham. Improvements in the process for producing extracts of malt, hops, and other substances. June 22.

14,305. F. Faulkner and J. E. J. Johnson. Improvements in or relating to brewing and the production of alcoholic beverages. June 28.

14,152. J. E. Carroll. Process and apparatus for maturing or treating wines and spirits. June 30.

14,731. O. Imray.—From G. H. Benjamin, United States. Improvements in the manufacture of ethyl alcohol. July 4.

15,133. J. Hendrick. Manufacture of manure from waste liquors from whiskey and like stills. July 9.

15,151. M. Otto. An improved process of and apparatus for the sterilisation of liquids by means of ozone. Complete Specification. July 9.

15,355. R. Ilges. A process and apparatus for continuously separating fusel oil and first short from raw spirit vapours in one operation. July 12.

15,384. J. Carson. Improved method of aërating or carbonating water or other liquids. July 13.

15,452. J. T. Hewitt. Improvements in methods of removing noxious ingredients from spirituous and other alcoholic liquors. July 14.

15,561. A. Collette, jun., and A. Boidin. Improvements in apparatus for the manufacture of alcohol by saccharification and fermentation by means of mucedineæ. Complete Specification. July 15.

### Complete Specifications Accepted.

1897.

16,609. W. B. Turner. Treatment of malt or grain for brewing or other purposes. June 22.

18,645. F. S. Masters and J. C. Shears. Manufacture of beer and other liquids, and apparatus therefor. June 29.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

### *A.—Foods.*

14,818. A. Sweetser. Improvements in preserving milk and other foods and apparatus therefor. July 5.

15,194. H. Ruda. A new or improved means for preserving fresh or sea water fish. July 11.

15,196. B. J. B. Mills.—From A. Dalmote, Belgium. An improved diastasic preparation to be employed in the manufacture of bread biscuits and the like. July 11.

15,535. J. Wohlgemuth. An improved process for the production of artificial butter and the like. Complete Specification. July 15.

15,654. A. Zimmermann.—From The Chemische Fabrik auf Actien vorm. E. Schering, Germany. Improvements in the preservation of meat and in apparatus therefor. July 16.

### *B.—Sanitation.*

13,438. J. A. Wanklyn and W. J. Cooper. Improvements in the treatment and purification of sewage. June 16.

13,697. R. O. Wynne-Roberts. Improvements in connection with filter beds for filtering sewage water or other liquids. June 20.

13,711. T. Voile. New or improved means for destroying soot in chimneys. June 20.

14,162. C. J. Whittaker and W. C. Bryant. Improvements in the treatment or purification of sewage. June 27.

14,997. G. D. Wild. Improvements in the treatment for the purification of sewage of all kinds, domestic or otherwise, including effluents from dye works, &c. July 8.

### *C.—Disinfectants.*

13,459. R. Willcox.—From Vereinigte Chininfabriken Zimmer and Co., Gesellschaft mit beschrankter Haftung, Germany. *See* Class XX.

### Complete Specifications Accepted.

### *A.—Foods.*

1897.

15,145. T. Hill Jones. Manufacture of a new alimentary extract. June 22.

17,915. S. Pitt — From The Chemische Fabrik auf Actien vormals E. Schering, Germany. The production of food preparations containing casein. July 29.

18,714. E. C. L. Kressel and T. Hill-Jones. The manufacture of an improved alimentary extract. July 6.

19,979. C. Estcourt. Production of a valuable food for animals. July 20.

### *B.—Sanitation.*

1897.

18,555. W. D. Scott-Moncrieff. Purification of sewage, and apparatus therefor. July 6.

1898.

6061. H. H. Leigh.—From La Société Anonyme pour les Applications Industrielles de l'Ozone, France. Apparatus for the sterilisation or the disinfection of waters of all kinds, treatment of sweetened syrups, and other analogous applications, by means of ozone, air, or other gases. July 6.

7094. J. Metzer. Process and machine for purifying and sterilising filtering media. June 22.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

13,689. T. H. Cobley. Improvements in the production of silicated and asbestosised pulp for the manufacture of non-inflammable paper and board for wood substitutes and as a non-conductor for coating surfaces. June 20.

14,696. P. W. H. Gray. A new or improved composition for manufacturing wall paper in relief, artificial flowers, and other articles. July 4.

15,202. O. Imray.—From W. Krische and A. Spitteler, Germany. Improvements in the manufacture of horn-like products from caseïn. July 11.

### Complete Specifications Accepted.

1897.

30,579. S. Jaques. Manufacture of paper. July 20.

1898.

12,096. A. Aberg. Apparatus for treating cellulose or paper pulp and the like. July 20.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

13,317. H. E. Burgess. A process for the manufacture of musk substitutes. June 14.

13,459. B. Willcox.—From Vereinigte Chininfabriken Zimmer and Co., Gesellschaft mit beschränkter Haftung, Germany. The manufacture and production of preparations of salicylic acid suitable for surgical antiseptic purposes. June 16.

13,688. J. E. T. Woods. Improvements in and connected with the manufacture of artificial camphor from hydrocarbon oils of the terpene series. June 20.

13,710. G. Lorenz. Process for the production of a permanent preparation containing the white corpuscles of the blood serum of swine immunised against swine fever. Complete Specification. June 20.

13,714. L. Sell. The manufacture of a new preparation from saligenin. June 20.

13,965. C. A. Steche. Improvements in and relating to the treatment or purification of sandalwood oil and to products obtained therefrom. Complete Specification. June 23.

14,404. J. H. Taylor. Ptyrazine. June 29.

14,895. W. H. Akester. Improvements in the production of ethylene. July 6.

15,138. W. H. Akester. Improvements in the production of ethylene. July 9.

15,210. J. C. Mewburn. — From L. D. Huguenin et Cie., and P. Barbier, Switzerland. The manufacture of new perfumes. Complete Specification. July 11.

### Complete Specifications Accepted.

1897.

10,535. J. W. Mackenzie.—From C. Schmid. Method for producing an artificial scent or musk-like substance. July 6.

18,698. T. Bird. Process for the treatment of artificial camphor, either in the crude state or after re-sublimation. July 6.

23,719. E. de Laire. Manufacture of a new product from ionone or pseudo-ionone. June 22.

## XXI.—PHOTOGRAPHY.

### Applications.

13,195. J. Hauff. Improved photographic developer. June 13.

13,315. C. M. Hepworth and C. Urban. Improvements in or relating to the printing and development of photographic film. June 14.

### Complete Specifications Accepted.

1897.

12,017. J. Wheeler. Production of films and apparatus therefor. June 22.

17,122. G. J. Sershall and J. H. Kirk. Production of coloured photographs. June 29.

19,168. C. T. Jago. Method of obtaining permanent coloured photographs. July 6.

27,033. J. C. Fell.—From M. W. McDonald, United States. Matt surface photographic paper, and process of making same. June 22.

29,093. K. Märten. Method of producing pictures upon glass by lithography in one or more colours. June 22.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

13,466. R. T. Maguire and D. P. Maguire. Improvements in and relating to lucifer matches. June 16.

13,609. J. Grossmann. New or improved explosive mixture. June 18.

13,883. G. Trench. Improvements in the manufacture of explosives. June 22.

14,175. W. G. Cordes. Improvements in the manufacture of matches. June 27.

14,870. G. Beneké. Improvements in the manufacture of blasting powders. July 6.

15,553. F. W. Jones. Improvements in or connected with the treatment of explosives. July 15.

### Complete Specifications Accepted.

1897.

16,777. J. Macnab. Blasting, and blasting cartridges. June 22.

18,929. C. D. Abel.—From The Gesellschaft für Lindes Eismaschinen, Germany. New explosive compound. July 20.

29,204. J. Bown. Fog signals. June 22.

1898.

1631. L. Gathmann and E. Gathmann. Safety fuses for high explosive shells. June 22.

## PATENTS UNCLASSIFIABLE.

### Applications.

13,283. J. Wheeler. Manufacture or production of certain substances which I term pyrobetulin and pyrobetulin anhydride. June 14.

13,289. A. Krefting. Improvements in the manufacture of organic products from seaweed. Complete Specification. June 14.

14,648. L. C. Reese. Improvements in the method and means of extracting chemical products from raw materials. July 2.

15,115. J. Macneil. An improved composition and process for its manufacture. Complete Specification. July 9.

### Complete Specifications Accepted.

1898.

12,277. A. Krefting. Manufacture of valuable organic products from seaweed. July 6.

13,151. A. Krefting. Manufacture of organic products from seaweed. July 20.

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THE JOURNAL

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H. R. Procter.
F. W. Richardson.
G. W. Slatter.
A. Smithells.

*Hon. Local Secretary and Treasurer:*
H. R. Procter, The Yorkshire College, Leeds.

## NOTICES.

It is with the deepest regret that we announce the death, on Sunday the 21st inst., of Mr. Edward Rider Cook, Hon. Treasurer of the Society since its foundation in 1881. Pending the preparation of a more detailed notice of his career, we take this opportunity of informing the members of the great loss which the Society has thus sustained, and of asking them to extend their sympathy to his family in their bereavement.

The Society's Office is closed from September 2nd to September 17th next, and the General Secretary will feel obliged if members who have changes of address to communicate will send them in after that date.

In consequence of the lamented death of the Hon. Treasurer, Mr. E. Rider Cook, all remittances should be made payable to the General Secretary until further notice.

### Annual General Meeting.

The group photograph, taken at University College, Nottingham, is a very successful picture. Members who desire a copy should send the sum of 4*s.* 6*d.* to Arthur Neale, Studio, St. Peter's Church Walk, Nottingham. Terms :—Cash with order.

### Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, the bulk of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer and Committee who have the work of correction in hand.

The prices are as follows :—

| | |
|---|---|
| To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List. | Each copy 10*s.* |
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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

### Telegrams.

The code address of Palace Chambers is "Palatable, London," and the Society's number is 59. Hence telegrams need only be addressed "59, Palatable, London," without words "Society of Chemical Industry."

## CHANGES OF ADDRESS.

Akitt, Thos.; Journals to Raeberry, Upper Skelmorlie, Ayrshire.

American Chemical Society; Journals to Prof. Edw. Hart, Easton, Pa., U.S.A.

Andreasch, F., l/o Vienna; Penketh Tanning Co., Ltd., Penketh, near Warrington.

Brown, R. B., l/o Leeds; 3, Alexandra Terrace, Headingley, Leeds.

Devey, A. C.; Journals to 43, Redcliffe Gardens, London, S.W.

Fulton, H. B., l/o West Kensington; 353, Mansion House Chambers, London, E.C.

Heap, Isaac H., l/o Basford; Turnhurst Hall, Chell, near Stoke-on-Trent.

Hodgkinson, Dr. W. R., l/o Park Villas; 18, Glenluce Road, Blackheath, S.E.

Kirkland, R., l/o Limerick; c/o The Condensed Milk Co. of Ireland, Tipperary, Analyst.

Lewis, A. E., l/o Dublin; 7, Prescot Street, New Brighton, Cheshire.

Myers, Wm. S.; Journals to Rutgers College, 15, Union Street, New Brunswick, N.J., U.S.A., Assoc. Professor of Chemistry.

Newcomen, Thos.; Journals to Woodside, Slad, Stroud, Gloucestershire.

Salis-Mayenfeld, Dr. E. von, l/o Blackley; Woodlea, Albert Road, Levenshulme, Manchester.

Smith, Ernest A., l/o London; The Assay Office, Leopold Street, Sheffield.

Smith, Jas. F., l/o Sparkhill; 80, Anderton Road, Sparkbrook, Birmingham.

Stuart, Harry T. R., l/o Glasgow; Know Mill House, Entwistle, near Bolton.

Townsend, O. C., l/o Glasgow; Kirkland Works, Leven, Fife.

Trobridge, A., l/o Oldbury; 153, Whalley Road, Accrington, Lancashire.

Walker, H. V., l/o New York; 38, Clinton Street, Brooklyn, N.Y., U.S.A.

Weston, R. S.; Journals to 21, Wyman Street, Brockton, Mass., U.S.A.

Wilson, J. Arthur, l/o Rose Cottage; Forest House, Newchurch, near Manchester.

Wilson, W. W., l/o Strathblane; c/o Inglis and Wakefield, Ltd., Busby, near Glasgow.

## Deaths.

Edw. Brooke, Fieldhouse, Huddersfield.

Edw. Rider Cook, Woodford, Essex. August 21st.

## New York Section.

*Meeting held at the College of Pharmacy, on Friday, May 20th, 1898.*

PROF. CHAS. F. CHANDLER IN THE CHAIR.

### MODERN SYNTHETIC MEDICINAL PRODUCTS.

BY V. COBLENTZ, PH.D., F.C.S.

The discovery of kairin by O. Fischer in 1882 opened up a new field in modern therapeutics: it demonstrated the possibility of building up compounds of similar structure and medicinal action to those found in nature. Additional impetus was given this subject through the accidental discovery of the antipyretic properties of acetanilide. With these two prototypes before them, modern chemists and pharmacologists began a thorough and systematic research, and after having prepared a very large number of synthetic products, they established the fact that the presence of certain groupings influences physiological action. As a result, most of nature's medicinal products have been not only successfully imitated, but decidedly improved upon; in fact, the most exacting requirements of modern medicine are being met in the construction of synthetic medicinal products. Thus, the local anæsthetic alkaloid, cocaine, possesses irritating and toxic properties; these undesirable features have been practically eliminated in the synthetic local anæsthetics, eucaine "B," orthoform, and holocaine; nature's mydriatic alkaloid, atropine, which likewise possesses objectionable features, has been improved on in homatropine, euphthalmine, and mydrol; lithium, the old remedy for gouty affections, has found its superiors in piperazine, lycetol, lysidine, urotropine. The first successful synthetic antipyretic, acetanilide, possessed undesirable secondary effects; these were overcome by the introduction of other groupings, resulting in the production of a number of substances, among the more successful of which is the ethoxy-derivative, phenacetine; this, however, possessed the disadvantage of insolubility, which has later been overcome in such derivatives as phenocoll, citrophen, apolysin, phesin, &c. The natural compound of iron and protein substance, such as occurs in the liver and other organs, has been successfully imitated in the artificially-prepared substance known as ferratin. Nature's hypnotic, opium, has found an almost unlimited number of superior substitutes.

Several classifications of this extensive list of synthetic products have been made; they are based either upon their chemical constitution and are arranged in classes accordingly or an arrangement is adopted whereby they are classified into groups according to their physiological action. The chemical classification is unsatisfactory to the physiologist, since it throws remedies of dissimilar medicinal action into a confused mass under one head. A classification according to the second method was made by Dujardin-Beaumetz, who classed the aromatic hydroxyl derivatives (as phenol, naphthol, resorcinol) as antiseptics, amido derivatives (as acetanilide, kairine, thalline) as antipyretics, and such compounds in which the hydrogen of the amido group is replaced by a fatty radicle as hypnotics; for example, antipyrine, exalgin (methyl acetanilide), phenacetin (ethoxyacetanilide). As was pointed out by Ville, such a classification is incorrect from a chemical standpoint, for the bodies classed under the amido group do not belong together, since acetanilide contains nitrogen in the side chain, while in thalline and kairine it is in the nucleus as a derivative of quinoline; likewise exalgin and phenacetin are derivatives of aniline, while antipyrine is a derivative of pyrazole. Hodgkin classified the aliphatic derivatives and aldehydes as hypnotics and anæsthetics, whilst those of the aromatic series were classed of antipyretics and antiseptics. Although this may be satisfactory in a very general way, it is very defective and erroneous when applied in detail.

The method of classification adopted here consists in arranging the bodies into general groups according to medicinal action; as, for example, antipyretics, antiseptics, hypnotics, &c. Under these groups the compounds are arranged into their various chemical classes. The object of this has been to give prominence to the presence of certain groupings which occur in each of these classes, and to the influence exerted by the introduction of new groupings; in other words, to show, wherever possible, the relationship between chemical constitution and physiological action.

## ANTIPYRETICS.

### *Group I.—Acetylated Amines and Derivatives.*

As prototype of this class we have the well known—

*Antifebrine*, $C_6H_5NH.CH_3CO$: Acetanilide or Phenylacetamide.

Closely related to antifebrine are—

*Antisepsine*, $C_6H_4(Br).NH(CH_3CO)$, Para-bromo-acetanilide.

*Iodo-Antifebrine*, $C_6H_4(I).NH.CH_3CO$, Para-iodoacetanilide.

*Acet-p-Toluide*, $C_6H_4(CH_3).NH.CH_3CO$: The *ortho* compound is also employed.

*Methacetine*, $C_6H_4(OCH_3).NH.CH_3CO$: Para-oxymethyl-acetanilide.

*Exalgine*, $C_6H_5N.(CH_3).CH_3CO$: Methylphenylacetamide.

*Cosapyrine*, $C_6H_4(SO_3Na).NH.CH_3CO$: Acetyl-sodium-sulphanilide.

*Diacetanilide*, $C_6H_5N(CH_3CO)_2$.

*Benzanilide*, $C_6H_5NH.C_6H_5CO$.

*Bromamide*, $C_6H_2Br_3NH_2.HBr$: Tribromaniline hydrobromide.

Erdmann and von Nencki have shown that the introduction of the carboxylic or sulphonic acid group into bodies of well-defined toxic properties, results in a marked diminution of this action; thus benzene and its derivatives, aniline, naphthalene, phenol, pyridine, and quinoline, are far more toxic to the organism than their carboxylic derivatives; as benzoic acid, naphthalene, pyridine, and quinoline carboxylic acids; and the same is true of the corresponding sulphonic acids. The first-named poisonous class of aromatic bodies on entering the system readily submit to oxidation and reduction, giving rise to toxic action, but the stability of the carboxyl (COOH) group and its resistance to processes of further oxidation serve to protect the derivatives from further chemical change.

### *Group II.—Para-Phenetidine and Derivatives.*

In the human system para-amido-phenol—

$$C_6H_4\overset{(1)}{(OH)}\overset{(4)}{NH_2}$$

and para-acet-amido-phenol—

$$C_6H_4\overset{(1)}{(OH)}\overset{(4)}{NHCH_3CO}$$

cause a diminution of temperature of from 2° to 3° C. (dose 0·5 grm.), frequently accompanied by chilliness, vomiting, and tinnitus aurium. If the hydrogen of the hydroxyl group in the former be replaced by an alkyl group, the antipyretic properties remain unchanged, whilst the toxic action is thereby lessened.

The maximum antipyretic effect is shown by those bodies containing the methyl group, and the minimum by those containing the ethyl group.

If the hydrogen of the amido group in para-acet-amido-phenetol be replaced by an alkyl, a substance of narcotic properties is obtained; as, for example, methylphenacetin—

$$C_6H_4\overset{(1)}{(OC_2H_5)}\overset{(4)}{N(CH_3)}COCH_3$$

If an ethyl group enters, a lessening of narcotic effect results.

According to von Nencki and others, this class of compounds decomposes in the organism, with the production of *p*-amido-phenol or *p*-acet-amido-phenol, upon the presence of which the analgesic properties of phenacetin and its derivatives depend; if the hydrogen of the amido group as above cited is replaced, a narcotic effect is manifested through the agency of the liberated alkyl groups.

The best known of these derivatives of phenetidine—

$$C_6H_4\overset{(1)}{(OC_2H_5)}\overset{(4)}{NH_2}$$

is the acetyl compound or *Phenacetin*—

$$C_6H_4\overset{(1)}{(OC_2H_5)}\overset{(4)}{NHCH_3CO}$$

aceto-para-phenetidine which is soluble 1 in 1,100 of cold water.

The success of phenacetin led to the introduction of a number of similar phenetidine derivatives, in which the acetyl radicle was replaced by other acidyl groups, the object being to eliminate all toxic effects, and increase rapidity of action and solubility.

Owing to the insolubility of phenacetin, one of the earliest endeavours was to obtain a compound of the same properties, but sufficiently soluble to enable it to be employed in solution. This was accomplished by the addition of a basic group, glycocoll ($CH_2NH_2.COOH$), which, through its amido group, is capable of uniting with acids, forming very soluble salts.

This compound was introduced under the title of—

*Phenocoll*, $C_6H_4(OC_2H_5).NH.CH_2NH_2CO$, amido-acet-*p*-phenetidine. The soluble salts are the hydrochloride, acetate, and salicylate (*Salocoll*).

*Lactophenin*, $C_6H_4(OC_2H_5).NH.CO-CH(OH)CH_3$: Lacto-*p*-phenetidine. Here the lactyl group replaces the acetyl of phenacetin. The formyl group (HCO), when introduced, produces an antiseptic. Lactophenin is soluble to the extent of 1 in 330 parts of water.

*Sedatin*, $C_6H_4(OC_2H_5)NH.C_4H_9CO$, valeryl *p*-phenetidine.

*Saliphen*—

$$\mathrm{C_6H_4(OC_2H_5)NH.C_6H_4(OH)CO}$$

Salicyl-*p*-phenetidine, insoluble in water.

*Triphenin*, $\mathrm{C_6H_4(OC_2H_5)NH(CH_3CH_2CO)}$, propionyl-*p*-phenetidine is soluble 1 in 2,000 parts of water.

*Apolysin*—

$$\mathrm{C_6H_4(OC_2H_5).NH.CO.CH_2.C.(OH)(CH)_2(COOH)_2},$$

mono-phenetidine-citrate is soluble 1 in 55 parts of water.

*Citrophen*, $\mathrm{[C_6H_4(OC_2H_5)NH.CO]_3C_3H_4OH}$: Citro-*p*-phenetidine. Here there are three phenetidine groups to one molecule of citric acid. Soluble 1 in 40 parts of water.

*Kyrofin*, $\mathrm{C_6H_4(OC_2H_5)NH.CH_2.OCH_3.CO}$, methyl-glycolic-*p*-phenetidine is soluble 1 in 600 parts of water.

*Malarin*, aceto-phenone-phenetidine-citrate. Insoluble.

*Phesin*, $\mathrm{C_6H_3(OC_2H_6)SO_3Na.NHCH_3CO}$, acet-*p*-phenetidine-sodium sulphonate. Very soluble.

*Oxyphenacetin.*

*Amygdophenin*, $\mathrm{C_6H_4(OC_2H_5)NH.CO.CH(OH)C_6H_5}$, phenyl-glycolyl-*p*-phenetidine. Insoluble in water.

*Vanillin-Para-Phenetidine*—

$$\mathrm{C_6H_3(OH)(OCH_3)CH.N.C_6H_4.OC_2H_5}.$$

*Para-Phenetidine-Furfural*—

$$\mathrm{C_6H_4(OC_2H_5)N.C_4H_3O.CH}.$$

### *Group III.—Diazo-Derivatives.*

Phenyl-hydrazine, $\mathrm{C_6H_5NH.NH_2}$, the mother-substance of this group of antipyretics, may be considered as a derivative of aniline, in which an atom of hydrogen is replaced by an amido group. Although it apparently does not possess any therapeutic value, its acetyl derivative (hydracetin) is an antipyretic, but owing to its toxic characters it has not been favourably received. This experience served its purpose in the production of antithermin, agathin, and orthin.

*Hydracetin or Pyrodin*, $\mathrm{C_6H_5NH.NH.CH_3CO}$: acetyl-phenyl-hydrazine.

*Antithermine*, $\mathrm{C_6H_5NH.N{:}C(CH_3)CH_2.CH_2.(CO)OH}$: phenylhydrazine-levulinate.

*Agathin*—

$$\mathrm{(C_6H_5)(CH_3)N.N{:}CH.C_6H_4.OH}$$

salicyl-α-methyl-phenyl-hydrazone.

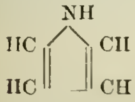
*Orthin*, $\mathrm{C_6H_3}\overset{(1)}{\mathrm{OH}}\mathrm{.NH.}\overset{(2)}{\mathrm{NH_2}}\mathrm{.}\overset{(4)}{\mathrm{COOH}}$, hydrazine-*p*-hydoxybenzoic acid.

*Ethylene-Phenylhydrazine-Succinic Acid*—

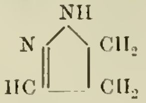
$$\mathrm{(C_6H_5.N.N)_2{:}H(CO.C_2H_4COOH)_2}$$

### *Group IV.—Pyrazolone Derivatives.*

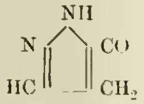
Antipyrine, the prototype of this group, discovered by Knorr in 1884, was obtained by condensation of phenylhydrazine (an antipyretic nucleus) and aceto-acetic ester; the resulting body, phenyl-methyl-pyrazolone, was unsuited, however, for therapeutic employment. Utilising Filehne's previous experience with the substances of like physiological action derived from quinoline, kairine, and kairoline, a methyl group was introduced which resulted in the formation of the well-known antipyretic. The relationship of antipyrine may be traced from the five-membered cyclic base pyrrole—



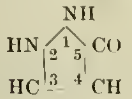
*Pyrrole.*



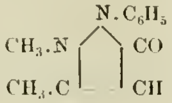
*Pyrazoline.*



*Pyrazolone.*



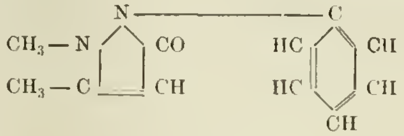
*Iso Pyrazolone 5.*



*Antipyrine.*

According as one or other of the two methylene ($\mathrm{CH_2}$) groups in pyrazoline is replaced by a carbonyl group, two ketones are obtained; these are distinguished as pyrazolone 4 and 5. Antipyrine is a derivative of the latter, being then a phenyl 1, dimethyl 2.3, isopyrazolone 5.

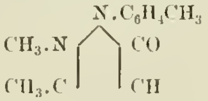
The antipyrine molecule consists of two ring nuclei, namely, the "pyrazolone" ring of Knorr and a benzene ring, thus:—



The question as to whether the physiological action is due to the combination, or to one of the rings, or to certain groupings in these rings, has been studied by Filehne, who demonstrated that the pyrazolone nucleus alone was not capable of producing the effects of antipyrine; also, the benzene ring possesses no medicinal value. However, with the introduction of other groupings the configuration is changed, in this case, by the presence of two methyl radicles.

Riedel introduced a methyl group into the benzene nucleus of antipyrin, and produced the valuable—

*Tolypyrine*—Tolyl-dimethyl-pyrazolone—

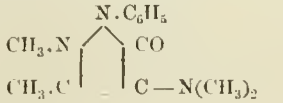


This is stated to be more active than antipyrine.

Thoms suggested the introduction of an oxy-alkyl group in the benzene nucleus, but the antipyretic produced had toxic properties.

More successful has been the introduction of the physiologically active di-methyl-amido group in the pyrazolone nucleus, yielding the valuable antipyretic:—

*Pyramidone*—Di-methyl-amido-antipyrine or dimethyl-amido-phenyl-dimethyl-pyrazolone—



It is claimed that pyramidone is about three times as active as antipyrine.

A large number of combinations have been made by fusing or heating together aqueous solutions of aromatic acids or phenols with antipyrine and tolypyrine, the resulting compounds possessing the properties of both constituents.

*Antipyrine Salicylate* or "Salipyrine"—

$$\mathrm{C_{11}H_{12}N_2O.C_6H_4(OH)COOH}.$$

*Tolypyrine Salicylate* or "Tolysal"—

$$\mathrm{C_{11}H_{11}(CH_3)N_2O.C_6H_4(OH)COOH}.$$

*Antipyrine Benzoate.*

*Antipyrene Naphthol or Naphthopyrine*—

$$\mathrm{C_{11}H_{12}N_2O.C_{10}H_7OH}.$$

*Resorcinol or Resopyrine*, $\mathrm{C_{11}H_{12}N_2O.C_6H_4(OH)_2}$.

*Pyrogallol Antipyrine.*

*Antipyrine Mandelate* or "Tussol"—

$$\mathrm{C_{11}H_{12}N_2O.C_6H_5CH(OH)COOH}.$$

*Iodopyrine*, $\mathrm{C_{11}H_{11}IN_2O}$

*Ferripyrine*, $\mathrm{(C_{11}H_{12}N_2O)_3Fe_2Cl_6}$.

Combinations of antipyrine with hypnotics possess hypnotic and analgesic action.

*Hypnal*—Chloral antipyrine, $\mathrm{C_{11}H_{12}N_2O.CCl_3.CH(OH)_2}$.

*Dichloral Antipyrine*, $\mathrm{C_{11}H_{12}N_2O(CCl_3CH(OH)_2)_2}$.

*Butyl Hypnal*, $\mathrm{C_{11}H_{12}N_2O.C_4H_5Cl_3O.H_2O}$.

### *Group V.—Quinoline Derivatives.*

This nucleus ($C_9H_7N$) furnishes us antiseptics as well as antipyretics. Among the simpler derivatives we have the salicylate, tartrate, citrate, and acetamido quinoline, which serve the purposes of both antiseptic and antipyretic.

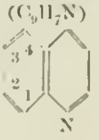
Through attempts at the synthesis of an artificial quinine (a derivative of quinoline) a number of valuable substitution products were obtained. Among the first was aceto-*o*-amido-quinoline, $C_9NH_6(CH_3CO)$, which represented the "antifebrin" analogue of quinoline. The ortho-phenol-sulphonic acid derivative "diaptherine" proved to be an antiseptic. Later, Filehne discovered that Fischer's methyl (Kairin M) and ethyl (Kairin A) derivatives of ortho-oxy-quinoline-tetra-hydride, possessed antipyretic properties. Thus the first steps were made towards the production of artificial alkaloidal antipyretics; owing, however, to the unpleasant secondary effects, the kairines were soon abandoned. This led, however, to a careful study of quinoline, resulting in a number of new discoveries, among which was Skraup's "thalline," a tetra-hydro-*p*-quinanisol, the sulphate and tartrate of which possess antipyretic properties. Among some of the more important of quinoline antipyretics are—

*Aceto-Ortho-Amido-Quinoline*, $C_9H_6N(NH.CH_3CO)$: This body is obtained by introduction of an acetylated amido group into the ortho (1) position of quinoline.

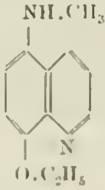
*Analgene or Benzanalgen*, is ethoxy (1) acetylamido (4), quinoline. This compound is analogous to benzoyl-*p*-phenetidine.

*Thermifugin*, $C_9H_8N(CH_3)(OH)CO.ONa$: The sodium salt of methoxy-hydro-quinoline-carboxylic acid.

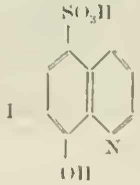
To this same class of oxy-quinolines belongs that excellent antiseptic, loretine (*m*-iodo-*o*-oxyquinoline-ana-sulphonic acid (4)), the structural formula of which is shown along with the other members of this class.



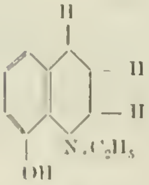
Quinoline.



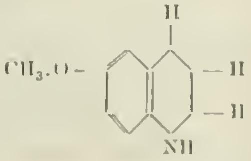
Analgene.



Loretine.



Kairine.



Thalline.

### *Group VI.—Urethanes.*

As type of this group we have the esters of carbamic acid—

$$CO(OH)NH_2$$

or urethane. These bodies, like their prototype, ethyl urethane—

$$CO(OC_2H_5)NH_2$$

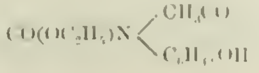
are classed under the hypnotics; if, however, we replace a hydrogen atom of the amido group by benzene residues, antipyretics result.

*Euphorine*—

$$CO(OC_2H_5)NHC_6H_5$$

Phenyl urethane. This body possesses slight sedative and antiseptic properties. If an oxy-phenyl group is introduced, we obtain a para-oxy-phenyl-urethane, the acetyl derivative of which constitutes the entering group in—

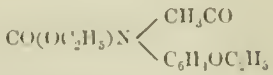
*Neurodine*—



$$CO(OC_2H_5)N\begin{cases}CH_3CO\\C_6H_4.OH\end{cases}$$

Acetyl-*p*-oxy-phenyl urethane. The ethyl ether of neurodin gives us—

*Thermodine*—



$$CO(OC_2H_5)N\begin{cases}CH_3CO\\C_6H_4OC_2H_5\end{cases}$$

Acetyl-*p*-ethoxy-phenyl-urethane.

### *Group VII.—Salicylic Acid Derivatives.*

*Di-Iodo-Salicylic Acid*—

$$C_6H_2I_2(OH)COOH$$

*Benzacetine*—

$$C_6H_3(OC_2H_5)(NHCH_3CO)COOH$$

Acetamido-methyl-salicylic acid.

*Salophen*—

$$C_6H_4(OH)COO.C_6H_4.NHCH_3CO$$

Acetamido-salol or Acetyl-*p*-amido-phenol-salicylic-ester.

*Salicylo-Sulphonate of Sodium*—

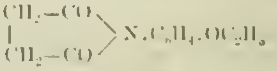
$$C_6H_3(OH)(COOH)SO_3Na$$

*Sodium Anisate*—

$$C_6H_4(OCH_3)COONa$$

also anti-rheumatic.

*Pyratin*,—*p*-ethoxyphenyl-succinamide—



$$\left.\begin{matrix}CH_2-CO\\|\\CH_2-CO\end{matrix}\right\rangle N.C_6H_4.OC_2H_5$$

## ANODYNES AND HYPNOTICS.

### *Alcohols.*

The primary alcohols are less narcotic than the secondary, and these less so than the tertiary. In the tertiary alcohols, the physiological action depends upon the nature of the alcohol radicle, which is united to the tertiary carbon atom; if the radicle $CH_3$ be present as in tri-methyl-carbinol (tertiary-butyl-alcohol $(CH_3)_3 : C.OH$), the action is relatively feeble; with introduction of ethyl groups ($C_2H_5$) it increases in proportion to the number of such groups united to the tertiary carbon atom. We have such an example in—

*Amylene Hydrate or Tertiary Amyl Alcohol*—

$$OH.C(CH_3)_2C_2H_5.$$

### *Ethers.*

We have another instance illustrating the above-mentioned influence exerted by the replacement of methyl by ethyl groups in the two following ethers or acetals, the second being far more effectual than the first—

*Methylal or Methylene Dimethyl Ether*—

$$CH_2(OCH_3)_2.$$

*Acetal or Ethylidene Diethyl Ether* =

$$CH_3.CH(OC_2H_5)_2.$$

### *Aldehydes.*

Among the polymeric forms or chlorine substitution products of acet-aldehyde we find a number of hypnotics; the simple aldehydes are of an irritating nature and antiseptic in their action. This character disappears upon the introduction of chlorine, reaching a maximum of hypnotic effect in the tri-chlor-substitution product (chloral).

*Paraldehyde*, $(CH_3COH)_3$.
*Metaldehyde*, $(CH_3COH)_n$.
*Thioaldehyde, or Sulphaldehyde*, $(CH_3CSH)_n$.

### CHLORAL COMBINATIONS.

*Chloral Ammonium*—

$$CCl_3CH(OH)NH_2.$$

*Chloralamide* (formamide), $CCl_3.CH(OH).NH.COH$.
*Chloralimide*, $(CCl_3.CH:NH)_3$.

*Chloral Cyanhydrate*—

$$CCl_3.CHO.HCN.$$

*Chloralose*—Anhydro-gluco-chloral, $C_8H_{11}Cl_3O_6$.

*Oximes of Chloral.*—These break up in the system into chloral (or chloroform), and the oxime consequently partakes of the medicinal properties of both.

*Chloral Acetaldoxime*, $CCl_3.CH(OH).O.N{:}CH.CH_3$.
*Chloral Acetoxime*, $(CH_3)_2{:}C{:}N.O.(CCl_3H.OH)$.
*Chloral Camphoroxime*, $CCl_3.CH(OH).O.N{:}C_{10}H_{16}$.
*Chloral Benzaldoxime*, $CCl_3.CH(OH).O.N{:}CHC_6H_5$.
*Chloral Nitroso-β-naphthol*—

$$CCl_3.CH(OH).O.NO.C_{10}H_6.$$

*Bromal Hydrate*—Tri-brom-aldehyde hydrate =

$$CBr_3.CH(OH)_2$$

*Butyl Chloral Hydrate* = $CH_3.CHCl.CCl_2.CH(OH)_2$.

### Ketones.

*Propion*, $C_2H_5.CO.C_2H_5$, di-ethyl-ketone.
*Hypnone*, $C_6H_5.CO.CH_3$, methyl-phenyl-ketone.
*Oxy-Camphor*—

$$C_8H_{14}(CO)CH.OH.$$

### Disulphones.

For convenience these bodies are classed here, being derived from ketones by combination with a mercaptan and subsequent oxidation. According to Baumann, only those disulphones which contain *ethyl* groups are active, and the intensity of effect depends upon the *number* of such groupings contained in the molecule.

A disulphone containing but one ethyl group—

$$CH(CH_3)(SO_2CH_3)(SO_2C_2H_5$$

produces an effect only half as intense as that of one containing two ethyl groups (sulphonal).

*Sulphonal*—Di-ethyl-sulphone-di-methyl-methane—

$$C(CH_3)_2(SO_2.C_2H_5)_2.$$

Again sulphonal is far less active than trional, a disulphone containing three ethyl groups.

*Trional*—Di-ethyl-salphone-methyl-ethyl-methane—

$$CH_3(C_2H_5).C(SO_2C_2H_5)_2.$$

The most active of this group is tetronal, a disulphone containing four ethyl groups.

*Tetronal*—Di-ethyl-methane-di-ethyl-sulphone—

$$C(C_2H_5)_2(SO_2C_2H_5)_2.$$

A disulphone containing four methyl groups is without medicinal action. In certain configurations the ethyl group plays a definite pharmacological part, which under like circumstances is not shown by the methyl group, as in the above compounds. Baumann administered doses of 1·5 grms. pro kilo. of the tetra-methylated disulphone without any visible effect, whilst a dose as small as 0·3 grm. pro kilo. of the tetra-ethylated disulphone (tetronal) produced powerful toxic effects.

Hospital experience throws some doubt, however, upon the superiority of tetronal: it apparently possesses all of the disadvantages of sulphonal with an action more markedly sedative than hypnotic. In practice trional is evidently supplanting the less active sulphonal.

### Amides.

*Carbamic Acid Esters or Urethanes*, $CO(OR)NH_2$: Urethane is a safe, mild, and effective hypnotic particularly adapted for children.

*Urethane.*—Ethyl-ester of carbamic acid, $CO(NH_2)OC_2H_5$: To Schmiedeberg we owe this hypnotic, he assumed that by the insertion of an alkyl radical, hypnotic action would result, whilst the carbamic acid residue would serve as an arterial stimulant. Most other hypnotics act as arterial depressants.

*Methyl Urethane.*—Methyl-ester of carbamic acid, $CO(NH_2)OCH_3$: The introduction of a phenyl group in ethyl urethane yields the antipyretic, euphorin—

$$CO.(O.C_2H_5)NH.C_6H_5.$$

This action is probably due to the splitting off of the para-oxy-amido-phenol grouping in the organism.

Products of greater hypnotic power are obtained by the introduction of substituted aldehyde groups as in—

*Uraline.*—Chloral urethane—

$$CO(OC_2H_5)NH.CCl_3CH.OH.$$

*Somnal.*—Chloral urethane alcoholate—

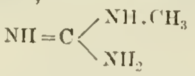
$$CO(OC_2H_5)NH.CCl_3CH(OC_2H_5).$$

### Caffeïne Derivatives.

*Ethoxy-Caffeïne.* — $C_8H_9N_4O_2(O.C_2H_5)$: The corresponding methoxy-derivative possesses the properties of a local anæsthetic and an anti-neuralgic.

*Caffeïne-Chloral*, $C_8H_{10}N_4O_2.C_2Cl_3.OH$: From the above we observe the influence exerted by the presence of an ethoxy group in producing a hypnotic from caffeïne, which is classed as a cardiac and cerebral stimulant. Of further interest in this connection is "symphorol" or "nasrol," a sodium sulpho-caffeate, though not belonging under this class, illustrates the point in view—that is, the influence exerted by the introduction of groupings. With the introduction of sulphonic acid, caffeïne loses its effect upon the vasomotoric centre while its diuretic action is retained.

*Methyl-Guanidine*,



$$NH=C\begin{cases}NH.CH_3\\NH_2\end{cases}$$

*Bromalin.* — Hexa-methylene-tetramine-bromo-ethylate $(CH_2)_6N_4.C_2H_5Br$. (Sedative.)

*Hypnoacetin.* — Acetophenone-acetyl-*p*-amido-pheuyl-ether. $CH_3.CO.NH.C_6H_4.OCH_2.CO.C_6H_5$.

*Methyl and Ethyl Phenacetin*—

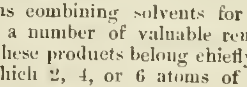
$$C_6H_4\overset{(1)}{(OC_2H_5)}.N(R)\overset{(4)}{CH_3}CO.$$

$$R=CH_3 \text{ or } C_2H_5.$$

### URIC ACID DIATHESIS.

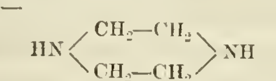
Through the endeavours to find better substitutes for the lithium salts as combining solvents for the sodium acid urate deposits, a number of valuable remedies have been discovered. These products belong chiefly to the class of diamines in which 2, 4, or 6 atoms of hydrogen in two molecules of ammonia are replaced by alkylenes. The first to appear in the market was—

*Piperazine*—



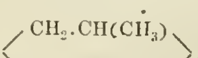
Di-ethylene-diamine. Usually employed as hydrochloride.

*Lycetol*—



Di-methyl-piperazine. The tartrate of this base constitutes commercial lycetol.

*Lysidine*—



Methyl-glyoxalidine or ethylene-ethenyl-diamine. It is claimed that lysidine possesses five times the power of piperazine as a uric acid solvent. It is usually employed in the form of a bitartrate.

*Urotropine* or *Formine.*—$N_4(CH_2)_6$:—Hexa-methylene-tetramine. The iodine derivative, "Iodoformine," is highly spoken of as an antiseptic.

*Saliformin*—

$$(CH_2)_6.N_4.C_6H_4(OH)COOH.$$

Hexa-methylene-salicylate.

*Tetra-allyl-ammonium Alum*, $N(C_3H_5)_4Al_2(SO_4)_3.12H_2O$.

*Tetra-ethyl-ammonium Hydroxide*, $(C_2H_5)_4N.OH$.

Inorganic salts such as are employed under this class are to be found under Lithium and Sodium compounds.

## ANÆSTHETICS.

### I. *Those producing Total Anæsthesia.*

*Di-chlor-methane*, methylene chloride, $CH_2Cl_2$.

*Tri-chlor-methane*, methenyl chloride, $CHCl_3$; chloroform.

Chloroform and ether constitutes Billroth's mixture.
Chloroform, ether, and alcohol is A.C.E. mixture.
Chloroform and methyl alcohol = Richardson's methylene chloride.

*Tetra-chlor-Methane*, carbon tetrachloride, $CCl_4$ = Katharin or Phœnixin.

*Ethylene Chloride* = $C_2H_4Cl_2$; Dutch liquid.

*Ethylidene Chloride* = $CH_3CHCl_2$.

*Chlor-ethylene Chloride*, vinyl tricloride, $ClCH_2.CHCl_2$.

*Tri-chlor Ethane*, methyl chloroform, $CH_3.CCl_3$.

*Chlor Butane*, $C_4H_9Cl$.

*Iso Amylene-β*-Pental—

$$(CH_3)_2C:CH.CH_3$$

*Ethyl Ether*, ordinary ether $(C_2H_5)_2O$.

*Methyl Ether*, $(CH_3)_2O$: A gas sold in ethereal solution.

The introduction of chlorine into the molecule of a hydrocarbon imparts toxic effects, increasing with the number of chlorine atoms introduced, thus, from minimum to maximum—

$$CH_3Cl,\ CH_2Cl_2,\ CHCl_3,\ CCl_4.$$

### II. *Local Anæsthetics.*

#### (*a.*) *By production of Cold through Rapid Vaporisation.*

*Methyl Chloride*, $CH_3Cl$.

*Ethyl Chloride*, $C_2H_5Cl$: "Kelen."

"Chloryl" is a mixture of the above two.

*Methyl Iodide*, $CH_3I$.

*Anesine* = a 1 per cent. aqueous suspension of tertiary-trichlorbutyl-alcohol—

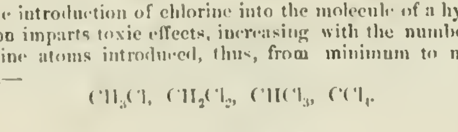
$$(CH_3)_2C.OH—CCl_3$$

*Hydramyl Ether* is a mixture of "amylhydride" and ether.

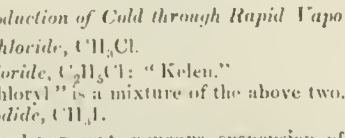
*Koenig's Ether Anæstheticus* is mixture of ether, rhigolene, and petroleum ether.

#### (*b.*) *By Production of Local Anæsthesia.*

The best known of this class is the alkaloid cocaine, which is the methyl ester of benzoylated ecgonine—



*Cocaine.*



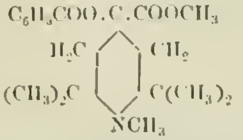
*Ecgonine.*

On removing the benzoyl or methyl group from cocaine, the resulting benzoyl or methyl ecgonine or ecgonine is devoid of local anæsthetic action. Filehne found that such benzoylated alkaloids as benzoyltropine, morphine, quinine, &c., which possess no relationship to cocaine, produced more or less local anæsthesia. From this he inferred that the presence of a benzoyl radicle was essential for the production of a local anæsthetic. Later, as a result of the examination of the other alkaloids of the coca leaf, it was found that if the benzoyl radicle in the ecgonine ester was replaced by other radicles, only certain ones (called by Ehrlich "anæsthiospores") produced anæsthetic products.

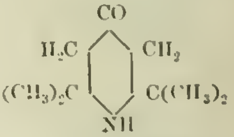
This question was partly solved by Merling who, considering that ecgonine consisted of the two ring nuclei, namely, hexa-hydro-benzene and (*n*) methyl-piperidine, prepared the benzyl methyl ester of the latter, resulting in synthetic eucaine (B).

The first synthetic substitute for cocaine was found in the older preparation known as

*Eucaine* "*A*" which is a derivative of the nucleus triaceton amine—



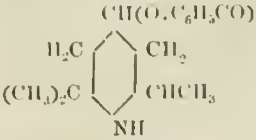
*Eucaine A.* = (*n*) methyl-benzoyl-tetra-methyl-γ-hydroxy-piperidine-carbonic acid—methyl-ester.



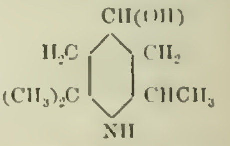
*Triaceton-amine.*

This preparation, which proved to be somewhat irritating, has been improved upon through the introduction of—

*Eucaine* "*B*" which possesses advantages over cocaine in that it is free from any irritating properties, is more active, less toxic, and does not decompose on boiling or standing in aqueous solution. It is 3¾ times less toxic than eucaine "A." Eucaine "B" is a benzoyl derivative of vinyl di-acetone-alkamine, thus—

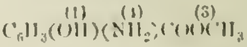


*Eucaine B* = benzoyl-vinyl-diaceton-alkamine.



*Vinyl-diaceton-alkamine.*

Einhorn and Heinz have prepared various derivatives of the above mentioned other half of the ecgonine complex, namely hexa-hydro-benzene. They found that all the esters of the aromatic hydroxy-amido acids possessed local anæsthetic properties, and particularly so the methyl ester of para-amido-meta-oxy-benzoic acid—



$$C_6H_3\overset{(1)}{(OH)}\overset{(4)}{(NH_2)}\overset{(3)}{COOCH_3}$$

which is called *orthoform*; the hydrochloride is usually employed because of ready solubility. It is claimed that orthoform is absolutely non-toxic, non-irritating, stable, and antizymotic.

Another local anæsthetic which has of late attracted considerable attention is:—

*Holocaine*, which is obtained by the interaction of phenacetin and para phenetidine—

$$OC_2H_5.C_6H_4.NH\ COCH_3 + NH_2.C_6H_4.OC_2H_5 =$$
$$OC_2H_5.C_6H_4.NH.C(CH_3):N.C_6H_4OC_2H_5 + H_2O.$$

Holocaine is then a para-diethoxy-ethenyl-di-phenyl-amidin.

*Pyrocaine.*—A non-toxic, lasting, local anæsthetic, chemically a guaiacol-benzyl-ester—

$$OCH_3.C_6H_4.OCH_2.C_6H_5$$

*Methoxy-caffeïne*—

$$C_8H_9(OCH_3)N_4O_2$$

is used subcutaneously as local anæsthetic.

The corresponding ethoxy derivative is an active sedative.

*Formanilide*, $C_6H_5NH.COH$ : Used subcutaneously.

*Eugenol acetamide*—

$$C_6H_3(CH_2.\overset{(1)}{C}H:CH_2)(O\overset{(3)}{C}H_3)(OCH_2(\overset{(4)}{N}H_2)CO)$$

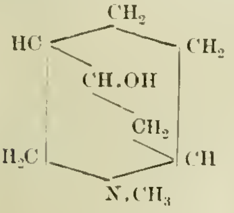
*Guaiacyl*—

$$[C_6H_4(OCH_3)(OSO_3)]_2Ca,$$

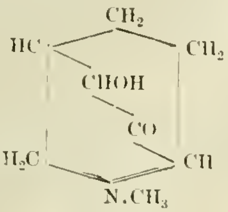
is the calcium salt of guaiacol-sulphonic acid.

### MYDRIATICS.

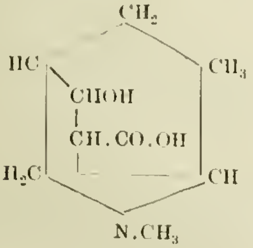
As derivatives of pyrolidine, we find a very close relationship in chemical constitution existing between the mother substances tropine, scopoline, and ecgonine, which, as tropate, atropate, and methyl benzoate, yield alkaloids possessing mydriatic properties, the latter one being employed as local anæsthetic, while the former two, in addition to the mandelate of tropine (homatropine), are simply mydriatics.



Tropine.

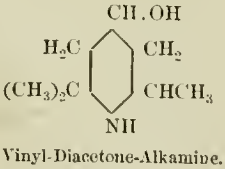


Scopoline.



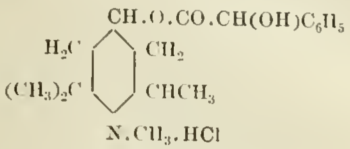
Ecgonine.

Merling ascribed the local anæsthetic powers of cocaïne to the presence of the ($n$)-methyl-piperidine ring nucleus of ecgonine, and in preparing a derivative of this he obtained eucaine "B" (benzoyl-methyl-ester of ($n$)-methyl-piperidine), a local anæsthetic, which also possesses mydriatic powers. This eucaine "B" may also be considered as a methylated benzoyl derivative of vinyl-diacetone-alkamine (belonging in this same series), which in time furnishes us with a new synthetic mydriatic called euphthalmin (an amygdalate of eucaine "B"). Of interest in this connection is a series of patents taken out on the employment of the unsymmetrical cyclic bases of the aceton alkamine series, as starting points for the synthesis of active alkaloidal bodies. For example, in the labile (unstable) forms of the unsymmetrical ($n$)-alkylated bases of the tri-acetone-alkamines, the hydrogen of the hydroxyl, when replaced by the benzoic, mandelic, tropic, or phenylacetic acid radicles, yields mydriatic substances.



Vinyl-Diacetone-Alkamine.

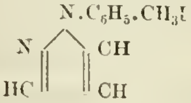
If a mandelic acid residue is introduced in methyl-vinyl-diaceton-alkamine, the already-mentioned mydriatic *euphthalmine* results. It is a hydrochloride of ($n$)-methyl-vinyl-diacetone-alkamine-phenylglycoyl or the hydrochloride of the mandelic acid derivative of eucaine "B."



Euphthalmine.

Of simpler constitution and belonging to the pyrrolidene derivatives, is the mydriatic.

*Mydrol.*—Phenyl-pyrazole-methiodide—



*Thermine.*—A tetrahydro-beta-naphthylamine hydrochloride $C_{10}H_{11}NH_2.HCl$.

This title would apply better to an antipyretic (antithermic).

### ANTISEPTICS.

Under this head are included the antiseptics proper, disinfectants, and internal antiseptics. For convenience of chemical classification they are divided into two groups, one including the disinfectants and antiseptics, external and internal, omitting, however, such as are intended to act locally in the intestines; the second group includes the intestinal antiseptics of the salol type.

#### I.—*Aliphatic Aldehydes with Combinations.*

Among the aliphatic aldehydes we have a valuable antiseptic in formaldehyde which is furnished in aqueous or alcoholic solution under a variety of names.

*Formalin or Formol* is a 40 per cent. aqueous solution of formaldehyde.

*Camolin* is a 1·5 per cent. aqueous solution of formaldehyde.

*Steresol* is a saturated solution of formaldehyde in a solution of milk-sugar.

*Holzin* is a 60 per cent. solution of formaldehyde in methyl alcohol.

*Holzinol* is a solution of formaldehyde in methyl alcohol, containing menthol.

*Formaldehyde Casein.*

*Glutol* is formaldehyde gelatin.

*Formyl Phenetidine*, $C_6H_4(OC_2H_5).NH.COH$.

*Salubrol* is a bromated methylene-di-antipyrine.

*Paraform or Triformol* is paraformaldehyde $(HCOH)x$.

*Amylo Iodoform* is a compound of formaldehyde, starch, and iodine.

*Chinoform* is a compound of formaldehyde and cinchotannic acid.

*Eka-iodoform* consists of iodoform containing 0·05 per cent. of formaldehyde.

*Protogen* is a compound of albumin with formaldehyde.

*Amyloform* is a condensation product of formaldehyde and starch.

*Dextroform* is a condensation product of formaldehyde and dextrin.

*Tannoform* is a condensation product of formaldehyde and gallo-tannic acid.

*Acetaldehyde*, $CH_3.COH$.—Used in 2 per cent. solution.

#### II.—*Mono-hydroxy-benzenes and Derivatives.*

*Phenol*, $C_6H_5OH$: Benzophenol.

The Ca, Bi, Hg, Zn, Na, phenates used as disinfectants.

*Mono-chloro-phenol* (ortho), $OH.C_6H_4Cl$ (1 : 2).

„ „ (para) „ (1 : 4).

*Tri-chloro-phenol*, $OHC_6H_2Cl_3$ (1 : 2 : 4 : 6).

Chlorolin = a mixture of mono- and trichlorophenol.

*Mono-bromo-phenol* (ortho), $OHC_6H_4Br$.

*Tri-bromo-phenol* (bromol), $OHC_6H_2Br_3$.

*Di-iodo-phenol-iodide*, $C_6H_3I_2OI$.
*Iodo-anisol*, $C_6H_4I.OCH_3$.
*Anethol*, para allyl-phenyl methyl-ether—

$$C_6H_4\overset{(4)}{(C_3H_5)}.O.\overset{(1)}{CH_3}$$

### *Sulphonic Acid Derivatives.*

*Aseptol*, sozolic acid, orthophenol sulphonic acid—

$$\overset{(1)}{OH}.C_6H_4.\overset{(2)}{SO_3H}$$

Various salts of the above are those of sodium, potassium, zinc, magnesium, lead, aluminium ("sozal"), &c.

*Sozoiodolic Acid*, di-iodo-*p*-phenol sulphonic acid—

$$\overset{(1)}{OH}.C_6H_2I_2.\overset{(4)}{SO_3H}$$

Chief salts are those of mercury, potassium, sodium, lead, and zinc.

### *Hydroxylated Homologues of Benzene.*

*Cresols*, hydroxy-toluenes—

$$OH.C_6H_4.CH_3$$

The compounds of this class are of great antiseptic power, but have the disadvantage of slight solubility in water. There are three isomerides.

*Tricresol.*—A purified mixture of the three isomerides is three times as active as carbolic acid.

For ordinary purposes of a disinfectant the crude product fractionated from coal tar has been extensively introduced under various names; there are combinations with soap, oleïc acid or sulphuric acid, which, when added to water, give a clear solution or emulsion; their names are creolin, izal, kresin, lysol, paracresol, sanatol, saprol, solutol, solveol, phenolin.

*Benzoyl-p-cresol*—

$$C_6H_4(OC_6H_5CO).CH_3$$

*Traumatol*, iodo-cresol—

$$OH.C_6H_3I.CH_3$$

*Losophan*, tri-iodo-*m*-cresol—

$$OH.C_6HI_3.CH_3$$

*Thymol*, methyl-isopropyl-phenol,

$$\overset{(1)}{OH}.C_6H_2\overset{(6)}{(C_3H_7)}.\overset{(3)}{CH_3}$$

The introduction of the propyl group enfeebles the antiseptic properties of this phenol derivative.

*Aristol*-di-thymol-di-iodide—

$$[C_6H_2(OI)(C_3H_7).CH_3]_2$$

*Carvacrol Iodide*, methyl-isopropyl-phenol-iodide—

$$C_6H_3\overset{(1)}{(OI)}\overset{(2)}{(C_3H_7)}.\overset{(4)}{CH_3}$$

*Europhen*, isobutyl-cresol-iodide—

$$(C_6H_3(C_4H_9)(CH_3)O)_2HI$$

### III.—*Di-hydroxy-benzenes and Derivatives.*

$$C_6H_4(OH)_2$$

The three isomeric di-hydroxy-benzenes are antiseptics.

*Catechol*, ortho-di-oxy-benzene.
*Resorcinol*, meta-di-oxy-benzene "resorcinol."
*Hydroquinone or quinol*, para-di-oxy-benzene.
*Guaiacol*, catechol-mono-methyl-ether—

$$\overset{(1)}{OH}.C_6H_4\overset{(2)}{(OCH)_3}$$

Guaiacol constitutes the main active constituent of beech-wood-tar creosote, which has proved itself to be a valuable antiseptic in the internal treatment of phthisis. In order to avoid the stomach disturbances caused by the creosote, various combinations have been introduced, as creosote carbonate (creosotal), oleate, phosphate, valerate ("eosote"), and tannosal (tannic acid ester). Preference has been shown of late years to the more active guaiacol in the form of its esters as—

*Guaiacol* benzoate or "benzosol"—

$$C_6H_4(OCH_3)O.C_6H_5CO.$$

,, carbonate, $CO_3(C_6H_4.OCH_3)_2$.
,, cinnamate or "styracol"—

$$C_6H_4(OCH_3).O.C_6H_4(CH:CH.CO).$$

,, phosphate, $[C_6H_4(OCH_3)O]_3 \vdots PO$.
,, salicylate, $C_6H_4(OCH_3).O.C_6H_4(OH)CO$.
,, succinate, $C_6H_4(OCH_3).O.C_2H_4C_2O_2$.
,, valerate or "geosote"—

$$C_6H_4(OCH_3).O.COCH_2.CH=(CH_3)_2$$

,, ethylenate, $C_6H_4(OCH_3)O.C_2H_4$.
,, piperidine, $C_6H_4(OCH_3)O.C_5H_{11}N$.
,, "bisulphonate of quinine" or "guaiaquin"—

$$C_6H_4(OCH_3)OHSO_3Qu.$$

Among the lately offered substitutes for guaiacol we have—

*Guaethol.*—Catechol-mono-ethyl-ester—

$$\overset{(1)}{OH}.C_6H_4\overset{(2)}{(OC_2H_5)}.$$

*Guaiacetin.*—Catechol-mono-sodium acetate—

$$C_6H_4(OH)OCH_2COONa.$$

*Eugenol.*—Propenyl guaiacol—

$$\overset{(1)}{OH}.C_6H_3\overset{(4)}{(C_3H_5)}\overset{(3)}{OCH_3}$$

The antiseptic and local anæsthetic properties of this derivative have long been known through clove oil, the old remedy for toothache.

The benzoyl and cinnamyl esters of eugenol have found favour as antiseptics, the H of the OH group being replaced.

*Eugenol Acetamide* = $C_6H_3(OCH_3).(C_3H_5).O.CH_2NH_2CO$, *p*-acetamido-eugenol.

*Acetaminal*, *p*-Acet-amido-benzyl-eugenol—

$$C_6H_3(OCH_3).(C_3H_5)O.CO.C_6H_4NHCOCH_3$$

*Iodo-eugenol* = $C_6H_3(OCH_3)(C_3H_5)OI$.

### IV.—*Trihydroxy-benzenes.*

$$C_6H_3(OH)_3$$

Of the three isomeric tri-oxy-benzenes, only one, pyrogallol (1:2:3), commands attention, which, however, is rarely employed alone, because of its intensely poisonous character.

We have an excellent antiseptic dusting powder in—

*Helicosal*, basic bismuth pyrogallate—

$$(C_6H_3=(OH)_2O)BiOH.$$

*Gallacetophenon*, methylketo-trihydroxy-benzene, Alizarin Yellow C, $C_6H_2(OH)_3.CO.CH_3$.

### V.—*Naphthols, $C_{10}H_7(OH)$.*

*α- and β-Naphthol* constitute the two isomerides.

*Naphtho-Aristol.*—Di-iodo-β-naphthol, $C_{10}H_5I_2(OH)$.

*Microcidin.*—β-naphthol-sodium, $C_{10}H_7ONa$.

The mercury and bismuth ("orphol") compounds of β-naphthol are useful additions.

*Lactol.*—Lactic ester of β-naphthol—

$$C_{10}H_7O-CH_3.CH(OH).CO.$$

*β-Naphthol* carbonate, $CO_3(C_{10}H_7)_2$.

These latter two are also included under the intestinal antiseptics.

### *Sulphonic Acid Derivatives of β-Naphthol.*

*Asoprol.*—β-naphthol-sulphonate of calcium—

$$(C_{10}H_6(OH)SO_3)_2Ca.$$

*Alumnol.*—β-naphthol sulphonate of aluminium—

$$(C_{10}H_6(OH)SO_3)_6Al_2.$$

### *Organic Acids and Derivatives.*

The antiseptic organic acids belong almost exclusively to the benzene series. The first of these, benzoic acid (phenyl-carboxylic acid), is almost altogether devoid of antiseptic properties, owing to the absence of the hydroxyl group, so characteristic of salicylic acid and its derivatives. For convenience, three derivatives of aliphatic acids are introduced here:—

*Ethyl Formate*, $HCOOC_2H_5$, antiseptic for air passages.
*Phenyl Acetic Acid*, $CH_2.C_6H_5.COOH$.
*Phenyl Propionic*, or hydrocinnamic acid—

$$C_6H_5.CH_2.CH_2.COOH.$$

### *Monohydroxy Acids.*

*Salicylic Acid.*—Ortho-hydroxy-benzoic acid—

$$\overset{(1)}{OH}.C_6H_4.\overset{(2)}{COOH}$$

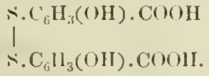
The isomeric meta and para acids are devoid of antiseptic power.

Esters formed by replacing the hydrogen of the carboxyl group by organic radicles are employed chiefly as intestinal antiseptics; hence, will be considered separately.

*Di-Iodo-Salicylic Acid*, $C_6H_2I_2(OH)COOH$.
Di-iodo-salicylic methyl ester—"sanoform,"—

$$C_6H_2I_2(OH)COOCH_3.$$

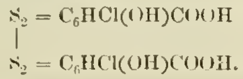
Di-iodo-salicylic phenyl ester, $C_6H_2I_2(OH)COOC_6H_5$.
*Di-Thio-Salicylic Acids*—



$$\begin{array}{l} S.C_6H_3(OH).COOH \\ | \\ S.C_6H_3(OH).COOH. \end{array}$$

Of these thio acids nine isomerides are possible, but only two are of medicinal value, being employed in form of their sodium salts, one of which, known as No. 2, is an antiseptic, whilst that known as No. 1 is an antipyretic. A mixture of these two salts is sold under the title of "Dithion."

"*Thioform*" is an antiseptic basic bismuth salt of both.
*Tetra-Thio-Chloro-Salicylic Acid*—



$$\begin{array}{l} S_2 = C_6HCl(OH)COOH \\ | \\ S_2 = C_6HCl(OH)COOH. \end{array}$$

*Salicylo-Acetic Acid*—

$$C_6H_4(OCH_2COOH)COOH$$

*Salicylamide*, $C_6H_4(OH).CONH_2$.

*Iodo-Salol*—

$$OH.C_6H_2I_2(COOC_6H_5)$$

*α-Hydroxy-Naphthoic Acid*, $C_{10}H_6(OH)COOH$.
*Anisic Acid*—Methyl-*p*-hydroxybenzoic acid—

$$C_6H_4(OCH_3)COOH$$

The phenyl ester of anisic acid, $C_6H_4(OCH_3).COOC_6H_5$.
*Phenyl Salicylic Acid*, ortho-hydroxy-diphenyl-carboxylic acid $C_6H_3(OH)(C_6H_5).COOH$.
*Cresotic or Hydroxytoluic Acids*—

$$OH.C_6H_3(CH_3)COOH$$

These bear the same relation to toluene $C_6H_5.CH_3$ as salicylic acid does to benzene. Of the three isomers, the para compound is the only one employed in medicine; this in the form of its sodium salt.

### TRI-HYDROXY ACIDS.

### *Gallic Acid, $C_6H_2(OH)_3COOH$, and Derivatives.*

In these derivatives the hydrogen of the carboxyl group is replaced by inorganic basic metals or organic residues.
*Gallobromal*, dibromo gallic acid, $C_6Br_2(OH)_3COOH$.
*Dermatol*, basic bismuth gallate—

$$C_6H_2(OH)_3.COO(Bi(OH)_2).$$

*Gallal*, basic aluminium gallate.
*Airol*, "oxyiodide of basic bismuth gallate"—

$$C_6H_2(OH)_3COOBiOHI.$$

*Gallanol*, gallic acid anilide, $C_6H_2(OH)_3COC_6H_5NH$.
*Gallicin*, methyl ether of gallic acid, $C_6H_2(OH)_3COOCH_3$.
*Iodo-Gallicin*, bismuth-iodo-methyl gallol—

$$C_6H_2(OH)_2(COOCH_3)OBiOHI.$$

### *Di-Gallic Anhydride or Tannic Acid Derivatives.*

The inorganic derivatives act as astringent disinfectants and antiseptics.
*Tannal (soluble)*, aluminium tannic tartrate—

$$Al_2(C_4H_6O_6)_4(C_{14}H_9O_9)_2.$$

*Tannal (insoluble)*, basic aluminium tannate—

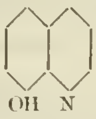
$$Al_2(OH)_4.(C_{14}H_9O_9)_2.$$

*Cutal*, aluminium boro-tannico-tartrate.
*Tannosal or Creosal*, compound of tannin and creosote.
*Salitannol*, condensation product of gallic and salicylic acids.
*Mercurous* oxide tannate.

### *Quinoline Derivatives.*

The base quinoline ($C_9H_7N$) is antiseptic, antizymotic, and antipyretic. This important base with pyridin ($C_5H_5N$) constitute the nuclei of the plant alkaloids, as well as a large number of artificial alkaloids which possess antipyretic or antiseptic properties, or both together. A number of the organic salts of quinoline are antipyretic and antiseptic. Among these we have the salicylate, tartrate, thiocyanate, quinoline iodoform ($3(C_9H_7N) + CHI_3$), quinoline resorcinol ($2(C_9H_7N) + C_6H_6O_2$).

### *Hydroxyquinolines.*



These compounds, in which the hydroxyl group is contained in the benzene nucleus, show the characters of a phenol and a base. As well-known derivatives we have the antipyretics, analgene, kairine, thalline, &c. (compare under antipyretics). In this class we have a most formidable competitor of iodoform first prepared by Claus and known as—

*Loretine*, meta-iodo-ortho-hydraoxy-quinoline-ana-sulphonic acid, $C_9H_4I.N(OH)SO_3H$. (See structural formula under antipyretics.) We have various inorganic salts which are employed as antiseptic dusting powders, as bismuth and calcium loretinate.
*Methyl-Loretine*, $C_9H_3(CH_3)I.N(OH)SO_3H$.
*Lorenit*, $C_9H_5ISO_2.OH.N.$, Para-iodo-ana-hydroxy-quinoline-ortho-sulphonate.
*Chinosol*, potassium hydroxy-quinoline-sulphonate—

$$C_9H_6N.OSO_3K.$$

*Diapthol*, quin-aseptol, ortho-hydroxy-quinoline-*m*-sulphonic amine, $C_9H_5(OH)SO_3HN$.
*Diaptherin*, hydroxy-quin-aseptol, a compound of *o*-phenol-sulphonate of *o*-hydroxy-quinoline and *o*-hydroxy-quinoline—

$$C_6H_4(OH)SO_3H.C_9H_6(OH)N.C_9H_6(OH)N.$$

### Intestinal Antiseptics.

This class includes such compounds which pass through the stomach unchanged into the intestinal tract, where, under the influence of the alkaline secretions, they are broken up into their constituents, which, in turn, serve as local antiseptics, or, in some cases, as astringents.

As excellent prototype of this group we have *Salol, Salicylic Phenyl Ester*—

$$OH.C_6H_4.COOC_6H_5.$$

This compound splits up in the intestines into salicylic acid and phenol.

*Cordol*, tribromosalol = $C_6H_4(OH)COOC_6H_2Br_3$.

*Nitrosalol*, $C_6H_4(OH)COOC_6H_4NO_2$.

*Chlorosalol* (ortho and para), $C_6H_4(OH)COOC_6H_4Cl$.

In the following compounds we replace the phenyl radicle of salol by those of cresol, xylenol, guaiacol, resorcin, and pyrogallol. Their decomposition takes place like that of salol.

*Cresalols*, salicylic cresylic esters (*o, m, p*)—

$$C_6H_4(OH)COOC_6H_4.CH_3.$$

*Xylenol Salol*, $C_6H_4(OH).COO.C_6H_3(CH_3)_2$.

*Guaiacol Salol*, $C_6H_4(OH)COO.C_6H_4.OCH_3$.

*Resorcinol Salol*, $C_6H_4(OH)COO.C_6H_4(OH)$.

*Pyrogallol Salol*, $C_6H_4(OH)COO(C_6H_3)_2$.

*Betol*, salicylic β-naphthyl ester =

$$C_6H_4(OH)COO.C_{10}H_7.$$

*Alphol*, salicylic α-naphthyl ester.

*Salacetol*, salicyl acetol, $C_6H_4(OH)COOCH_2COCH_3$.

*Salithymol*, salicylic thymol ester—

$$C_6H_4(OH)COOC_{10}H_{13}O.$$

*Benzo Naphthol*, β-naphthol benzoate—

$$C_{10}H_7O.(C_7H_5O).$$

*Chinaphthol*, β-naphthol-α-mono-sulphonate of quinine = *Qn* $(C_{10}H_6(OH)SO_3H)_2$.

*Bismal*, methylene-digallate of bismuth—

$$4[CH_2(C_6H(OH)_3COOH)_2] + 3Bi(OH)_3.$$

*Lactol*, lactic ester of β-naphthol—

$$C_{10}H_7O.CH_3.CH(OH)CO.$$

*Tannigen*, di-acetyl tannin = $C_{14}H_8(CH_3CO)_2O_9$.

*β-Naphthol Carbonate* = $CO_3(C_{10}H_7O)_2$.

*Tannalbin* = A compound of tannin and albumin.

*Tannon* = A condensation product of tannin and hexamethylene-tetramine.

*Tri-Benzoyl-gallic Acid* = $C_6H_2(O.C_6H_5CO)_3.COOH$.

*Paraform*, $(CH_2O)_3$, tri-oxymethylene.

*Salicylic Aldehyde*. $C_6H_4(OH)COH$.

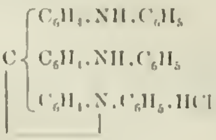
*Basic Bismuth Salts*, as cresolate, loretinate, naphtholate, subgallate, sulphophenate, hydroxy-iodo-pyrogallol, tribromophenate, salicylate.

### Aromatic Substituted Amines.

#### *Antiseptic Aniline Colours or Derivatives.*

*Diphenylamine*, $NH(C_6H_5)_2$.

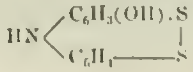
*Diphenylamine Blue*—



Tri-phenyl-*p*-rosaniline hydrochloride.

*Diphenylamine Orange* or "Aurantia," $(C_6H_2(NO_2)_3)_2NH_4$, ammonium salt of hexanitrodiphenylamine.

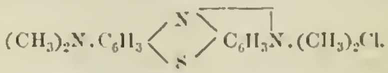
*Sulphaminol*, thio-hydroxydiphenylamine—



To this class also belongs a diamine derivative derived from dimethyl-*p*-diamidobenzene—

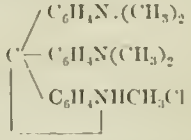
$$C_6H_4(\overset{(1)}{NH_2})\overset{(1)}{N}(CH_3)_2.$$

*Methylene Blue*, tetramethyl-*p*-thionine hydrochloride—

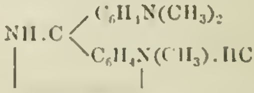


This compound is also useful as an antimalarial.

*Pyoktanine* (blue) = A mixture of penta-methyl-*p*-rosaniline and hexa-methyl-*p*-rosaniline hydrochlorides, also called "methyl violet"—



*Pyoktanin* (yellow) = The hydrochloride of imidotetramethyl-di-*p*-amido-diphenyl-methane, also called "auramine"—



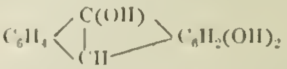
*Chrysoidine* = Di-amido-azo-benzene hydrochloride—

$$C_6H_5 - N = N - C_6H_3(NH_2)_2.HCl.$$

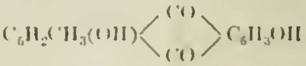
### Miscellaneous.

In order to avoid introduction of class groupings for the sake of a single product, the following are made into a miscellaneous list:—

*Anthrarobin*—Dihydroxy-anthranol is

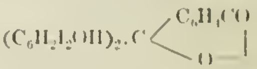


*Chrysophanic Acid*—Dihydroxy-methyl-anthraquinone—



The bismuth salt = "Dermol."

*Nosophen*—Tetra-iodo-phenol-phthaleïn—



*Cinnamic Acid*—Phenyl acrylic acid—

$$C_6H_5 - CH = CH - COOH.$$

*Aminoform*, or Urotropin, or Formin—Hexa-methylene-tretramin = $(CH_2)_6N_4$.

**Iodoform Substitutes:**

*Iodoformine* = an inodorous combination of hexa-methylene-tetramine and iodoform = $(CH_2)_6N_4.CHI_3$.

*Iodol* = Tetra-iodo-pyrrol, $C_4I_4NH$.

*Iodoformogen* = compound of iodoform and albumin.

*Di-Iodoform* = ethylene periodide = $C_2I_4$.

*Thiophene Di-iodide* = $C_4H_2I_2S$.

**Sulphuretted Tarry Products:**

*Ichthyol* = ammonium ichthyol sulphonate.

*Thiol* = sulphurated hydrocarbons obtained from peat.

*Tumenol* = sulphurated products of the action of sulphuric acid on the unsaturated hydrocarbons of mineral oils.

*Thiuret* = sulphurated oxidation product of phenyl-thio-biuret, $C_8H_7N_3S_2$.

*Di-Fluor-Diphenyl* = $C_6H_4F - C_6H_4F$.

*Hydramyl, amylhydride, pentylene, pentylhydride* = Fraction products of petroleum ether.

*Iodo-Caseine.*

*Cancroin* = tri-methyl-vinyl-ammonium-citrate, neurine citrate, $[N(CH_3)_3C_2H_3]H_3C_6H_5O_7$.

### Inorganic Antiseptics.

Among the more recent inorganic antiseptics are—
*Fluorol,* sodium fluoride.
*Bismuth* oxyiodide, BiOI.
*Aluminium* bromide.
*Magnesium Fluoride and Permanganate.*
*Calcium iodate and Permanganate.*
*Silver Fluoride.*
*Tri-Bromide* of Iodine.
*Tri-Chloride of Iodine.*

Salts of the rarer metals have received some attention, among which are the nitrates of cerium, didymium, and lanthanum. A solution of 1 part in 1,000 prevents all development of bacteria.

### *Silver Antiseptic Treatment.*

The silver treatment for septic diseases is based on the well-known fact that silver and its salts possess effective antiseptic properties, being at the same time free from any action on the organism. It has been found by Credé and Beyer that metallic silver when applied to a septic surface combines with the secretions of the micro-organisms containing lactic acid, and forms silver lactate, which in turn is very destructive to them. Among these preparations we have some which contain metallic silver in the colloidal condition, while others are organic salts. These are designed to replace the usual nitrate of silver, which is open to the objection of combining with the albumin and sodium chloride of the tissue fluids, forming an insoluble compound, thus losing in penetrative power. These newer preparations of silver possess great penetrability; also their solutions are not precipitated by saline and albuminous solutions. In most instances their aqueous solutions are not decomposed by weak acids, alkalis, alkali sulphides, or sodium chloride. In others the addition of concentrated hydrochloric acid or sodium chloride causes a precipitate of metallic silver to form, which, however, redissolves upon further addition of water. The activity of this class of antiseptics is made evident by the fact that a 1 to 1,000 solution of one of them destroys micro-organisms in five minutes, and a 1 to to 50,000 solution arrests their development.

*Silver Gauze,* muslin uniformly impregnated with finely sub-divided metallic silver.

*Hydrogol,* a solution of colloidal silver.

*Protargol,* a compound of protein and silver (8 per cent Ag).

*Argonin,* a compound of casein and silver (4 per cent. Ag).

*Largin,* a silver albumin compound containing 11·1 per cent. of silver.

*Argentamin,* ethylene diamine silver phosphate (6·5 per cent. Ag).

*Actol,* silver lactate, $AgC_3H_5O_3$.

*Itrol,* silver citrate, $Ag_3C_6H_5O_7$.

*Argentol,* silver quin-aseptolate, $C_9H_5N.OH.SO_3Ag$.

### More Recent Medicinal Metallic Compounds.

Only the more important of these classes are enumerated.

### *Aluminium.*

Of the recent aluminium compounds, the organic predominate, these retaining the astringent properties of aluminium in addition to the antiseptic or disinfectant properties of the organic or inorganic acid. The following belong exclusively to this class:—

Aluminium, aceto-tartrate "Alsol."
,, boro-formate.
,, bromide.
,, boro-tartrate, "Boral."
Aluminium, boro-tannico-tartrate, "Cutal."
,, fluoride.
,, gallate, basic, "Gallal."
,, β-naphthol-disulphonate, "Alumnol."
,, *p*-phenol-sulphonate, "Sozal."
,, potassium salicylate, "Alkasol."
,, salicylate, insoluble "Salumin."
,, ammonium salicylate, soluble "Salumin."
,, sozoiodolate.
,, tannate basic, insoluble Tannal.
,, tannico-tartrate, soluble Tannal.
,, potassium-*p*-phenol sulphonate.

### *Bismuth.*

Of recent years the number of basic organic salts of bismuth have increased considerably, attracting much attention among the medical fraternity. The majority of these combinations are intended for the treatment of gastric or intestinal disturbances. A number have already been classified among previous groups.

### *For Internal Use (mostly Antiseptics).*

Bismuth albuminate.
,, benzoate, basic, $Bi(C_6H_5COO)_3.Bi(OH)_3$.
,, borate, $BiBO_3$.
,, cerium salicylate.
,, cresolate.
,, loretinate, also external antiseptic.
,, β-naphtholate, "Orphol," $Bi_2O_2.OH.C_{16}H_7O_3$.
,, methylene di-gallic acid "Bismal."
,, naphthalin benzoate, "Intestin."
,, pancreatin.
,, peptone.
,, phenylate, also externally, $Bi:(OH)_2.(C_6H_5O)$.
,, phosphate, $BiPO_4$.
,, pyrogallate, "Helcosol"—

$$[C_6H_3(OH)_2.O]BiOH).$$

,, resorcinate.
,, salicylate, basic, $Bi(C_7H_5O_3)_3.Bi_2O_3$.
,, subgallate, "Dermatol,"—

$$C_6H_2(OH)_3.CO_2.Bi(OH)_2.$$

,, sodium benzoate and salicylate.
,, ,, iodide, $BiI_3.4NaI$.

### *External Antiseptics.*

Bismuth borophenate, "Markasol"—

$$Bi_2O_3.B(C_6H_5)CO_3.3H_2O.$$

,, chrysophanate, "Dermol," $Bi(C_{15}H_9O_4)_3Bi_2O_3$.
,, Iodogallicin—
$(C_6H_2(OH)_2.(COO.CH_3)Bi.OH.I.$
,, Oxy iodide, BiOI.
,, oxy iodo-sub-gallate, "Airol"—

$$C_6H_2(OH)_3.COO.Bi.OH.I.$$

,, oxy iodo-sub-pyrogallol—

$$C_6H_3(OH)_2.O.Bi.OH.I.$$

,, permanganate basic $Bi(MnO_4)_3.(Bi_2O_3)$.
,, sulpho-phenate.
,, sulphite, $Bi_2(SO_3)_3$.
,, tri-brom-phenate, "Xeroform"—

$$(C_6H_2Br_3OH)BiOH.Bi_2O_3.*$$

,, di-thio-salicylate, "Thioform"—

$$(S.C_6H_3(OH)COO)_2.BiO-BiO_2-Bi_2O_2+2H_2O.$$

### *Cæsium Salts.*

Recommended as antiepileptics of more pronounced activity than the alkali bromides, are caesium ammonium bromide, caesium bromide, carbonate, sulphate and caesium rubidium ammonium bromide—

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* Also given as $(C_6H_2Br_3OH)Bi_2O_3$.

$$((CsRb)Br_2 + 3NH_4Br.$$

The caesium bitartrate is useful in nervous heart palpitation, while the iodide is used as a substitute for the potassium salt for like reasons cited under rubidium iodide.

### *Rubidium Salts.*

The rubidium ammonium bromide (Rb. Br. $(NH_4Br)_3$) and Rubidium Bromide (Rb. Br.) are recommended as substitutes for potassium bromide in epilepsy, having the advantage of a more marked sedative action. The indications for the use of rubidium iodide are the same as for potassium iodide, but the former possesses the advantage of greater activity, a minimized action upon the heart, absence of gastric disturbance, and far less tendency to produce iodism.

### *Calcium.*

Calcium cresylate: disinfectant.
„ iodate: antiseptic.
„ sulpho-phenate: antiseptic,
„ loretinate: antiseptic.
„ permanganate, antiseptic.
„ salicylate: internal antiseptic.
„ ferro-phospho-lactate: rachitis.
„ albuminate: rachitis.
„ glycerino-phosphate: neurasthenia.
„ β-naphthol-α-mono-sulphonate: analgesic "Asaprol."

### *Lithium.*

Lithium benzoate: uric acid diathesis.
„ caffeine-sulphonate, uric acid diathesis "Symphorol Lithium."
„ dithio-salicylate: uric acid diathesis.
„ formate: „ „
„ guaiacate: „ „
„ hippurate: „ „
„ iodate: „ „
„ bitartrate: diuretic "Tartarlithine."
„ theobromine salicylate: diuretic.
„ potassium tartrate: „
„ acetate: „
„ boro-sulphite: antiseptic.
„ sulpho-ichthyolate: „
„ sozoiodolate: „
„ glycerino phosphate: neurasthenia.

### *Magnesium.*

Magnesium benzoate: antiseptic-antarthritic.
„ boro citrate: „ „
„ borate: „ „
„ fluoride: „
„ ichthyolate: „
„ loretinate: „
„ permanganate: „
„ phenol-sulphonate: antiseptic, intestinal.
„ salicylate: „ „
„ sozoiodolate: „ „
„ lactate: laxative, gravel.
„ ferro-lactate: „ „
„ phospho- „ „ „
„ malate „ „
„ creosote, "Kresolid," tuberculosis.

### *Mercury.*

The majority of mercurials introduced, consist of soluble salts adapted for subcutaneous use, among which are the amido-propionate, asparagate, benzoate, formamide, diethylate, di-methylate, glutino-peptonate, glycocollate, mercuric-chloride urea, imido-succinate, naphtholate, phenyloacetate, phloro-glucino-acetate, silico-fluoride, succinimide, resorcino-acetate, tri-brom-phenol-acetate.

Among the general list of newer salts we have the acetate, albuminate, di-phenate, gallate, peptonate, oxy-cyanide, pyro-borate, resorcinate, salicylate (basic), sozoiodolate, tannate, thymolate, thymo-acetate, casein mercury.

### *Sodium.*

Sodium chloroborate: antiseptic.
„ anthrarobinate: „
„ borosalicylate: analgesic and antiseptic.
„ p-cresotinate, $C_8H_7O_3Na$: analgesic and antiseptic.
„ di-nitro-cresolate, $NaC_7H_5N_2O_5$: antiseptic.
„ di-thio-salicylate, I., $Na_2C_{11}H_8O_6S_2$: „
„ „ II., $Na_2C_{14}H_8O_6S_2$: antirheumatic, antiseptic.
„ fluoride, "Fluorol": „
„ sulpho-salicylate, $C_6H_3(OH)COOHSO_3Na$: antiseptic.
„ formate, $NaCHO_2$: antitubercular antiseptic.
„ sulphophenate, $C_6H_5SO_4Na$: internal antiseptic.
„ sulpho-tumenolate: $C_{41}H_{31}O_2SO_3H$, antiseptic.
„ sozoiodolate, $C_2H_2I_2(OH).SO_3Na$: antiseptic.
„ oxynaphthoate, $C_{10}H_6(OH)Na$: antiseptic.
„ thiophenate, $C_4H_3S.SO_4Na$: antiseptic.
„ thiophen-sulphonate, $C_4H_3S_2O_3Na$.
„ acet-sulphanilide: $C_6H_4NH.(CH_3CO).SO_2ONa$, analgesic.
„ anisate, $C_6H_4(OCH_3)COONa$: analgesic.
„ di-iodo-salicylate, $NaC_7H_3I_2O_3$: analgesic.
„ boro-benzoate: kidney disturbances.
„ boro-citrate: kidney disturbances.
„ magnesium-boro-citrate: kidney disturbances.
„ hippurate, $CH_2(C_6H_5CO).NH.COONa$: uric acid diathesis.
„ sulpho-caffeate, "Symphorol": uric acid diathesis.
„ cantharidate: throat, tuberculosis.
„ cinnamate, $C_6H_5CH:CH.COONa$: tuberculosis.
„ tellurate, $Na_2TeO_4 + 5H_2O$: tuberculosis.
„ ethylate, $CH_3.CH_2ONa$: caustic.
„ kakodylate, $As(CH_3)_2ONa$: arsenical.
„ glycero phosphate, $Na_2PO_4.(C_3H_5(OH)_2$: neurasthenia.
Sodio-caffeo bromide: soluble caffeine double salts.
„ „ benzoate.
„ „ cinnamate.
„ „ salicylate.

### *Manganese.*

Manganese being a natural constituent of the blood, has found use in the treatment of anemia, chlorosis, &c. The older salts have been supplanted by soluble indifferent preparations, as the albuminate, dextrinate, mannitate, peptonate, saccharate, &c.

The iron preparations are taken up among the nutrient and tonic albuminoids.

### NUTRIENT AND TONIC ALBUMINOIDS.*

These constitute nourishing foods and restoratives, which contain a high percentage of albuminoid matter in palatable form and small bulk.

*Aleuronate.*—A patented vegetable albuminoid, containing 80 per cent. of albumin and 7 per cent. of carbohydrates.

*Carniferrin.*—A tasteless combination of phospho-carnic acid and iron, containing 30 per cent. of the latter.

*Carniferrol.*—A fluid containing 10 per cent. of meat peptone and 0·4 per cent. of iron.

*Casein-peptone.*—A predigested casein.

*Casein-sodium.*—A soluble compound of casein and sodium, 10 grains of which represents one-half a litre of milk.

*Eucasin.*—A food similar to nutrose, obtained by passing dry ammonia gas over dry casein.

*Nutrose.*—A soluble neutral compound of casein and sodium, containing 13 per cent. of nitrogen.

*Protogen.*—An egg albuminoid preparation, which does not coagulate on heating.

*Sanose.*—An albumin preparation, containing 80 per cent. of casein and 20 per cent. of albumose.

*Somatose.*—A soluble powder, which contains the albuminoid constituents of meat. Its composition is mainly:

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* This list includes a few representative nutrient albuminoids known under specific titles, without any allusion to the innumerable list of proprietary predigested albumin preparations.

albuminoids, 90·5 per cent.; phosphates, 7·5 per cent.; peptones, 0·2 per cent. Five parts represent 30 parts of beef.

*Ferro-somatose.*—Above preparation, containing 2 per cent. of iron.

*Milk-somatose.* — The albumoses of milk, containing 5 per cent. of tannin.

### INDIFFERENT COMPOUNDS OF IRON.

Compounds of iron with the inorganic acids and halogens are characterised by the readiness with which they attack the teeth, mucous surfaces, and their astringent action, this latter being a serious objection where prolonged treatment is necessary. These features have been overcome in the class known as the "indifferent compounds of iron," which are combinations of iron with albumin, peptone, and the like, being especially adapted where prolonged administration of iron is indicated, as in persistent anæmia and chlorosis.

Bunge first demonstrated the presence of iron combined with albumin in food-stuffs, preparing a nucleo-albumin iron compound from the yelk of eggs, calling it *Hæmatogen.* This combination of iron (hæmatogen) is distinguished from ordinary iron salts as well as albuminates, in that its ammoniacal solution, upon the addition of a little ammonium sulphide, does not react at once, but only after the lapse of some time, when a dark coloration takes place through the formation of iron sulphide. Bunge reasoned that if iron occurred as a nucleo-albuminate in the egg, it subsequently appeared as such in the blood of fowls; also that this same compound would be found in the human organism, having been introduced through food. A natural source for hæmatogen being impracticable, artificial methods have been devised based on the following:—As is well known, when animal or vegetable albumins are heated with alkalis, do not coagulate, owing to the formation of alkali-albuminates: upon the addition of acids to such solutions, an albuminoid substance of acid character precipitates. Such acid albumins combine with bases forming albuminates; thus, on adding a neutral ferric salt to a neutral solution of potassium albuminate, a precipitate of iron albuminate forms which is soluble in alkalis forming double salts; these give an immediate black colour with ammonium sulphide. On heating such an alkaline solution of iron-albuminate, a change in colour takes place, and the product reacts much slower with ammonium sulphide. On adding a diluted acid to this albuminate, a brown flocculent precipitate forms which dissolves readily in diluted alkalis; this solution is known as a "ferrated albuminic acid," and is distinguished from the above iron albuminate in that the blackening with ammonium sulphide takes place only after a time, a decomposition of the compound through the action of this reagent being first necessary. Again, this ferrated albuminic acid also differs from salts of iron in that by electrolysis of its ammoniacal solution hydrogen gas is evolved at the negative electrode instead of a deposition of iron taking place. Such compounds contain from 4 to 8 per cent. of iron.

Marfori endeavoured to prepare organic iron compounds from such internal organs as the liver; in this he was successful in producing a compound closely corresponding to the ferrated-albuminic acid containing 2 to 4 per cent. of iron; however, he was not able to obtain uniform products with each operation. This difficulty was overcome by Schmiedeberg, who obtained a compound of constant composition by mixing finely chopped swine livers with an excess of water, heating gradually up to boiling temperature, filtering, then adding to the filtrate tartaric acid, whereby a precipitate of "ferrated albuminic acid" was obtained. This forms a light brown powder containing 6 to 8 per cent. of iron soluble in alkaline liquids. This product is known commercially as:—

*Ferratin.*—The method of production on a commercial scale consists in the preparation of acid albumin, as above cited, but differing in the length of time of heating of the alkali albumin solution, this being continued until the fluid has lost its slimy consistency, whereby another form of acid is produced, the ferric compound of which, after drying, yields a more soluble product.

*Hæmatogen* (Bunge).—A nucleo-albuminoid preparation of iron (about 0·3 per cent.).

*Hæmatogen* (Hommel).—Defibrinated blood from which the serum has been removed, condensed to small bulk, with a minute percentage of creosote. About 0·5 per cent. of iron is present.

*Hæmatogen* (Marfori).—A soluble ferrated albuminic acid containing 0·7 per cent. of iron (Fe).

*Hæmalbumin.*—A preparation containing salts and albuminoid constituents of blood in the form of acid-non-coagulating albuminoids.

*Hæmalbumin* (*Dr. Dahmen*).—An indifferent iron preparation containing hæmatin, hæmoglobulin, serum albumin, and paraglobulin, along with the inorganic constituents of the blood.

*Hæmatin-Albumin.*—A ferrated albumin, consisting of dried blood.

*Hæmatigum.*—A red-brown clear hydro-alcoholic fluid, containing indifferent compounds of iron.

*Hæmochromogen.*—Stated to be the product of the action of acids or alkalis on hæmoglobin by absence of oxygen.

*Hæmoferrum.*—An iron albuminoid prepared by drying blood from which the serum has been removed.

*Hæmogallol.*—This with hæmol constitute two excellent indifferent preparations of iron, discovered by Kobert, being obtained by the action of reducing agents upon the hæmoglobin of ox-blood. These are distinguished from the other albuminoid preparations by greater digestibility, since they have already undergone the reduction necessary before entering the system. Pyrogallol is the reducing agent employed in the preparation of hæmogallol.

*Hæmol.*—Allied to hæmogallol, obtained by reduction of hæmoglobin through zinc dust, contains 0·2 per cent. of iron. A number of combinations of hæmol with other metals are in use, possessing the tonic properties of the former in conjunction with the medicinal properties of the latter.

*Arsen-Hæmol* contains 1 per cent. of $As_2O_3$.

*Bromoted Hæmol* contains 2·7 per cent. of bromine.

*Copper Hæmol* contains 2 per cent. of copper.

*Hydrargyro-Iodo-Hæmol* contains 12·35 of Hg and 28 per cent. of iodine.

*Iodo-Hæmol* contains 16·6 per cent. of iodine.

*Zinc Hæmol* contains 1 per cent. of zinc.

*Hæmoglobin* (*Hæmatropin*).—Red colouring matter of the blood.

*Methæmoglobin.*—Obtained by oxidation of hæmoglobin.

*Ferrum Albuminatum.*—This albumin compound contains about 5 per cent. of $Fe_2O_3$.

*Ferrum Albuminatum* (peptonised).— 0·25 per cent. of Fe.

*Ferrum Natrium Albuminatum.*

*Ferrum Caseinatum.*—52 per cent. of $Fe_2O_3$.

*Ferrum Dextrinatum.*—10 per cent. of $Fe_2O_3$.

*Ferrum Inulatum.*—10 per cent. of $Fe_2O_3$.

*Ferropyrin.*—A compound of antipyrin and iron, containing 54 per cent. of the former and 12 per cent. of the latter.

*Ferrosol.*—A saccharated ferrous oxide containing sodium chloride.

### ORGANO-THERAPEUTIC AGENTS.

#### *Animal Remedial Preparations.*

ONE of the first to call attention to this class of remedial agents was Brown-Séquard, who advanced the hypothesis that "all the glands of the body, whether they have excretory canals or not, give to the blood useful principles, the absence of which is felt when these glands are extirpated or destroyed by disease." In conjunction with D'Arsonval this hypothesis was extended to include various organs from all parts of the body, and that in a diseased condition of an organ, an extract prepared from the same organ of a healthy animal serves as a remedial agent.

Thus, for example, the testicle secretes spermatozoa and a fluid, the latter being aborbed into the system acting as a nervous stimulant and tonic. This same fluid was employed subcutaneously by Brown-Séquard when symptoms indicated such a tonic was necessary, as is the case in neurasthenia, tabes dorsalis, syphilis, &c. Again, a certain form of diabetes is due to a disorder of the pancreas, it has been found that extirpation of this organ produces the same diabetic symptoms in certain animals; according to this the internal secretion of the pancreas are important functions of this organ. This same theory has been extended to such glands and organs as the supra-renal capsules, thyroid gland, ovaries, kidneys, brain, pancreas, spinal-cord, spleen, thymus gland, testicles, &c.[*]

Concerning the chemical nature of the organic principles occurring in these secretions comparatively little is known, owing to the minute quantities present and the great difficulties of isolation, even then it is a matter of speculation as to whether an isolated principle represents the activity, until opportunities are offered to give it a thorough physiological test. To illustrate some theories applied to the action of these extracts, Dr. J. Althaus, in his study of *cerebine alpha*, an active brain extract of his preparation, suggests that its action upon the nervous system may be due, first, to the injection of a highly specialized pabulum of nervous matter, second, to the decomposition of the lecithin and protagon, which it contains, through the alkaline blood into choline, glycero-phosphoric acid and stearic acid. Choline, by reason of its oxidizing action on the blood acts as an antitoxin. The value of glycero-phosphoric acid as a nervine is well known, so that these two decomposition products might be said to represent the activity of brain extract.

These animal medicinal products are usually presented to us in either of two forms, the powder or liquid extract. Owing to the readiness with which these organs undergo putrefactive changes, the greatest care must be observed to obtain a safe preparation. The organs or glands are removed with sterilised instruments, then carefully cleaned, to remove all extraneous matter, fat, connective tissue, &c. and after slicing they are ready for extraction. The organs should always be removed by the operator himself from the freshly slaughtered animal, also all instruments, vessels, as well as the hands, should be rendered as aseptic as possible by thorough cleansing with soap and antiseptic solutions.

The powdered extract is usually prepared by expressing the juice from the glands, mixing with a definite quantity of sugar of milk, then spreading in thin layers upon glass, and drying at low temperature. These dried extracts are made to represent a definite quantity of the fresh organ, and are usually two or three times the strength of the liquid extract In other instances the cleansed glands or organs are ground up and dried entire at low temperature, observing precautions of sterilization. These products should be kept in a very dry and cool place in order to avoid any putrefactive changes.

The liquid extracts are prepared by digesting the properly cleansed, sliced, and bruised organs in a mixture of glycerin and sterilized water, or glycerin alcohol and boric acid, or glycerin and 0·5 per cent. carbolised water: the resulting solution should be strictly asceptic, particularly if employed subcutaneously. Another class of liquid extracts (opo-therapeutic) represent the active secretions of the various organs deprived of their inert albuminoids. Owing to the basic character of these secretions (belonging to the class of leucomaines) advantage is taken of the readiness with which many of these form very soluble and diffusible double salts with sodium chloride, the resulting extracts representing from 10 to 20 parts of the fresh organ.

### Thyroid Extracts.

Employed in treatment of psoriasis, eczema, lupus, sporadic cretinism, exophthalmic goitre, myxœdema, obesity, uterine fibroma, &c.

The glands from freshly slaughtered sheep are preferred.

*Thyreoidinum Siccatum.*—The purified and dried gland, 1 part representing 6 parts of fresh gland. Natkin claims to have isolated two active albuminoids from the normal gland, thyroproteid and thyreoidin.

*Thyroproteid.*—Forms dry scales, stated to be a uniform compound and very toxic. Acts first as an excitant then as paralysant on the central nervous system.

*Thyreoidin.*—Forms dry scales, consists of two albuminoids, one a globuline and the other an important enzyme. Dose 0·01 grm. once or twice daily. When employed in aqueous solution these extracts require the addition of a few drops of chloroform to prevent any decomposition.

*Iodothyrin*—[*Thyro-Iodin or Thyrein*].—A compound isolated by Prof. Baumann from the thyroid glands of sheep by means of a 10 per cent. sulphuric acid. It forms a brown powder containing 9·3 per cent. of iodine and 0·5 per cent. of phosphorus. Dose, 1 to 2 grm. daily.

*Thyraden*—[*Thyreoidin—Extract Thyreoda Hauf.*]—A stable, uniform preparation, without any unpleasant side action. One grm. of this sugar of milk trituration is equivalent to 2 grms. of the fresh gland containing 0·7 mgrm. of iodine.

*Thyreoantitoxin.*—A nitrogenated crystalline principle free from iodine. Supposed to be a guanidine derivative.

*Thyroidine* [*Hammond*].—A sterilized liquid extract of thyroid gland of sheep. Dose, 5 drops.

*Aiodin* [*Roche*].—Tablets containing active matter of thyroid gland, contains 0·4 per cent. of iodine.

*Opo-thyreoidin.*—A liquid extract, see opo-therapeutics.

### Thymus Extracts.

Prepared from the thymus gland of young sheep or pigs. Employed in like disturbances as the thyroid gland, the chief distinction being that preparations of the thymus gland do not produce any disturbances of the heart or loss in weight. Also used in struma and Basedow's disease.

*Thymus Siccatus Pulv.*—Each 0·05 grm. of the preparation represents 0·3 grm. of the fresh gland. Dose, 2·5 to 5 grm. daily.

*Nucleohiston.*—An albuminoid body prepared from the lymph of the thymus gland of calves.

*Opothymiin.*—A liquid extract, see opo-therapeutics.

### Brain Extracts.

The products of the grey brain matter of calves are employed in treatment of neurasthenia, agoraphobia, chorea, psychosis, &c.

*Cerebrum Exsiccatum.*—The dried and powdered organ, 1 part of which represents 5 parts of the fresh organ.

*Cerebrinin or Cerebrine Alpha.*—A sterilised liquid extract; extract prepared by Dr. J. Althaus.[*]

*Cerebrinum* [*Paul*].—A liquid extract.

*Cerebrine* [*Hammond*].—A sterilised liquid extract.

*Liquor Cerebri Sterilisatus.*

### Pituitary Body Extracts.

Prepared from the pituitary body of the sheep. This is a small mass at the base of the brain, and weighs about $\frac{3}{4}$ grm. From the idea that acromegalia arose from a functional disturbance of the pituitary body, Marinesco applied extracts of this organ in such cases with considerable success. He employed tablets containing 0·1 grm. of the dried body.

*Hypophysis Cerebri Sicc. Pulv.*—One part of this powdered gland represents 6·5 parts of the fresh product.

*Opo-Hypophysin.*—A liquid extract. See opo-therapeutics.

### Kidney Extracts.

Employed chiefly in nephritis.

*Renes Siccati.*—The dried and powdered kidney of the sheep or calf, one part of which represents six parts of the fresh organ. Dose, 0·5 to 1 grm. three times daily.

*Renes* [*Roche*].—Dried extract made into tablets.

*Opo-Renin.*—A liquid extract. See opo-therapeutics.

*Succus Renalis* (*E. and B.*).—A liquid extract of kidneys prepared like the succus e testibus.

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[*] An interesting paper upon this subject is that of C. E. Stuart on Animal Extracts, Phar. Jour. 25, 177.

[*] *Lancet*, December 2, 1893.

### Suprarenal Capsules.

When administered internally, extracts of these organs cause a contraction of the arteries with a corresponding increase of blood pressure; a tonic influence is also exerted upon the heart. Employed in Addison's disease, diabetes insipidus, neurasthenia—in fact, all disturbances arising from a loss of vasomotoric tone.

*Glandulæ Suprarenalis Sicc. Pulv.*—The dried and powdered suprarenal capsules of recently slaughtered sheep or cattle, one part of which represents five parts of the fresh organs. Dose, 0·2 to 0·5 grm.

*Sphygmogenin.*—An active preparation of the capsules made by Dr. Fraenkel.

*Succus Glandulæ Suprarenalis (E. and B.)*—A liquid extract prepared like the succus e testibus.

*Suprarenaden.*—A dry extract of the capsules. Dose, 1 to 1·5 grm.

*Supradin [Roche].*—A dry extract containing 0·015 per cent. iodin.

*Opo-Suprarenaliin.*—A liquid extract.

*Ext. Suprarenal Hæmostatic [Merck].*—An extract prepared from the dried capsules, the aqueous solution of which causes a strong contraction of the blood vessels of mucous surfaces; hence employed (usually in conjunction with cocaine) as hæmostatic in capillary hæmorrhages.

### Bone-Marrow Extracts.

Latest investigations have established the fact that the red blood corpuscles are chiefly developed in the red marrow of bones; hence the value of these preparations in anæmia and chlorosis. Dr. Mason prepares a glycerin extract by macerating the red cancellous tissue obtained from the head of the femur of calves.

*Medulla Ossium Rubra Sicc. Pulv.*—The powdered red marrow.

*Opo-Medullin.*—A fluid preparation of the red marrow.

*Opo-Ossiin.*—A fluid preparation of the bone marrow.

To these may be added *Medulladen* and *Myelen.*

*Ossagen.*—A calcium salt of the fatty acids of red bone marrow.

### Spleen Extracts.

The various preparations of the spleen are employed in treatment of leucocythemia, Hodgkin's disease, anæmia, myxœdema, rachitis, enlarged spleen, &c.

*Lien Sicc. Pulv.*—The dried and powdered spleen of calves or sheep, one part of which represents five parts of the fresh organ.

*Linadin* (Roche).—A dry extract made into tablets, containing in addition about 1 per cent. of iron and from 0·015 to 0·02 per cent. of iodin.

*Nuclein (Horbaczewski).*—A brown powder being a digestive product of the spleen.

*Nucleohiston.*—An albuminoid powder obtained from the lymphatic glands and spleen.

*Eurythol.*—A syrupy extract.

*Opo-Lienin.*—A liquid extract.

Of the heart extracts we have the *Cardine* (Hammond), a sterilised extract of the heart of the ox. Employed as heart tonic.

### Prostate-Gland Extracts.

Preparations of the prostate gland of the bull are employed in hypertrophic conditions of the prostate gland.

*Prostata Siccata Pulv.*—The dried and powdered gland, one part of which represents six parts of the fresh.

*Prostata (Roche).*—A dry extract in tablet form.

*Opo prostiin.*—A liquid extract.

### Testicle Extracts: Orchitic Fluid.

Prepared from the testicular fluid of the ram, and employed in treatment of mental diseases, hysteria, neurasthenia, &c.

*Spermin (Testicular Fluid).*—This, a sterilised testicular fluid, so warmly advocated by Brown Séquard, appears to be best adapted in such mental diseases as ataxy and delirious epilepsy. Dr. Poehl found the principle (leucomaine), spermin, in various other organs of the body. he not regarding it as a specific for any particular form of malady, but as a promotive of the oxidations of the body.

*Spermin (Poehl).*—A solution of the hydrochloride of the base spermin, $C_5H_{14}N$, which occurs in the testicular fluid and other glands. Spermin is a powerful tonic stimulant for the entire nervous system, and is employed in all diseases caused by auto-intoxication, as neurasthenia, tabes dorsalis, phthisis, scurvy, marasmus, &c. The preparations employed are, spermin (Poehl), a 2 per cent. sterilised extract, and also an alcoholic solution of spermin sodium chloride.

*Succus e Testibus Paratus* (Egasse and Bouyé).—A liquid extract prepared from the testes of the bull. The preparation No. 0 is forced through d'Arsonval's filter by means of carbonic acid pressure, while No. 1 is sterilised in d'Arsonval's autoclave under a pressure of carbonic acid.

*Testaden.*—An extract of the testes of the bull.

*Didymin.*—A dry extract of the testes of the bull prepared in tablet form.

*Testine* (Hammond).—A sterilised extract of the testes of the ram.

*Testidin.*—A brown liquid extract of the testes.

*Testis* (Roche).—A dry extract prepared in tablet form.

*Testin* (Stroschein).—Tablets containing 0·2 per cent. of extract.

*Opo-orchidin.*—A liquid extract from the testes.

*Testes Siccati Pulv.*—Dried and powdered testes of the bull. One part represents six parts of the glands.

### Ovaries Extract.

Extracts of the fresh ovaries of the cow or swine are employed in amenorrhœa, caused by atrophy of genitals, also difficulties after total or partial extirpation of uterus or ovaries, also in chlorosis.

*Ovariinum Siccum.*—The dried and powdered ovaries of the cow, one part of which represents five parts of the fresh organs.

*Ovadin.*—A dry extract, in tablet form, containing 0·00127 to 0·0048 per cent. of iodin.

*Ovaraden.*—One part of this dry extract represents two parts of the fresh ovaries.

*Oophorin.*—An extract, one part of which represents six parts of the fresh ovaries.

*Opo-ovulin.*—A liquid extract.

### Miscellaneous.

*Musculine* (Hammond).—An extract of the muscular tissue of the ox.

*Mammæ* (Roche).—A dry extract prepared from the fresh udder of the cow.

*Mammæ Sicc. Pulv.*—One part of the dry powder represents 8·75 parts of the fresh cow's udder.

*Pulmonin.*—An extract prepared from calves' lungs.

The author was indebted to Messrs. Schering and Glatz, to the Farbenfabriken Company of Elberfeld, the Fischer Chemical Co., Bischoff and Co., and also to the College of Pharmacy for the specimens exhibited that evening.

### Discussion.

Dr. Marston T. Bogert said that although many investigations had been and were now being carried on to determine the exact physiological action of various chemical groups, this part of chemical science was yet in its infancy, and offered a fruitful field for further research. In studying the physiological action of such complex molecules as some of those reviewed by the author, it was no easy task to determine just which chemical group was responsible for any given symptom. Even in the relatively easier task of studying the changes in the physiological effect when the hydrogen atoms of a hydrocarbon were successively replaced by other groups, it was not only a question of the chemical composition of the product, but of other factors, such as solubility, molecular weight, boiling point, &c. Take such an apparently simple case as that of methane and its chlorine derivatives. When one hydrogen atom was substituted by a chlorine atom the anæsthetic effect of the marsh gas was greatly increased, while the gaseous state of the product caused its rapid elimination from the system. As more hydrogens were substituted by chlorine, the boiling point rose very considerably, the vapour became correspondingly heavier, and remained longer in the lungs; and although chemically the resultant bodies should be more vigorous anæsthetics than the mono-chlor, the increasing density of the vapour caused a gradual retardation both in action and in recovery, a longer duration of the convulsive stage, with gathering danger of depressing the heart. So that not only must the peculiar action of the atom or group itself be studied, but also the physical properties of the resulting compound. Thus, many substances which, as far as their chemical structure indicated, should be exceedingly energetic in action, were practically useless because of their insolubility.

The caffeine group, to which the author had referred, was worthy of special notice on account of Bouchard's theory of sleep. It was maintained by this eminent investigator that the alkaloids produced during the day, when the human mechanism was at work, were sedative in character and tended to induce sleep; while those produced during sleep, while the machinery was at rest, were stimulating in nature, and, by their accumulation, excited the nervous system and caused the person to awaken. This theory found an interesting corroboration in the caffeine group. Many of the alkaloids of the human body were closely related to this group, and the author (Prof. Coblentz) had shown how the introduction of an alkyloxy group changed the cerebro-spinal stimulant caffeine into an almost equally powerful sedative. Why should not nature accomplish similar results in the laboratory of the human system?

A very interesting explanation of the action of antipyretics had been given by Prof. T. Lauder Brunton. In brief, it was to the following effect:—The protoplasmic cells of the body contain, in addition to the protoplasm itself, also the paraplasm or cell-juice. This paraplasm offers considerable resistance to the diffusion of oxygen through it, thereby restricting the amount which reaches the protoplasm, and controlling the combustion going on in the cell. When the protoplasm is contracted, as it is supposed to be in the healthy body, it assumes a spheroidal shape, offering a minimum surface for the absorption of oxygen, and protected on all sides by a maximum thickness of paraplasm, so that little oxygen gets through and the fire burns low. When, however, the protoplasm is extended, as it is assumed to be in fevers, and as Lister has shown it to be in inflammations, there will be offered a maximum surface for the absorption of oxygen, with a minimum thickness of protecting paraplasm; and accordingly, combustion goes on rapidly, and the fire burns fiercely. Now, the antipyretic is believed to act upon these protoplasmic cells in such a way that there is produced an immediate contraction of the protoplasm, cutting down thereby the supply of oxygen, closing the damper, and checking the fire. In confirmation of this theory, it should be recalled that Binz has shown that quinine causes the leucocytes of the blood to contract.

Mr. R. C. Woodcock said that recently a physician who was administering Methyl Violet to a patient had asked him to make a microscopic examination of the urine, and afterwards an examination was also made of the blood. Finding the urine coloured, and the leucocytes in the blood dark, he concluded that the leucocytes were dead. Some years ago, when he was examining yeast, a brewer would bring a sample and state that 50 per cent. of the yeast was dead; it was very difficult to determine in the laboratory whether 50 or 60 or 80 per cent. was dead. But when he came to examine it microscopically, and applied one of the aniline dyes, the dead cells became coloured, but not the living ones; and it was found by this means that 50 per cent. of the yeast was dead. The living protoplasm could not be stained, but when dead it was stained with the greatest facility, and he therefore presumed there was a large number of dead leucocytes in the system.

Dr. Endeman said that the chemist who had investigated living and dead protoplasm most thoroughly was Prof. Oscar Loew, who was at one time in New York, later in Munich, and Tokio, Japan, and was now again in Munich. His experiments showed that living protoplasm immersed in metallic solutions reduced metallic oxides, and the living protoplasm became darkly coloured; whilst if the life of the protoplasm were destroyed, metallic solutions produced at most a very faint brown colour upon the cell. Loew's experiments should be combined with those of Mr. Woodcock; a sample of yeast should be treated with Methyl Violet, and if it should be found that 10 per cent. of the cells were coloured, which might be assumed to indicate that the cells were dead, he should propose that a sample of the same yeast be taken and treated according to the method of Prof. Loew with a silver solution, when it would be expected that 90 per cent. of the cells would be coloured. The two experiments combined would solve the question whether the Methyl Violet test was reliable.

Prof. S. A. Tucker asked if those compounds that were decomposed by the alkalinity of the pancreatic juice did not suffer decomposition in passing through the stomach where they met the hydrochloric acid of the gastric juice. It was known that the unorganised ferments contained in pancreatin were decomposed in acid solution, and therefore, was it not quite possible that they were decomposed on meeting the dilute hydrochloric acid of the gastric juice?

Dr. E. E. Smith said that pancreatin probably belonged to the class of proteoses or albumoses, and in the stomach underwent digestion. Its physiological action was lost after passing through the acid gastric juice. But in the case of these other compounds a problem of a different nature was presented. In regard to the question raised by Mr. Woodcock, a series of articles were published some eight or ten years ago in one of the British periodicals, by a woman who did some work on the lower forms of animal life. She was able to introduce the aniline dyes into the protoplasm of amœba, and they yet retained their active movements, retained their evidences of life. It was thus quite possible that the leucocytes of the blood might take up aniline dyes without being killed.

Dr. V. Coblentz, in answer to Dr. Bogert, said that until they were actually tried, the physiological action of these synthetic substances could not be determined, for it was found in many cases that those which were readily broken up by reagents frequently did not suffer any decomposition in passing through the system and *vice versâ*. Take the case of the iodides of the alkalis. It was not the percentage of iodine that determined activity, for if so, then then sodium iodide would be the most active and rubidium iodide the least. Erdmann had shown that the potassium salts owed their more prompt action, as compared to sodium, to their higher molecular weight and greater electrical conductivity; from this the rubidium salts might naturally be expected to take a higher position for similar reasons. The electrolytic conductivity of the iodides of the alkali metals showed an ascending progression like that of their molecular weights. Their data not only represented the actual and relative rapidity with which the different iodides were distributed throughout the organism, but also afforded a direct measure of their degree of medicinal activity. Physiological activity was conditioned by the ability to enter into chemical reactions, and, according to Ostwald, chemical activity was proportional to the electrolytic conductivity. This explained why the practitioner preferred potassium iodide to the sodium salt, although the latter contained 85 per cent. of iodine, compared to 76 per cent. of the former. Upon the same grounds, rubidium iodide, containing about 60 per cent. of iodine, was claimed to be far more active than either of the others.

In reply to Prof. Tucker, the compounds which were decomposed by the alkalinity of the pancreatic juice did not suffer decomposition to any great extent; for some of them—as, for example, salol—were made use of very largely for coating pills containing medicinal agents which were intended to act on the intestines. When taken internally these pills passed through the stomach into the intestines, and there the alkaline secretions decomposed the coating.

A vote of thanks was then tendered to the Chairman for the excellent way in which he had filled the office and his devotion to the interests of the Society, and to the College of Pharmacy for granting the use of their meeting room.

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ERRATUM.

## PROCEEDINGS OF THE SEVENTEENTH ANNUAL MEETING.

(*This Journal, July* 1898, p. 637, Col. 1.)

In Mr. Forbes Carpenter's remarks, 5th line, the word "advertisements" should be "investments."

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# Scottish Section.

## THE REDUCTION OF NITRIC OXIDE BY COPPER WITH SPECIAL REFERENCE TO DUMAS' METHOD OF ESTIMATING NITROGEN.

BY THOMAS GRAY, B.SC.

In the execution of Dumas' absolute method of estimating nitrogen in organic substances, various experimenters (Frankland, Phil. Trans. vol. 147, p. 63 (1857); Hempel, Fres. Zeit. 1878, p. 415; Pflüger, Fres. Zeit. 1879, p. 301; O'Sullivan, this Journal, 1892, p. 327; Mörner, Fres. Zeit. 1898, p. 6), have experienced a difficulty in completely reducing the nitric oxide by means of heated copper. O'Sullivan (*loc. cit.*) quotes the results of a very large number of analyses, using Frankland and Armstrong's modification of the method, and states as his experience that "the gas obtained invariably contains an appreciable quantity of nitric oxide, and that the amount is not much influenced by the presence of the usual copper in the combustion tube." Thus he found in two combustions of malt that the resulting gas contained 8·14 and 9·55 per cent. of nitric oxide, while the gas from a combustion carried out under the same conditions, but using a copper spiral contained 9·0 per cent. of NO. As the result of a large number of estimations by Frankland's method of the amount of nitrogen in malt and barley, he shows that the gases obtained did not contain much more nitric oxide when a plug of asbestos was substituted for the usual copper spiral. These observations caused me to undertake some experiments on the reduction of nitric oxide by means of copper, the results of which are given below.

A combustion tube, containing three closely rolled, tightly fitting, copper spirals, reduced in alcohol vapour, was placed on a furnace, so that about 14 in. of the copper could be effectively heated, and connected by means of a T tube to a supply of pure nitric oxide, and to a Kipp charged with air-free hydrochloric acid and boiled-out marble. The air was first expelled from the tube by means of a current of $CO_2$, and the copper spirals were then heated to redness during the passage of the $CO_2$ to expel any occluded gas; the $CO_2$ was then stopped, a current of nitric oxide was passed through the tube, and the gas which escaped was collected in an azotometer over caustic potash in the ordinary way. From this the gas was transferred to a Hempel burette and measured; the nitric oxide present was estimated by absorption by means of ferrous sulphate solution in a Hempel absorber. The volume of gas collected was usually large, and was only estimated to 0·1 c.c., as no special accuracy was desired.

In order to obtain a rough idea of the reducing power of the copper a rapid current of nitric oxide was passed over the heated metal. The resulting 90·5 c.c. of gas were found to contain 12 c.c. or 13·2 per cent. of nitric oxide. The rate at which the gas was passed in this preliminary experiment was not taken, and this is all the more unfortunate, as in no other case did the quantity of unreduced nitric oxide even approach this value. As it occurred to me at a later stage of this work that a spiral which had been several times oxidised and reduced might prove more efficient than a new spiral on account of the larger surface exposed by the repeated oxidation, two experiments were carried out with a mixture of nitrogen and nitric oxide containing 15·9 per cent. of the latter, using in one case an old spiral which had been repeatedly oxidised and reduced, and in the other one freshly made. In both cases the passage of the gas was so regulated, that approximately equal quantities (about 50 c.c.) were collected in 4 to 4½ mins. The gas which had been passed over the old spiral contained practically no nitric oxide (less than 0·1 c.c.), while that from the new spiral contained 0·3 c.c. This seemed to indicate a difference in efficiency, but as it was not sufficient to account for the large amount of nitric oxide which escaped reduction in the first experiment, this point was not further investigated. The only explanation I can offer is that the copper spirals may not have been at a sufficiently high temperature. In subsequent experiments care was taken to raise the temperature of the copper to a good red heat before admitting the gas; the rate was estimated by counting the bubbles as they entered the measurer, and the time was taken in each case. The following table gives the results:—

| Volume of Gas Collected. | Time. | Volume of NO. |
|---|---|---|
| C.c. | Minutes. | |
| 87·9 | 25 | None. |
| 45·5 | 19 | None. |
| 49·1 | 8 | None. |
| 53·0 | 5 | None. |
| 87·7 | 2¼ | None. |
| 75·9 | 1½ | 0·8 c.c. |
| 87·6 | 1½ | None. |

In the last three experiments the gas passed so quickly through the tube that it was not possible to count the bubbles as they entered the azotometer. The above results show that copper, if sufficiently heated, is a very efficient reducing agent; only in one case did any nitric oxide escape reduction, and the gas in that experiment was passed over the copper at a rate very much greater than that at which the gas is evolved in a well-conducted combustion.

In order to test the copper under conditions more nearly approaching those of a nitrogen estimation, some experiments were next made with mixtures of nitrogen and nitric oxide, and a spiral 5 in. long. The mixtures were made roughly by admitting the gases into a graduated cylindrical gas-holder, filled with mercury, and the composition was afterwards more accurately determined by absorbing the nitric oxide by means of ferrous sulphate.

*Mixture I.*

$N_2$, 89·4 per cent.; NO. 10·6 per cent. 5-in. spiral.

| Volume of Gas Collected. | Time. | Volume absorbed by $FeSO_4$. |
|---|---|---|
| C.c. | Min. | C.c. |
| 30·3 | 9 | 0·2 |
| 33·9 | 6½ | 0·1 |
| 30·2 | 2¼ | 0·1 |
| 49·9 | 2 | None. |

*Mixture II.*

$N_2$, 84·1 per cent.; NO, 15·9 per cent. 5-in. spiral.

| | | |
|---|---|---|
| Cc. | Min. | |
| 47·6 | 4¼ | None. |
| 57·3 | 2 | None. |
| 59·3 | 1½ | None. |

The results of these experiments seemed to indicate that the efficiency of the spiral increased with repeated use. An experiment was therefore carried out at this stage with Mixture II., using a new 5-in. spiral: 46·7 c.c. of gas were collected in 4½ minutes, and found to contain 0·3 c.c. of NO, a result which confirmed this view.

A mixture of approximately the same composition as II. was next made, in order to compare the effect of 3 in. and 4 in. spirals with that of the spiral 5 in. long, and further experiments were subsequently made with mixtures containing nitric oxide in larger quantity. The results were as follows:—

*Mixture III.*

$N_2$, 85·1 per cent.; NO, 11·9 per cent.

| Volume of Gas collected. | Time. | Volume of NO. | Length of Spiral. |
|---|---|---|---|
| C.c. | Min. | C.c. | Ins. |
| 48·5 | 3 | None | 3 |
| 49·3 | 1¾ | 1·0 (2 per cent.) | 3 |
| 51·3 | 1¼ | 1·0 (1·95 „ ) | 3 |
| 46·8 | 1¾ | 1·0 (2·1 „ ) | 4 |
| 33·2 | 1 | 0·8 (2·4 „ ) | 4 |

*Mixture IV.*

$N_2$, 72·25 per cent.; NO, 27·75 per cent.

| Volume of Gas collected. | Time. | Volume of NO. | Length of Spiral. |
|---|---|---|---|
| 32·9 | 3½ | 0·1 | 3 |
| 35·3 | 1¼ | 0·9 (2·5 per cent.) | 3 |
| 36·0 | 1 | None | 5 |

*Mixture V.*

$N_2$, 52·8 per cent.; NO, 47·2 per cent.

| Volume of Gas collected. | Time. | Volume of NO. | Length of Spiral. |
|---|---|---|---|
| 29·6 | 4 | None | 3 |
| 31·8 | 1 | None | 5 |
| 30·4 | 1½ | 1·2 (3·95 per cent.) | 4 |

*Pure Nitric Oxide.*

| Volume of Gas collected. | Time. | Volume of NO. | Length of Spiral. |
|---|---|---|---|
| 30·5 | 3½ | None | 5 |

An examination of those results shows that the 3-in. spiral effected complete reduction of the nitric oxide when the mixed gases were passed over it at such a rate that approximately 30 c.c were collected in about 3 minutes, but that part of the nitric oxide escaped reduction when the speed was increased much above this; with a 5-in. spiral complete reduction took place even when the rate of passage was about three times as great. This rate is very much greater than that at which a combustion is conducted, but the conditions are not identical, as the gases in the latter case are diluted to a considerable extent by the carbon dioxide and water vapour produced by the oxidation of the carbon and hydrogen of the substance. The results, however, indicated the possibility of completely reducing the nitric oxide, though they could not be considered to prove this conclusively, and the gases obtained from a number of combustions were therefore examined. The combustions of acetanilide and of uric acid were conducted by students who were carrying out the method for the first time. A 5-in. spiral was used in all cases, and instructions were given to heat this strongly. The numbers in the second column give the time between the first appearance of gas in the azotometer and the end of the combustion.

*Acetanilide.*

| Volume of Gas Collected. | Time. | Amount of NO. |
|---|---|---|
| C.c. | Hours. | |
| 13·9 | 3 | None. |

In a combustion of acetanilide by Frankland and Armstrong's method, but without a spiral, Mörner (*loc. cit.*) found 0·78 c.c. of nitric oxide in 8·91 c.c. of gas collected.

*Uric Acid.*

| Volume of Gas Collected. | Time. | Amount of NO. |
|---|---|---|
| C.c. | Minutes. | |
| 85·5 | 45 | None. |
| 31·6 | 75 | 0·2 c.c. |
| 55·0 | 43 | None. |
| 44·7 | 40 | 0·2 c.c. |
| 43·1 | 45 | None. |

During the first four combustions of uric acid the magnesite was heated, so that a slow current of carbon dioxide passed through the tube. In the last combustion and in the subsequent experiments with dinitrobenzene and hippuric acid, the current of $CO_2$ was completely stopped during the combustion.

A combustion of uric acid carried out without a spiral, gave 41·1 c.c. of gas, of which 2·4 c.c., or 5·8 per cent., was nitric oxide. O'Sullivan, in two combustions without copper, obtained 5·45 per cent. (vacuum broken) and 4·80 per cent. (vacuum unbroken).

As it seemed probable that a nitro-body would yield a gas richer in nitric oxide, two combustions of *m*-dinitrobenzene were carried out, at my request, by Mr. Wm. S. Denham. The nitrogen in both cases was quite free from nitric oxide.

| Weight of Substance. | Volume of Gas. | Time. | Percentage of Nitrogen. | |
|---|---|---|---|---|
| | | | Found. | Calculated. |
| Gr. | C.c. | Hours. | | |
| 0·1565 | 22·8 | 1¼ | 16·97 | 16·66 |
| 0·1640 | 24·0 | 1½ | 16·74 | .. |

Two combustions of *m*-dinitrobenzene were next carried out without a spiral, with the unexpected result that the amount of nitric oxide in the gas obtained was much less than in the case of uric acid.

| Volume of Gas. | Amount of NO. | Percentage of Nitrogen. |
|---|---|---|
| C.c. | C.c. | |
| 21 | 0·4 | 1·9 |
| 23 | 0·4 | 1·7 |

After correction for the nitric oxide, the quantities of nitrogen obtained were 16·69 per cent. and 16·68 per cent.

*Hippuric Acid.*

Mörner (Fres. Zeit. 1898, p. 11) quotes the results of four combustions of hippuric acid by Frankland and Armstrong's method using a copper spiral. He found that the gas remaining, after absorbing the $CO_2$ in every case, contained an appreciable quantity of nitric oxide, and in two cases as much as 11·6 per cent. and 12·8 per cent.

These observations caused me to examine the gas obtained by the combustion of hippuric acid, with the result that the nitrogen obtained in the two experiments carried out was found to be perfectly free from nitric oxide.

| Weight of Substance. | Time. | Volume of Gas. | Percentage of Nitrogen. | |
|---|---|---|---|---|
| | | | Found. | Calculated. |
| Gr. | Min. | C.c. Mm. | | |
| 0·1926 | 70 | 12·8 at 17° and 757 | 7·68 | 7·82 |
| 0·2020 | 75 | 13·4 at 15° and 752 | 7·68 | .. |

An examination of the results stated above shows that the gas obtained in all but two cases was free from nitric oxide. In these two cases the amount of NO was small, *viz.*, 0·2 c.c.; the error introduced by its presence amounted only to 0·1 c.c. in 31·6 and 44·7 c.c. respectively, an error which for most practical purposes may be neglected. It should further be noted that the magnesite was heated during these two combustions, and that the gas was thus considerably diluted with carbon dioxide, a condition which militates against the complete reduction of the nitric oxide.

It is interesting to note that the five experimenters, referred to at the beginning of this paper, who have reported the presence of nitric oxide, all used the modification of the method in which the combustion is carried out in a vacuum; and, so far as I know, there is no published record of the occurrence of considerable quantities of NO in the gas obtained by the ordinary method, in which the air is expelled from the tube by means of carbon dioxide. O'Sullivan has shown that the amount of nitric oxide in the gas is substantially the same, whether the substance is burned directly in the evacuated tube or only after destroying the vacuum by heating a mixture of cane sugar and copper oxide, placed at the end of the tube for this purpose. The presence of the nitric oxide can therefore only be explained on the assumption that the temperature of the copper spiral was not sufficiently high to effect the reduction; using ordinary combustion tubing, it would probably not be possible to heat the copper strongly enough without fear of collapse, due to the pressure on the walls of the evacuated tube.

Hempel (*loc. cit.*) recommends copper powder, prepared by reducing granulated copper oxide in hydrogen as a very efficient reducing agent in this method. "Das so bereitete Kupferpulver ist schon bei schwacher Rothgluth ein unvergleichliches Reductionsmittel. Eine dichte Schicht von 5 bis 8 cm. Länge vermag selbst in Vacuo mit vollständigster Sicherheit das bei der Verbrennung der am stärksten nitrirten Verbindungen, freiwerdende Stickoxydgas vollständig zu zerlegen, was in der Luftleere mit den gebräuchlichen Kupferspiralen nicht möglich ist." Pflüger (*loc. cit.*) in his experiments on the simultaneous estimation of carbon, hydrogen, and nitrogen by combustion in a vacuum, does not seem to endorse this completely. "Spiralen von Kupferdrahtgewebe anzuwenden war bei dieser Methode nicht genügend, da die sehr viel grössere Oberfläche des Kupferpulvers noch nicht einmal ganz ausreichte um immer alles Stickoxyd zu Stickstoff völlig zu reduciren." The various experimenters, however, agree that a copper spiral at the temperature which may be safely maintained in the evacuated tube does not effect the complete reduction of the nitric oxide.

The numerous results which have been published of analyses carried out by the method in its original form, and the absence of any statement regarding the presence of nitric oxide seem to prove that any error due to the occurrence of the latter in the nitrogen is inconsiderable; the freedom from nitric oxide must be explained by the higher temperature to which the spiral is heated in the ordinary method. As the result of my experience of this method in which the air is expelled by a current of carbon dioxide, I am of the opinion that the presence of any appreciable quantity of nitric oxide need not be feared if the following conditions are observed:—A spiral, 5 in. long should be used; this should be tightly rolled and of such a thickness that some pressure is necessary to push it into the combustion tube. The spiral should be strongly heated, and the current of carbon dioxide, which is used to expel the air from the tube, should be stopped while the combustion is taking place.

## Obituary.

### JOHN ALEXANDER REINA NEWLANDS, F.I.C., F.C.S.

Discoverer of Periodicity between the Atomic Weights of the Elements;
Davy Medallist of the Royal Society.

It is with deep regret that the record of the death of J. A. R. Newlands has to be made. He passed away July 29, at his residence at Lower Clapton, N.E., at the age of 60 years. Though not a member of this Society, he was frequently present as a friend and visitor at the Meetings, in company with his brother, Mr. B. E. R. Newlands, a former chairman of the London Section of this Society, and with whom for many years Mr. J. A. R. Newlands had carried on practice in partnership as an analytical and consulting chemist in London.

When new relationships in the compounds, salts, or derivatives of a particular metal or radicle are discovered not only is a benefit conferred upon chemistry as a science, but the field of research is widened, wherein also the fruits (*viz.*, new products of commercial importance and of direct benefit to mankind) may be reaped. But when far-reaching relationships between all the known, and even so far unknown, elementary bodies are foreshadowed in a discovery, then the mere "widening" of a field or tract is scarcely adequate as a simile; the process may be more aptly likened to a universal opening of new fields of research. John A. R. Newlands was the first to evolve a definite conception of the periodic relations between the atomic weights of the elements in his Law of Octaves, and as surely he thus foreshadowed the system which has been more recently elaborated by Mendeléef and by Lothar Meyer. The new law, Newlands provisionally termed the "Law of Octaves," and it was first mooted in the "Chemical News," in Vol. VII., p. 70, Feb. 7, 1863. In another paper, in July 1864, a list of all the known elements was given, both in order of their atomic weights, and in a table giving a horizontal arrangement of the more important elements, also in order of the atomic weights, with blanks corresponding to some of the missing members of the various groups. Thus, in the trivalent group commencing with boron there was a blank next below zinc, since filled by gallium, and another below cadmium, now filled by indium. On Aug. 20, 1864, Newlands announced the existence of a simple relation between the elements when thus arranged, to the effect that the eighth element, starting from any given one, was a sort of repetition of the first. Some years later, Sir Henry Roscoe showed that the atomic weight of vanadium was 51·2 instead of 137, this reduction at once placing vanadium in the same line as the phosphorus group, and thereby confirming the periodic law.

A space of 24 years may seem a long period for a prophet to wait for recognition, even in his own country and among his own kin, but at length, in 1887, John A. R. Newlands was awarded the Davy Medal of the Royal Society for his work on the periodic law of the elements.

But though his paper, read before the Chemical Society on March 1, 1866, on the "Law of Octaves and the Causes of Numerical Relations among the Atomic Weights," was refused publication in the Journal under a rule excluding purely theoretical matter, and although from almost all quarters the new theory received discountenance and discouragement, Newlands' enthusiasm and belief in the final triumph of truth were not quenched. No one enjoying the advantage of his personal friendship would wonder at this. He was of an enthusiastic, warm-hearted, cheerful, but withal sensitive nature, yet he bore no grudges and knew no ill-will. He was a man pre-eminently of large soul and tender heart, as all who really knew him would bear witness. In his earlier days, Newlands had been an energetic supporter of various social reforms in England, and he was one of the British volunteers who fought for Italian liberty under Garibaldi in 1860. Through his mother he had Italian blood in his veins; his father, the Rev. Wm. Newlands, M.A. (Glasgow), was a Presbyterian minister.

With regard to his earlier education, John A. R. Newlands, like C. R. Alder Wright, another great original thinker, received his education primarily and chiefly from his father.

His principal practical work in chemistry had been in connection with sugar refining.—W. S.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Centrifugal Apparatus for Draining Paper Pulp, Sugar, and other Materials, Impts. relating to.* J. Joiner and W. Sommerville, jun., both of Bitton, Gloucestershire. Eng. Pat. 17,972, July 31, 1897.

The apparatus consists of a parallel or tapered cylinder, mounted horizontally upon a hollow, or partly hollow, rotating shaft, and having on its outer surface a single or double helical web communicating with the hollow shaft. The cylinder is enclosed within another similarly-shaped cylinder, open at one end and closed at the other, and having a single or double surface provided with perforations of any sort. The outer cylinder is rotated faster than the inner one, and in either the same or the opposite direction. In use, the paper pulp, &c., is fed in at the open end of the shaft, and will exude through openings in the cylinder, and be carried around the cylinder, being squeezed between it and the outer cylinder. The liquid passes through the perforations in the latter to suitable conduits, while the semi-dry pulp is led by the helices to the open end, and deposited in a truck; or in a stationary chest having suitable beaters and connected with a rotary force pump for conveying the material away as required.—R. S.

*Pharmaceutical and other Powders for the Manufacture of Tabloids, Impts. in Apparatus for Compressing.* P. E. M. Jamain, Dijon, France. Eng. Pat. 23,355, Oct. 11, 1897. (Date claimed under Inter. Conven., Aug. 9, 1897.)

The powder to be formed into tabloids is placed in a hopper to which a horizontal reciprocating motion is given, and thence passes into a cylinder having a number of openings into which the powder is caused to enter by means of an agitator. This cylinder receives a movement of partial rotation by means of a wheel travelling over a fixed rack, as the cylinder is carried backwards and forwards with the hopper. The powder is forced through these openings by means of pistons, into conduits, from which it passes to moulds, a hammer striking the conduits at intervals to prevent the powder adhering thereto. Within the moulds the powder is compressed by punches, and is afterwards detached therefrom by a moving brush, which causes the tabloids to fall into a conduit, and also cleans the punches for the next operation.—R. S.

*Drying, Cooking, Processing, and Treating Organic and other Substances; Impts. in Apparatus for.* A. T. Welch, Baltimore, U.S.A. Eng. Pat. 8069, Apr. 5, 1898.

The articles to be treated, for example bones and fish, are fed to one end of a rotating horizontal cylinder, provided internally with knife-edged shelves, which increase in number from the inlet to the outlet, whereby the material is alternately raised and allowed to fall through a flame, produced by the burning of carburetted air, and passing from end to end through the cylinder. The articles are thus alternately subjected to a high degree of heat when they fall through the flame, and to a low degree of heat when they rest on the cylinder, and are gradually removed from the hottest part of the apparatus, *viz.*, in this case,—the inlet, whilst the knife-edged shelves divide them into continually smaller particles. When the articles are to be processed, sulphuric acid is led from an upper tank through a chamber, and thence by a tube to the rotating cylinder. Claims are made for a special carburettor in which oil is used, for the method of preventing "refrigeration and stratification of carburetting liquid," for the method of drying, the drier, and other details of the complete system and apparatus.—R. S.

*Fabrics from Nitrated Yarns, chiefly designed for Filtering Purposes, A Process for the Manufacture of.* E. Müller, Cologne, Germany. Eng. Pat. 12,867, June 8, 1898.

*See under* V., *page* 756.

## II.—FUEL, GAS, AND LIGHT.

*"Gas Oils," Gasification of.* [*Petroleum.*] H. Eisenlohr. Zeits. angew. Chem. 1898, [24], 549—553.

The author's researches were undertaken to test the assumption that the unfavourable results attending the gasification of mixed oils are due to the necessity of special conditions of gasification for each component. The experiments were made with brown coal-tar oils containing paraffin, and Wernecke's apparatus was employed, the illuminating values being calculated by the aid of Helfer's formula.

In the first series, oils boiling below 360° C. were taken, and here it was found that the illuminating value (L 50) increases concurrently with the percentage of (soft) paraffin present, an oil containing 30 per cent. of paraffin (m.p. 15° C.) giving the value L 50 = 13·47, which receded to 7·28 after the removal of the paraffin, but increased to 14·02 on the subsequent addition of 35 per cent. of paraffin (m.p. 27° C.). Similar results were obtained with other oils with varying boiling points below the prescribed limits, without any necessity arising for altering the temperature of gasification, paraffin, though difficult to gasify when alone, being readily so when dissolved in oil. It was also found that mixed oils (boiling point below 360° C.) gave illuminating values corresponding to the mean of those yielded by the various components of the mixtures, the difference between their temperature of gasification being in no case sufficient to prejudice the result.

With oils boiling above 370° C., which, according to their paraffin-content, should have an illuminating value L 50 = about 14, this value was found to be only 11 to 12½, and as the deficiency was ascertained not to be due to the employment of acid in refining, but to be caused by some constituent of the higher fractions—creosote being thus precluded—the oil was treated, by agitation, with sulphuric acid until of constant weight after the removal of the acid tar, an operation which resulted in the elimination of 37 per cent. of high-molecular unsaturated compounds, from the fraction boiling above 370° C. The illuminating value was thereby increased $2\frac{1}{2}$ points, and as this result was confirmed in the case of other similar oils, the unsaturated compounds present in the higher fractions are thus revealed as the cause of the unsatisfactory behaviour on gasification of mixtures of light and heavy oils.

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* Any of these specifications may be obtained by post by remitting 9*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

The conclusions deduced from these experiments are applicable to the behaviour of gas oils worked on a practical scale, the only objection that could be raised, *viz.*, that the conditions under which the decomposition of the oil vapours is effected might influence the results, being practically devoid of foundation, the results being concordant throughout.—C. S.

*Incandescent Gas Light, Theory of the.* Drossbach. Chem. Zeit. Rep. 1898, **22**, [51], 162. From J. für Gasbeleuchtung, 1898, **41**, 352.

In the author's opinion the light emission from a mantle made from Auer's mixture is due to the influence which the ceria molecule exercises in bringing the heat vibrations of the thoria molecule into the most favourable harmony with the heat waves of the flame, so as to convert these into light waves. He very carefully prepared the purest nitrates of thorium and cerium, and found that the highest illuminating power was obtained from a mantle containing 99·1 per cent. of thoria and 0·9 per cent. of ceria. If the thoria be replaced by zirconia, no useful mantle results, so the author considers that the ceria does not act chemically and catalytically (see Killing's theory, this Journal, 1896, 794). It has been said that as thoria prepared from the nitrate is very bulky, the large surface which it exposes to the flame favours the catalytic action of the ceria, but the author shows that the sulphate, which yields a very compact oxide, gives quite as good results as the nitrate.
—H. B.

*Acetylene, The Occurrence of Ammonia in Commercial.* H. Bamberger. Zeits. f. angew. Chem. 1898, [31], 720—721.

The impurities in acetylene fall into two classes according to their origin, *viz.*: (1) those which result from impurities in the raw materials used in the manufacture of carbide, such as phosphuretted hydrogen and sulphuretted hydrogen, and (2) those produced through the process of fusion in the electrical furnace, such as ammonia and cyanogen compounds. As ammonia is oxidised to oxides of nitrogen when the gas is burnt, much importance attaches to means for preventing its occurrence. When calcium carbide is decomposed with water, ammonia is formed from both nitrides and cyanogen compounds. The nitrides result from the presence of aluminium or magnesium in the lime. In the electric furnace magnesia is reduced in the first instance to magnesium, which is then converted into the nitride through the agency of the nitrogen of the air, which always circulates to a certain extent in the furnace, or of the small quantities of nitrogen which exist in the coal, coke, or charcoal employed. Aluminium is contained as alumina in most samples of lime, and is present in the silicates in coke ash. Alumina is reduced by carbon to the metal or to aluminium carbide. Aluminium carbide always contains nitride, as may be proved by decomposing a sample in the cold with water, and when the evolution of almost odourless gas has ceased, adding excess of caustic soda and warming, whereupon ammonia will be given off in sufficient quantity to be readily detected by the smell.

Carbides, especially barium carbide, possess the power of absorbing nitrogen at high temperatures and producing cyanogen compounds. Cyanogen may be detected by its smell on opening a closed vessel in which hot carbide has been allowed to cool. Ammonia is formed by the action of superheated steam on calcium cyanide, and local superheating often occurs in the usual methods of producing acetylene from carbide. Carbide made from wood-charcoal and lime containing 0·5 per cent. of magnesia and 0·2 per cent. of alumina, was decomposed by dropping water on to samples weighing from 50 to 70 grms. The gas formed, was led through standardised sulphuric acid, and was found to contain from 0·05 to 0·15 per cent. by volume of ammonia. Even the outer crust of the blocks of carbide, and small fragments which had cooled while exposed to the air, did not yield a higher proportion of ammonia.

The method by which the acetylene is generated, affects the proportion of ammonia in the gas. The drip system of generation allows almost the whole of the ammonia to pass into the gas, and the high temperature favours decomposition of aluminium nitride and calcium cyanide. The dip and flooding systems occupy a mean position between the drip system and that in which carbide is dropped into water. The latter system is the best, because the trifling rise in temperature does not suffice to cause decomposition of calcium cyanide and aluminium nitride, and the surplus water in the generator absorbs the greater part of the ammonia. With an apparatus belonging to this system, which has been in continuous use since December 1897, ammonia has scarcely been detected in the gas, but is easily recognised in the water in the generator.—J. A. B.

*Acetylene and Cuprous Chloride, A Crystallised Compound of.* Chavastelon. Comptes Rend. 1898, **126**, 1810—1812.

When hydrochloric acid is poured on moist acetylide of copper the latter is dissolved, and there is no appreciable evolution of gas, provided the mixture be kept cool; but acetylene is evolved under the influence of heat. There is evidently produced a compound of acetylene with cuprous chloride, which is destroyed by heat. This compound may be formed by the action of a stream of pure acetylene on (1) a hydrochloric acid solution of cuprous chloride, or on (2) an aqueous or alcoholic solution of cupric chloride in the presence of copper. The first method gives the finest crystals. They are hexagonal prisms derived from the orthorhombic system. They change on withdrawal from the mother liquor, but if quickly washed with absolute alcohol and anhydrous ether and dried in a stream of dry acetylene, they may be isolated. They decompose without explosion when heated. Their composition agrees with the formula $C_2H_2Cu_2Cl_2$. They decompose, with escape of acetylene and production of a purple precipitate, when they are brought in contact with water or solutions of alkali chlorides. Ammonia converts them into acetylene red.—J. A. B.

*Blast-Furnace Gas, The Use of, for Motive Power.* A. Greiner. Iron and Steel Inst. Spring Meeting, 1898.

*See under X., page 765.*

## PATENTS.

*Electric Furnaces for the Manufacture of Carbides, Impts. in and relating to.* R. Memmo, Rome, Italy. Eng. Pat. 14,022, June 8, 1897.

This electric furnace is provided with a movable piston bottom, which enables either the whole or a part of the calcium carbide formed to be withdrawn without interrupting the working of the furnace. The carbon electrodes, which may be horizontal or inclined, are in the centre of the furnace, and slide hermetically in a cast-iron tube. The regulation of the arc is effected by means of the movement of the bottom as well as by toothed gearing connected to the carbons, and the latter may be arranged so as to obtain star-shaped arcs for use with polyphase alternating currents, or the arrangement may be in the shape of a star or of a ring of small rods connecting the terminals of the same phase so as to use these currents by incandescence. The calcium carbide is cooled in the furnace out of contact with the air to prevent its oxidation, the radiating heat being utilised for heating the air used for the combustion of the water gas formed, which is employed either in the furnace to increase its thermic efficiency, or outside it for heating, lighting, or motive power.—G. H. R.

*Carbide of Calcium, Impts. in the Manufacture of.* E. Tooth, Sydney, New South Wales. Eng. Pat. 8554, April 12, 1898.

Tar and lime, or tar and molasses, or sugar and water and unslaked lime, are mixed into a plastic mass, with or without the addition of coal dust or slack, and are subjected to the action of an electric current in moulds of refractory material, through which pass carbon electrodes which meet, or nearly meet, carbon diaphragms contained within the moulds.
—G. H. R.

*Metals or Metallic Alloys, Carbides; Process for the Extraction of Pure, and the Production of by Electric Heat.* H. Aschermann, Cassel, Germany. Eng. Pat. 7423, March 28, 1898.

*See under* XI. B., *page* 772.

*Furnaces, Regenerative; Impts. in or relating to.* A. J. Boult, London. From F. Wurtenberger, Sestri Ponenti, Italy. Eng. Pat. 14,408, June 14, 1897.

Four air regenerators filled with the usual chequer work, and two gas regenerators are used. All the chambers may be built beneath the furnace, but it is preferred that the gas regenerators should be outside, that they should be of small cross-section, and that, in place of the chequer work, fire-brick arches should be so arranged that the gases pass in zig-zag fashion from end to end. The air regenerators are worked in pairs, so that only one reversing valve is required for them, each flue from the valve to the furnace being divided into two. The current of air or of waste gases passing through each regenerator is controlled by a valve placed in the flue connecting that regenerator with the reversing valve. But the current of waste gases passing through the gas regenerator is separately controlled by a valve placed in the flue between the gas reversing valve and the stack, at a point before the gases from the air reversing valve enter this flue. A separate valve controlling the whole channel is placed between the stack and the point at which the flues from the two reversing valves, converge. The object of the arrangement is to heat the air used for combustion to as high a temperature as possible (to about 1300° C.), whilst the producer gas is only heated to 700° C., whereby the dissociation of this gas in the regenerators commonly observed in working regenerative furnaces, is obviated.—W. G. M.

*Smokeless Furnaces, Impts. in.* P. J. Beimare, Paris. Eng. Pat. 18,950, Aug. 16, 1897.

In front and just above the fire-bars is placed a feed-box, in which the fuel is preliminarily heated and moistened. The feed-box has two doors, one above the other, the lower being provided on its inner face with a removable cast block, through which and through the door is a hole for admitting air and the passage of a poker. Water enters continuously or intermittently through a rose on the top of the feed-box. When the fuel is sufficiently heated in the feed box, the upper door is opened, and it is raked on to the bars behind.—R. S.

*Fuel Gas Producers, Impts. in.* A. Kitson, Philadelphia, U.S.A. Eng. Pat. 21,296, Sept. 16, 1897.

This invention relates to certain improvements in the hearths of fuel gas producers, and in the means for automatically charging the fuel and in keeping it in a continuous and uniform state of agitation. Several arrangements of apparatus are shown, in each of which, however, a combustion chamber, having inwardly inclined walls and a constantly rotating inclined hearth, is employed. The hearth is composed of inclined plates, having openings therein, and its edge is close to the walls of the furnace, and is made to fit tightly therein by means of a number of arc-shaped adjustable pieces, each of which is acted upon continually by the toe of a weighted lever, pivotted beneath the hearth. The hearth is mounted on a central vertical pillar, which rotates on ball bearings, and carries a central blast pipe and a worm wheel for rotating the hearth. The central opening of the grate is covered by a double cap. The air also passes from the central pillar by holes to the space beneath the hearth, and thence upwards through the openings therein. The method of automatically feeding varies with the class of fuel employed. For anthracite and the like, a charging valve and charging bell are employed, and are operated by a loose revolving weight. For softer coal, the charging is done either—(*a*) from a hopper, in the bottom of which is a rotating perforated plate, or (*b*) by the fuel being forced up into the combustion chamber by means of screw conveyors, through passage ways in the combustion chamber walls.—R. S.

*Furnaces or Grates for the Consumption of Mineral Oils or the like as Fuel, Impts. in.* A. Mitchell, Devonshire Street, London. Eng. Pat. 7078, March 23, 1898.

The invention is shown applied to Lancashire, marine, and locomotive boilers. A horizontal plate is placed within the lower part of the boiler flue between the front portion of the fire-bridge and the grate surface, and on it is placed a layer of asbestos fibre or the like. Above and at a slight distance from the latter is a perforated plate, supporting several layers of angularly perforated balls or blocks of asbestos or fireclay. These layers may be separated by plates or rods. The oil is admitted from a slightly raised water-jacketted and felt-covered tank by tubes which project through the furnace bottom into the asbestos fibre, and are provided with rose nozzles. Air is admitted through horizontal tubes passing into and between the balls or blocks. These tubes are open on their outer ends, and closed on their inner ends, but near the latter they are provided with numerous holes or perforations. The oil-supply pipes are provided with suitable taps, and the flame is lit through a small door or opening.—R. S.

*Electrical Furnaces [excluding Air, &c.], Impts. in.* Siemens Bros, and Co., Ltd., London. From Siemens and Halske Aktien-Gesellschaft, Berlin. Eng. Pat. 11,438, May 19, 1898.

Objections are set out as against the admission of air to the electric arc through the layer of material to be treated. It is proposed to obviate the defects "by employing a layer of the pulverulent material to be treated of such a depth that neither the external air can penetrate through the same from the outside, nor can the gas produced by the electric arc penetrate through the same from the inside, and by employing, as the upper movable carbon electrode, a carbon tube extending up through the layer of material, so that the gases produced escape through the same without entering into combustion. The carbon tube is provided at its upper end with an iron cover having several openings, to which are connected flexible metal tubes, at the ends of which the gases escaping through them enter into combustion with the outer air, the tubes being made of such a size that the flames cannot strike back through them, even when, for purpose of tapping the furnace, air penetrate into the carbon tube from below." Reference is made to Eng. Pat. 21,879 of 1894 (this Journal, 1895, 1019). An electrical furnace of this description is claimed, and there is further claimed . . "the combination with the carbon tube of a receiver or condensing chamber for condensing the vapours or fumes produced by the electric arc, which chamber is embedded in the charge of material to be treated"; and "An electrical furnace having a carbon tube as the upper moveable electrode arranged for distilling [zinc] substances and condensing the products and operating as described with reference to . . . the drawings."
—J. C. R.

*Gases by Washing or Scrubbing, Impts. in Apparatus for Purifying.* H. H. Lake, Southampton Buildings. From Fellner and Ziegler, Bockenheim, Germany. Eng. Pat. 17,641, July 27, 1897.

The washer or scrubber is provided with a series of cross vertical partitions of gradually decreasing length from the gas inlet at one end to the outlet at the other. From the bottom of each partition is a horizontal perforated plate, with a short subsidiary vertical partition secured to its other edge. When the gas is turned on, the pressure thereof causes the liquid on each side of the partitions to stand at a different height, so that a step-like arrangement results. Any dust in the gas is stopped, and falls to the bottom of the vessel. An overflow arrangement is used at the exit end. Should the pressure fall, the washing liquid cannot be drawn back; the only result is that the step-like arrangement of water levels is reversed.—R. S.

*Gas, and the Obtainment of By-Products therefrom; Impts. in and Apparatus for the Manufacture of.* E. Gobbe, Jumet, Belgium. Eng. Pat. 17,976, July 31, 1897.

What the patentee claims is as follows:—"In the manufacture of gas and production of by-products, the separation of the rich from the poor gas so as to eliminate the by-products without the necessity of washing the whole of the gas generated."

"The injection of steam" for producing ammonia with the nitrogen of the fuel.

Finally, the various forms and apparatus described and illustrated.—R. S.

### *Lighting Oils, An Improved Method of and Compound for Treating.* J. McKee, Belfast. Eng. Pat. 20,082, Sept. 1, 1897.

To increase the luminosity of the flame obtained, to destroy the smell and smoke, and to neutralise the explosive properties of the oil, the latter is treated with a mixture of the following materials, which may be in the following proportions:—Powdered naphthalene, 1 cwt.; common salt, 7 lb.; and powdered camphor, 7 lb.—R. S.

### *Lamp Black and other Carbon of Great Density, An Improved Method of and Means for the Manufacture of.* G. Wegelin, Rondorf, Germany. Eng. Pat. 12,184, May 28, 1898.

The lamp black is made by burning any suitable combustible material in the usual way, the improvement consisting in producing a uniform pressure on all parts of the flame directly above the level of the base thereof. This is done by means of air, compressed or not, admitted through annular openings. By this means all parts of the flame are driven either to the centre, or against the external wall, according to whether the air is admitted outside or inside the flame, the resulting lamp black being thereby rendered much denser than usual.—R. S.

### *Burners for Petroleum and other Hydrocarbons, Impts. in or relating to.* A. J. Boult, London. From G. Barthel, Dresden. Eng. Pat. 10,911, May 12, 1898.

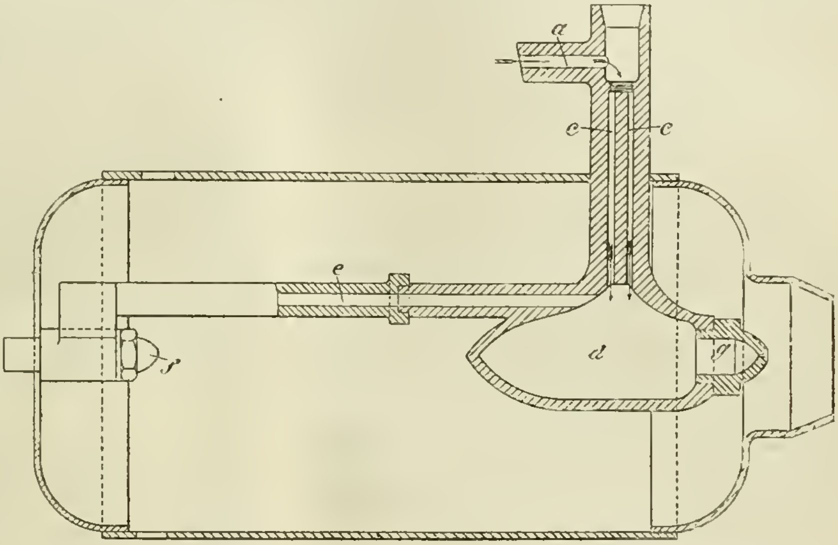
The improved burner, which is secured on to a reservoir containing the hydrocarbon, consists of an upright supply pipe provided with the usual collar-like "starting cup." The pipe terminates in a small gasifying chamber within a solid metallic body, shaped like an inverted cone and perforated with lateral air passages, and the vaporised hydrocarbon which blows upwards from an orifice in the gasifying chamber mingles with the air and enters the burner top. The latter is a double-walled mushroom cap, at the lower edge of which the combustible mixture issues circumferentially and burns with a ring-shaped flame which is directed upwards by a flange which extends up from the conical body. The flange conducts sufficient heat to the latter to effectively heat the gasifying chamber. The flange may be cast in one with the conical body or it may be detachable. The complete burner is claimed.—H. B.

### *Bunsen Burners for Petroleum and other Hydrocarbons, Impts. in or relating to.* A. J. Boult, London. From G. Barthel, Dresden. Eng. Pat. 10,912, May 12, 1898.

The petroleum rises in two diverging limbs of a bifurcated pipe, and the vapours produced pass up into two gasifying chambers, from which they pass down two inclined converging pipes which meet in a small chamber provided with an outlet orifice. The small chamber is closed by a screw-cap, so that the pipes may be readily cleaned from lamp-black when the cap is removed. The gas burns in a burner-top similar to that described in Eng. Pat. 10,911 of 1898 (see preceding abstract). In an alternative construction the gasifying chambers and the capped chamber are omitted, the converging pipes and the upper parts of the diverging pipes being cast in one, and screw-caps placed at the junctions of the two sets of pipes facilitate the removal of lamp-black. The three claims are for burners characterised by the two arrangements of pipes and the caps for facilitating the cleaning of the pipes.—H. B.

### *Vapour Burners, Impts. in and connected with.* L. Durr, Bremen. Eng. Pat. 25,955, Nov. 8, 1897.

A development of patentee's Eng. Pat. 12,771 of 1893. The illustration shows the simplest form of the present invention. There is claimed an oil-vapour burner provided with two co-operating burners *f* and *g*, and wherein the gasification of the petroleum is effected in a chamber *d* of ellipsoidal or other suitable form, over the outside of which the flame from the rearward burner *f* spreads, whilst the uninterrupted flame from *g* is used for heating or illuminating as required. The chamber *d* is first heated by a burning wad of, say, cotton-waste, and then a regulated supply of petroleum is admitted from *a* through the perforations *c*. The second claim is for dividing the chamber *d* into two compartments, one for each burner, each having its own oil supply, so that if the supply to *g* is temporarily stopped, *f* will continue to heat the vaporiser, and the burner *g* will at once enter into action when oil is again admitted to it. The third and fourth claims are for a burner arranged upon a wheeled carriage, and adapted for heating purposes, the figures illustrating an apparatus connected to a steam-boiler furnace by means of a cone which is provided with circumferential apertures to admit a supply of air, the air-supply being controlled by a slotted valve.—H. B.



### *Incandescence Gas Lighting, Impts. in.* E. Salzenberg, Crefeld, Germany. Eng. Pat. 20,319, Sept. 3, 1897.

In order to use gas under pressure of three atmospheres or upwards for incandescence lighting, the patentee proposes to prepare the mantle so that it will not be broken up or blown away. Several mantles in the unburned state are drawn over one another, and incombustible threads of asbestos, platinum, or the like are passed through them. After burning off, "the threads are so secured around the outermost layer of incandescence material, as well as secured to the burner or burner support, that the incandescence body is enclosed with a netting of incombustible threads or wires, and cannot be blown off or destroyed by means of the high gas pressure." The claims relate to mantles so prepared, and there is also claimed "the improved method for the production of an incandescence gas light, characterised by the supply of the gas to the incandescence body at a pressure at which a light of golden yellow colour is caused."—H. B.

*Incandescence Oil Lamps, Impts. in Burners for.* G. Kron, Copenhagen. Eng. Pat. 21,413, Sept. 14, 1897.

THE lamp is of the usual circular-wick type: the wick tubes are surrounded by a conical cap having at the top an opening, between the edge of which and the edge of a spreader is left a free, narrow, and annular space, placed almost over the upper edge of the wick. The mantle surmounts the whole as usual. In this invention there is slipped over the outer wick tube (but within the abovementioned conical cap) a cylindrical cover, the upper edge of which is bent over and rests upon the top of the wick. The cover may simply rest on the wick and be free to move up and down with it, or it may rest on an inner projection of the conical cap, so that the wick must be raised a little before it is in contact with the edge. The claim is for the burner fitted with this cover, and it is stated that the smoking of the flame, caused by uneven edges of the wick, is thus avoided, since the gas is only developed on the inner side of the wick.—H. B.

*Incandescent Vapour Lamps, Impts. in.* G. F. Redfern, London. From V. Posno and L. Morrison, Antwerp. Eng. Pat. 10,792, May 11, 1898.

THERE is claimed a lamp in which an auxiliary flame heats the tube containing the main wick, the vapour produced from the oil sucked up by the latter being burned in a Bunsen burner within a mantle as usual. The auxiliary burner (which is specially claimed) is of the Bunsen type, and must therefore itself receive a preliminary heating to start the evolution of vapours. For this purpose there is provided a device consisting of a cylinder within the reservoir, the screw cap of which carries a wire and plug which dips into the petroleum. The cap is unscrewed and removed with the plug; the latter is ignited, and is then used to give the preliminary heating to the auxiliary burner.—H. B.

*Acetylene Generators, Impts. in or connected with.* J. G. A. Kitchen, Manchester. Eng. Pat. 17,793, July 29, 1897.

A CARBIDE container has its base composed of a porous material which may be compressed by a gland in order to regulate the rate at which water contained in an exterior vessel may pass through the material to the carbide. In order to stop the action entirely, the bottom of the container may be screwed or otherwise pressed against a washer in the bottom of the exterior vessel. In another type of the apparatus, action is stopped by the pressure of the gas within the carbide container forcing the water back from the porous material, while the gas accumulates in a bell at the bottom of the exterior vessel.—J. A. B.

*Acetylene Gas, Impts. in and relating to the Manufacture of.* J. C. Bull, Erith. From Josse and Defays, Lille, France. Eng. Pat. 18,355, Aug. 6, 1897.

THE supply of water to carbide contained on superimposed trays in generating vessels is controlled by a spring which holds a cup containing mercury below the end of a pipe leading from a cistern, and thus seals the pipe. The seal is broken and water allowed to pass from the cistern to a generator by extension of the spring, which is brought about by the fall of the bell of the gasholder which receives the gas from the generators.—J. A. B.

*Acetylene Gas from Calcium Carbide, A Method of Manufacturing.* J. R. Wigham, Dublin. Eng. Pat. 18,971, Aug. 17, 1897.

A cylinder having a lid sealed in a mercury lute has divisions forming chambers for carbide. Water is brought on to the carbide from a cylinder of precisely the same capacity placed above the carbide cylinder. Taps, valves, washers, purifiers, and a gasholder of ordinary construction are provided.—J. A. B.

*Acetylene Gas, Impts. in and connected with Apparatus for the Manufacture of.* O. Munsterberg, Berlin. Eng. Pat 19,615, Aug. 25, 1897.

A VALVE on a pipe by which water can pass to a carbide holder is opened by a projection which descends with the bell of the gasholder; and when the first carbide holder is exhausted, a second projection operates on the valve on a pipe leading to a second carbide holder. A device for automatically cutting off communication between the gasholder and a carbide holder when opened for recharging, and automatically restoring communication when the carbide holder is closed again, is described. For use with generators in which carbide is fed into water, an endless band for delivering the carbide from a hopper to the generator is driven by a separate motor or by transmission gearing from the bell of the gasholder; or, by another device, carbide is delivered from chambers in a rotating drum into the water. A partition separating the shoot from the chamber in which the greater part of the gas is generated in apparatus in which carbide is fed into water, is also described.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Automatic Supply of Carbide of Calcium in the Production of.* F. A. Kieffer, Paris. Eng. Pat. 20,112, Sept. 1, 1897 (under Internat. Convention).

THE carbide is contained in a hopper attached to a bell floating in a tank with hopper-shaped base, from which the residue is removed through a cock. The bottom of the hopper has an outlet which is closed by a flap- or ball-valve operated through a rod by a float which is supported by the water in the tank when the bell descends below a certain level. When the bell is raised, the float being no longer supported by the water, keeps the valve pressed on its seat, and prevents carbide passing from the hopper into the water. —J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* H. de Thiersant and W. A. Coulson, both of London. Eng. Pat. 1584, Jan. 20, 1898.

CARBIDE in a suitable cage is surrounded by a perforated spiral tube, up which water passes from a cavity which communicates with an outer casing, into which the water is passed in the first instance by inverting the apparatus and opening a cock which communicates with a casing which has previously been filled with water. An exit pipe and safety tube are provided.—J. A. B.

*Acetylene Gas Generators, Impts. in.* A. Williamson, Greenock, Renfrewshire. Eng. Pat. 9545, April 26, 1898.

A VESSEL is divided into three superimposed compartments, the lowest of which contains carbide chambers which are fed with water from the middle compartment, into which the gas generated in the lowest compartment passes, and expels water through a sealed tube into the top compartment. The valves on the tubes by which water passes from the middle to the lowest compartment are opened when the lids of the carbide chambers are screwed home after the chambers are filled; the flow of water then regulates itself according to the rate at which gas is withdrawn for consumption from the upper part of the middle compartment.—J. A. B.

*Acetylene Gas Generator, Improved.* J. H. Schülke, Berlin. Eng. Pat. 10,305, May 5, 1898.

THE delivery pipes of a number of generators are sealed in one receptacle containing liquid, which may serve for washing the gas. The inlets to the generators may be at different heights and the delivery pipes sealed to different depths, so that the generators come into operation in turn automatically. Two or more liquid traps for the gas may be used in order to wash or purify it thoroughly.—J. A. B.

*Calcium Carbide, Treating, for use in Acetylene Generators.* J. Sartig, Cassel, Germany. Eng. Pat. 10,763, May 11, 1898.

In order to facilitate the fall of calcium carbide from hoppers into water, it is comminuted with 3 to 6 per cent. of talc, lycopodium or other material, which will act as a lubricant, but is unaffected by water or the gas.—J. A. B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Benzene, Absorption of, by Means of Water and Paraffin Oil.* E. Müller. J. für Gasbeleuchtung, **41**, [27], 433—435.

*Absorption of Benzene by Means of Water at Different Temperatures.*—This question is of importance, inasmuch as the gaseous products from gas works and coke ovens are passed through water in order to remove ammonia, an operation, which in the case of coal gas gives rise to a diminution of candle power, and in the coking of coal to a decrease in the yield of benzene. The apparatus used by the author for determining the absorption of benzene by water consisted of a 150 c.c. flask, filled with 100 c.c. of freshly boiled distilled water. The tube admitting the air charged with benzene, reached to the bottom of the flask, the aqueous vapour discharged from the latter being absorbed in two tubes filled with calcium chloride. The tubes and flask were weighed before and after each operation, the increase in weight being due to benzene absorbed. The results, which are given in the following table, show that benzene vapour is not taken up by water in direct proportion to its pressure, as at 15°, with air containing 7·59 per cent. of benzene, the proportion of water to the volumes of benzene absorbed was as 100 is to 51, whilst with air containing only 1·55 per cent., the ratio was as 100 to 23. Benzene vapours do not behave like a so-called "permanent gas," in accordance with Henry's law, and the solubility in water is thus not in direct proportion to the pressure. In a mixture of air and benzene vapour, the less the proportion of the latter present, the greater is that of the volume dissolved out. Although in practice this loss in dissolved benzene may be disregarded, it is nevertheless noticeable when an excessive quantity of water is used for scrubbing purposes, in consequence of the great value of benzene as a carburetting agent.

| Temperature. | Percentage of Benzene in the Air used. | Absorption of Benzene in 100 c.c. Water. | Calculated into per Cent. on the Benzene used. |
|---|---|---|---|
| ° C. | By Vol. | Grms. | |
| 16 | 8·17 | 0·143 | 2·83 |
| 16 | 7·69 | 0·145 | 3·10 |
| 15 | 7·59 | 0·167 | 3·58 |
| 15 | 6·88 | 0·157 | 3·74 |
| 15 | 6·66 | 0·138 | 3·34 |
| 10 | 5·90 | 0·190 | 4·89 |
| 10 | 5·82 | 0·174 | 4·64 |
| 15 | 4·30 | 0·118 | 4·47 |
| 10 | 3·04 | 0·087 | 4·69 |
| 15 | 1·55 | 0·076 | 8·02 |
| 18 | 1·60 | 0·061 | 8·73 |
| 17 | 1·01 | 0·059 | 9·72 |
| 16 | 0·99 | 0·072 | 8·63 |
| 18 | 0·94 | 0·045 | 8·00 |

*Absorption of Benzene in Paraffin Oil.*—The paraffin used was the "Paraffinum liquidum" of the German Pharmocopœia. It has a specific gravity of 0·880—0·890 at 15°, boils at about 360°, does not separate any solids when cooled to a temperature of 0° to −2°, or experience any loss or gain in weight when brought into contact with air. The apparatus consisted of a flask representing the volume of air used for each experiment. From this the air was made to pass through a safety bottle, two cylinders containing well-dried calcium chloride, and a carburetting bottle charged with benzene. The percentage of the latter was regulated by a device inserted between the last cylinder and the carburetting bottle, which made it possible to put the carburetting bottle wholly or partially out of use. The air charged more or less with benzene was then introduced into the absorption vessels, which were filled with paraffin oil and kept cold by immersion in ice. After the completion of each trial the absorption vessels were closed and not weighed until they had assumed the temperature of the laboratory. The air leaving the last absorption vessel was tested for benzene by passing it through a nitrifying mixture, the odour of nitrobenzene rendering it possible to detect the presence of 0·4 c.c. of benzene vapour in 10 litres of air. The duration of the trials, although different, had no marked effect on the results; the absorption in the separate vessels, however, varied with the percentage of benzene contained in the air employed; thus with air containing 3 to 8 per cent. (by vol.), about 90 per cent. of the total quantity of benzene was absorbed in the first bottle, 9 per cent. in the second, 1 per cent. in the third, and 0·1 per cent. in the fourth; whilst with air containing only about 1 per cent. of benzene, 40 per cent. was found in the first bottle, 30 per cent. in the second, 20 per cent. in the third, and 10 per cent. in the fourth. It was also found by experiment that efficient cooling greatly influences the solvent action of paraffin oil for benzene; thus the loss which, at a temperature of 17° amounted to 13·36 per cent., was reduced to 6 per cent. when the oil was cooled in a mixture of ice and salt.

This method has been used for some time for the estimation of benzene vapour in the gas from coke ovens.—D. B.

*Coking in By-Product Ovens.* J. H. Darby. J. Iron and Steel Inst., May 1898.

It is stated that though well-made beehive coke leaves little to be desired as regards economy in the blast furnace, data have been secured, which go to show that well-made retort coke gives equally good results; moreover, there are fuels, which cannot be coked satisfactorily in beehive ovens, which nevertheless produce a merchantable coke in retort ovens. There has, no doubt, been much prejudice against the use of retort-coke, for which the author considers there is little ground. It is, for instance, said to contain more water than coke produced in the beehive oven, owing to quenching outside the oven. Contracts for retort coke have, however, been carried out in more cases than one, where it was undertaken that the water and ash together should not exceed 12 per cent.. and seeing that 10 per cent. of ash is a fair average for good coke, very little room is left for an excessive quantity of water. Retort coke is not so bright in appearance as beehive, and it is different in shape. Whatever the effect of this dissimilarity may be, the fact remains that with the increased yield of coke per ton of fuel and the substantial return in by-products, the comparison is highly in favour of retort coke, which fetches the same price as beehive coke made from similar fuel.

Taking coke at 12*s.* per ton, tar at 17*s.* per ton, ammonium sulphate at 8*l.* 15*s.* per ton, and light oil (containing 72 per cent. of benzols) at 7½*d.* per gallon, the respective gross values obtained from 100 tons of suitable dry fuel, containing 30 per cent. of volatile matter, would be as follows:—

| | £ | *s.* | *d.* |
|---|---|---|---|
| Coke, 70 per cent. at 12*s.* per ton .................... | 42 | 0 | 0 |
| Tar, 4·75 per cent of fuel, and at 17*s.* per ton....... | 4 | 0 | 9 |
| Sulphate, 1·25 per cent. of fuel, and at 8*l.* 15*s.* per ton | 10 | 18 | 9 |
| Light oil, 3·3 gallons per ton, and at 7½*d.* per gall... | 10 | 6 | 3 |
| | £67 | 5 | 9 |

Deducting the cost of materials and manufacture the net values are:—

| | £ | *s.* | *d.* | £ | *s.* | *d.* |
|---|---|---|---|---|---|---|
| Coke, 70 tons at 9*s.* 3*d.* (11*s.* less 1*s.* 9*d.* per ton for labour, stores, &c.) less 100 tons washed and ground fuel at 4*s.* 9*d.*... | 8 | 12 | 6 | | | |
| Steam, 1·25 lb. per lb. of coke made, equal to 88 tons water evaporated, say, by 13 tons of fuel at 3*s.* 6*d.* ................. | 2 | 5 | 6 | | | |
| | | | | 10 | 18 | 0 |
| Tar, 4·75 tons at 17*s.*...................... | 4 | 0 | 9 | | | |
| Sulphate of ammonia, 1·25 tons at 5*l.* 15*s.* (8*l.* 15*s.* less 3*l.* for acid, labour, steam, stores, &c.) ............................ | 7 | 3 | 9 | | | |
| Light oil, 3·3 gallons per ton at 6*d.* (7½*d.* less 1½*d.* for labour, oil, stores, &c.)..... | 8 | 5 | 0 | | | |
| | | | | 19 | 9 | 6 |
| | | | | £30 | 7 | 6 |

"Note.—Nothing has been allowed for establishment charges or for depreciation. The finished benzenes have been calculated in the terms of light oil for sake of convenience and the figures are from actual practice."

Of the net profits obtained, coke and steam yield about 36 per cent. and the by-products 64 per cent. The cost of a beehive oven to produce 7 tons of coke per week will be about 60*l*. A retort oven to produce 25 tons of coke per week with full complement of chemical plant to recover the products, including 90 and 50 per cent. benzols and solvent naphtha, will cost about 800*l*. when 50 are erected. Hence to make the same quantity of coke as the retort oven there are required: —

| | £ | *s.* | *d.* | £ | *s.* | *d.* |
|---|---|---|---|---|---|---|
| 3½ beehive ovens, costing.................. | | | | 244 | 0 | 0 |
| Add proportion of boilers and chimney (90 ovens require at least two large Lancashire boilers) :— | | | | | | |
| Boilers......... | 630 | 0 | 0 | | | |
| Setting boilers.. | 100 | 0 | 0 | | | |
| Chimney....... | 250 | 0 | 0 | | | |
| | £980 | 0 | 0 | | | |
| —— × 3½....... | 90 | | | 10 | 0 | 0 |
| | | | | £254 | 0 | 0 |

The cost of the retort ovens and chemical plant per oven over beehive is therefore 546*l*. The net revenue per oven per annum in by-products at present prices is 35 tons of fuel per week × 50 weeks = 1750 tons, at 19*l*. 9*s*. 6*d*. per 100 tons as above, 340*l*. 16*s*. 3*d*. Most modern retort ovens represent long, narrow, horizontal chambers, in some cases 33 ft. long and about 5 ft. 6 ins. high. The width is varied to suit the fuel under treatment. As a general rule, highly bituminous coals require a wide oven, and, consequently, comparatively slow coking, to obtain the best results. Coals low in volatile matter require quick coking so as to produce a hard coke. As the retort oven furnishes its own gas for the coking of the fuel, it is essential to ensure continuous working of the ovens to provide a regular source of heat, and they should be kept at work continuously day and night throughout the week if the best results are to be obtained. It is of very great importance to introduce the fuel into an oven in the proper condition. The almost universal practice of washing coal for coking introduces a large percentage of water, and even if the fuel is allowed to drain, the percentage of moisture will often be 11 or 12 with very fine fuel, and unless adequate steps are taken to remove the surplus water, it is troublesome in many ways. The author has determined the heat carried away by water in fuel containing 4 per cent. of combined water and 4, 8, and 12 per cent. of added water, the latter being a common percentage. The following are the results compared with the heat carried away by the gas itself:—

| —— | I. | II. | III. | IV. | — |
|---|---|---|---|---|---|
| Water vapour........ | 2,844 | 5,688 | 8,532 | 11,376 | Calories. |
| Gas.................... | 4,166 | 4,166 | 4,166 | 4,166 | „ |

It will be seen that the heat in the water vapour in No. IV. is 2·7 times as much as the heat carried away by the gas. This is a source of very great loss. There is the heat required as shown to drive off the surplus water as steam, the largely increased condensing power to condense the water vapour in the gas from the ovens, the deterioration of the lining of the ovens, slower working, that is a smaller output of coke per oven, and the dilution of the ammonia liquor by the surplut water.

The points to be observed in the construction of a retort oven are, (1) that all parts should as far as possible be capable of examination: (2) that it should be possible to observe the heating flues on either side, so that the regulation of gas and air supplies can be properly controlled; and (3) the highly-heated lining should be independent of the main structure, so that it may be renewed when necessary, and not be used for carrying the upper portion of the oven. The efficiency of a recovery oven depends on the fact that all the gas liberated during the coking of the fuel is drawn off and treated for tar, benzene, ammonia, &c., before it is returned to burn in the heating flues of the ovens. With regard to the recovery of by-products, the question arises as to what the effect of a general adoption of retort ovens would be on prices. The demand for tar and its principal constituent, pitch, is likely to increase very considerably. The production of ammonium sulphate in the United Kingdom for the years 1896 and 1897 was:—

| —— | 1896. | 1897. |
|---|---|---|
| | Tons. | Tons. |
| Gas works............................. | 127,498 | 132,724 |
| Iron works............................ | 16,511 | 17,779 |
| Shale works........................... | 37,822 | 37,153 |
| Coke ovens, producers, and carbonising works................................. | 9,078 | 10,624 |
| Total........................ | 190,909 | 198,280 |

When sulphate was about 7*l*. per ton, early in 1897, many of the sodium nitrate mines in Chili were closed owing to the consequent low prices of nitrate. The production of nitrate amounted in 1897 to 1,020,000 tons; there is, therefore, room, with the margin of profit shown, to extend the trade in sulphate to almost an unlimited amount and replace nitrate. With regard to benzene, its qualities as an enriching agent for illuminating gas are so great that one volume is equivalent to nearly 2½ volumes of light petroleum spirit, so that a gasmaker could afford to pay nearly 2½ times the price of petroleum spirit for 90 per cent. benzene for enriching purposes, in which case the consumption would be almost unlimited.

Steam must also be considered as a by-product. The heated gases leaving the ovens are conducted into a main flue, and before they pass into the chimney their temperature is reduced by passing them through flues of boilers arranged in series with the chimney. It may be that a large portion of this steam is employed in working the machinery necessary for the retort ovens, such as exhausters, stills, pumps, &c., but in determining the value of the by-products the cost of steam has been allowed for.—D. B.

*Ketopentamethylene, Occurrence of, in Oil of Wood-Tar.* H. Metzner and D. Vorlander. Ber. **31**, [12], 1885-1886.

According to Vorlander and Hobohm (this Journal, 1896, 682), ketopentamethylene can be readily detected in wood-tar oils by its condensation with aldehydes. Its absence in most oils examined by the authors, is attributed to the circumstance that in subjecting the crude oils to rectification, the gaseous products are almost invariably passed through milk of lime, which retains all ketones.

The authors have obtained ketopentamethylene by the destructive distillation of calcium succinate, the fraction boiling at 100°—160° yielding dibenzalketopentamethylene when shaken with benzaldehyde and caustic soda. By cooling the gases leaving the receiver in a long upright condenser and subsequently passing them through a series of receptacles surrounded by a freezing mixture, a colourless mobile liquid is obtained boiling at 20°—40°. Its vapour was found to impart a green colour to a pine shaving moistened with hydrochloric acid, a reaction pointing to the presence of furfurane (furane) $C_4H_4O$, and its homologues.—D. B.

*Oil Field of Texas, The Recently Developed.* T. D. Miller. Eng. and Mining J. 1898, **65**, [25], 734.

Petroleum was discovered at Corsicana, Texas, prior to 1894 by A. Beaton, but no attempt was made to collect it until that year, when wells were sunk, varying in depth from 1,000 ft. to 1,040 ft. to the top of the oil-bearing sand, which is from 10 to 40 ft. thick. The oil rock is soft grey shale; it lies very regularly and dips to the south-east about 1 ft. in 100. At the present time there are over 100 producing wells, some of them not more than 200 ft. apart, and there is still no appreciable diminution in the flow of the individual wells. Of the total number of wells drilled, not quite 10 per cent. have proved non-producers or dry holes. A regular system of drilling is being carried out for the purpose of determining the exact extent of the oil field, and, in the course of these operations, an extensive gas field has been discovered. At one place where a flow of natural gas was discovered at a depth of 1,040 ft., the static pressure of the gas was over 200 lb. Up to the present all of the oil obtained has been used for fuel, except a small portion, which has been used for gas-making. For the latter purpose, the oil gives very satisfactory results, yielding from 5·50 to 5·75 candles per gallon of oil used, whilst as high as 32 C.P. gas can be made with it.

A sample of the crude oil gave the following results when examined by E. H. English:—The oil was very dark brown and opaque, but thin and fluid at 60° F.; sp. gr. at 60° F., 0·8292. On fractional distillation the oil yielded:—

| Temperature. | Per Cent. by Volume. | Specific Gravity. |
|---|---|---|
| 130°—200° F. ........ | 2·80 | 0·6653 |
| 200°—300° F. ........ | 12·70 | 0·7191 |
| 300°—400° F. ........ | 17·60 | 0·7680 |
| 400°—500° F. ........ | 15·70 | 0·8012 |
| 500°—600° F. ........ | 14·20 | 0·8345 |
| 600°—665° F. ........ | 32·20 | 0·8627 |
| Above 665° F. ...... | 2·70 | 0·9350 |

—A. S.

### *Bitumens, Californian; The Technology of.* S. F. Peckham. J. Franklin Inst. **146**, [1], 45—54.

The author records in this paper the work done in connection with the chemistry and technology of Californian bitumens since 1865. From personal experience in 1866, he concluded that California oil could not, when refined, compete with Pennsylvania oil in illuminating power, an opinion confirmed more recently in an attempt to produce a first-class burning oil from the former, regardless of cost. Very good lubricating oils are, however, obtainable, the products of the green, brown, and black crude oils standing, in point of quality, in the order given.

On extracting the nitrogenous basic oils—discovered in California oil by Salathé—by the aid of sulphuric acid, and subjecting them to distillation, pyridine and quinoline were detected. These oils dissolved readily in acid, their hydrates being then precipitable, by sodium hydroxide, as cream coloured flakes, which on exposure to the air turned brown and gradually evaporated completely, at the ordinary atmospheric temperature. The acid tar obtained in refining the lubricating oils was soluble in alkaline solutions, and that from the green oil was brown and more nearly fluid than that from the black oil.

The author found the so-called "crude paraffin" from Torrey cañon to be an unsaturated hydrocarbon, presumably a solid olefine or naphthene, and he agrees with Mabery (this Journal, 1898, 38), in ascribing the main constituents of California oil to the $C_nH_{2n}$ series, his personal experiments tending to demonstrate the presence of a large proportion of benzene and its homologues—a circumstance explaining the difficulties in the way of preparing a good illuminant from these oils.

He attributes the polymerisation of the oils and the rapidity of their conversion into "maltha" and asphaltum, largely to their content of unstable compounds of nitrogen and sulphur.—C. S.

### *Petroleum from Kleczanys, Galicia, The Heat of Vaporisation of Fractions of.* V. Syniewski. Zeits. angew. Chem. 1898, [27], 621.

The author has submitted the petroleum, obtained from Kleczanys in Galicia, to fractional distillation, and has determined the specific heat and, by a specially devised apparatus, the heat of vaporisation of each of the fractions, which distil at intervals of 20° C. between the limits of 110° C. and 250° C. separately. Above 250° C. determinations could not be made with accuracy, owing to experimental difficulties, arising out of the high temperature required.

The author corroborates, in the case of the Kleczanys petroleum, the results, obtained by Pawlewski [Kosmos, 1893, **6**] with other petroleums, that the specific heat diminishes as the boiling-point of the fractions rises, though the actual values for the specific heats of the Kleczanys fractions are somewhat higher than those found by Pawlewski for the other petroleum distillates.

The heat of vaporisation for the fraction boiling between 110° C. and 130° C. is 63·54 calories, and decreases to a minimum of 60·00 calories, for the fraction boiling between 170° C. and 190° C. Above 190° C. it increases with the boiling-point to a value of 62·56 calories for the fraction boiling between 230° C. and 250° C.

It is suggested that this increase at high temperatures is due to the partial breaking down of the heavier hydrocarbons into lighter ones, which have a greater heat of vaporisation.—J. F. B.

### *Petroleum Spirit from Baku, Detection of Di-isopropyl in.* O. Aschan. Berichte, **31**, [11], 1801.

*See under* XXIII., *page* 798.

### *Naphtha (Petroleum) Acids, Some Properties of the Salts of the, and Qualitative Distinction of the latter from Salts of the Fatty Acids.* K. Charitschkow. J. russ. phys.-chem. Ges. **29**, 691—696; Chem. Centr. 1898, **1**, [23], 1191.

According to Holde's experiments, naphtha acids are formed from neutral petroleum by treating it with air in presence of alkali. Lissenko and Zalosetzky have shown that small quantities are formed by distillation of the petroleum. The author separated out from the crude distillate a liquid, which, after purifying by re-distilling several times, was obtained colourless and nearly odourless, had a specific gravity of 0·961 at 20° C., and distilled undecomposed at 100 mm. Notwithstanding the constitution of the liquid ($C_nH_{2n-2}O_2$), it absorbs neither the halogens nor the hydrogen compounds of the halogens. It dissolves in concentrated sulphuric acid with a slight development of heat, and on long standing is resinified; by an immediate addition of water it is separated out unchanged. It readily absorbs sulphur, especially on warming, but the latter is easily set free again by alkalis and alcohol.

The salts of the naphtha acids are all amorphous, with the exception of the calcium compound, which may be obtained as a fibrous mass. The alkali and silver salts are insoluble in hydrocarbons; the former are soluble in water, the latter slightly soluble. The salts of the earth metals are slightly soluble in water and insoluble in hydrocarbons, whilst the salts of the heavy metals and of aluminium are slightly soluble in water and soluble in hydrocarbons. With regard to the latter solvent, the iron and copper salts are most soluble, the zinc salt in the proportion 5 : 100. It is very difficult to remove the last traces of petroleum spirit, ligroin, &c., from the salts which have been dissolved in them, there being probably formed an intermediate molecular compound between salt and hydrocarbon. The solubility of the copper salt in petroleum spirit, together with the characteristic green coloration of the solution, may be used as a very sensitive reaction for naphtha acids and their salts, which are often used for adulterating moderate kinds of soap. It is stated that the copper salts of naphtha acids are effective disinfectants, and it has been proposed to use them for the impregnation of wood.—A. S.

### *Mineral Oils, Experiments on the Utilisation of the Waste Acid from the Purification of, and on the Recovery of Soda from Sodium Phenolates.* W. Heinrici. Zeits angew. Chem. 1898, [23], 525—526.

The dirty acid ("Vitriol-tar") from the washing of tar and mineral oils has always been one of the most troublesome waste products to dispose of, and although a number of processes for dealing with it have been suggested, none of them seem to have found general adoption. The method recommended by the author is based on the disposal of the waste acid by working it up into sulphur. For this purpose the vitriol tar is heated in a still with one-third of its weight of coke, whereby the acid is decomposed, sulphurous and carbonic anhydrides and water being formed. Having removed the water in great part, the extraction of empyreumatic substances is effected by passing the gaseous mixture through a scrubber containing heavy paraffin oil. The gas is then made to traverse a hot porcelain or stoneware pipe filled with pieces of coke in order to reduce the sulphurous anhydride to sulphur, the temperature being regulated to the point at which complete reduction takes place without the possibility of forming carbon bisulphide. The condensation of the sulphur is performed in chambers provided with suitable means for cooling, the carbonic anhydride which escapes from the chambers in a slow and regular stream being utilised for the decomposition of the alkaline solution of phenols, *i.e.*, for the purpose of recovering the soda as carbonate, and using it over again after causticising. The sulphur obtained in this manner is very pure and without smell. Even when recovered from vitriol tar containing arsenic, it is possible to obtain a pure product providing the temperature in the still be kept within proper limits. Experiments on a small scale showed that the acid is completely decomposed at 120° to 165°. Vitriol tar containing 10 per cent. of sulphuric acid yielded from 11 to 11·5 per cent. of sulphur, the theoretical amount being 13 per cent.

On decomposing sodium phenolate with carbonic anhydride, the end of the reaction is recognised by the separation of a white flocculent precipitate of aluminium hydroxide, due to the presence of sodium aluminate in commercial caustic soda. This is not acted upon by carbonic anhydride until the sodium phenolate has been completely decomposed. After removing the phenols the solution of sodium carbonate is filtered, concentrated, if necessary, and allowed to crystallise, the solution being stirred at intervals to prevent the formation of large crystals. With proper management the mother-liquor should be charged with sodium sulphide, and readily soluble sodium salts of acid substances (fatty acids) not yet investigated, whilst sodium carbonate should be present only in small quantities. After draining and, if necessary, washing the crystals with cold water, they are heated in a flat iron pan to about 34°, at which temperature they melt. The evaporation is then continued until a white powder separates, which has the composition $Na_2CO_3 + H_2O$, and upon the formation of which the purification depends. On causticising this powder, a perfectly colourless soda solution is obtained.—D. B.

*Paraffin, Quantitative Estimation of, in Distillates from Crude Petroleum and Brown-Coal Tar.* D. Holde and A. Allen. Mitth. königl. tech. Versuchsanst. **16**, [2], 97.

*See under* XXIII., *page* 803.

*Paraffin, Commercial, Determining the Solidifying Point of.* B. Kissling. Chem. Rev. Fett- und Harz-Ind. **5**, [5], 87.

*See under* XXIII., *page* 803.

## IV.—COLOURING MATTERS AND DYES.

*Benzophenone, Crystallisation of Cooled.* K. Schaum. Zeits. physik. Chem. **25**, 722—728; Chem. Centr. 1898, **1**, [22], 1177.

The author shows that the limits of temperature at which cooled benzophenone is converted from the "metastabile" into the labile condition, are below − 20° C. and above − 79° C. At − 79° C. all the samples of benzophenone examined solidified, although the solidification proceeded more slowly in some cases than in others. Small fragments of crystallised benzophenone and also small pieces of other solid bodies, such as minute fragments of broken glass, possess the power of promoting the crystallisation. It was found that benzophenone, when strongly cooled, always solidified directly, in the "metastabile" condition; the crystals of the unstable modification obtained formerly, were not observed in these experiments.—A. S.

*Chloroproteïnochrom.* C. Beitler. Ber. **31**, [11], 1604—1610.

Gmelin observed that the addition of chlorine water to the products of the action of the pancreatic juice on albumin gave rise to the formation of a red dyestuff. Bromine acts similarly, and Stadelmann (Zeits. f. Biol. 1890, **27**, 491) termed the parent substance proteinochromogen and the bromine product proteïnochrom. The author deduces the following conclusions from his investigations:—1. Albumin contains a complex chromogenic group, which separates in the pancreatic digestion and is precipitated by chlorine water as a red chlorine substitution product. 2. This so-called chloroproteïnochrom which, according to analysis, has the formula $C_{96}H_{116}Cl_3N_{21}O_{31}S$ is decomposed in the cold by alkalis and metallic salts or by boiling with alcohol or acetic ether, yielding non-characteristic products. When it or the bromine product is fused with potash, it yields together with pyrroline, skatole, and indole. 3. Proteinchromogen, $C_{96}H_{119}O_{31}N_{21}$, is also a very easily decomposable substance. It diffuses through membranes, and cannot be precipitated by metallic salts from its aqueous solution, but only by phosphotungstic acid, from which the proteinochromogen cannot be re-obtained.—T. A. L.

*Safranine Azo Dyestuffs.* [*Basic Blues.*] E. Lauber. Färber Zeit. 1898, **9**, [14], 216—217.

Holliday patented (Eng. Pat. 1637 of 1881) the production on the fibre of Safranine-azo-naphthol dyestuffs, and Koechlin and Galland later (this Journal, 1890, 1126) communicated a process for the same purpose. Both methods were unsuccessful, the second giving a compound which was not sufficiently insoluble to be of any use for textile colouring in this manner.

Beyer and Kegel in 1885 prepared a number of hydroxyazo (Ger. Pat. 38,319) and amidoazo (addition to Ger. Pat. L. 3377, refused) derivatives of Safranines and their sulphonic acids. It was probably owing to the high price of such dyestuffs at the time, that they did not immediately find application in practice.

In 1891 it was discovered (Eng. Pat. 4513, this Journal, 1892, 236) that the compounds resulting from the combination of diazotised Safranines and naphthols in the presence of excess of alkali, were transformed by treatment with acids, for instance, hydrochloric acid, into soluble dyestuff salts. The compound from one of the Safranines and beta-naphthol has been named by the patentees, the Badische Anilin und Soda Fabrik, "Indoine Blue."

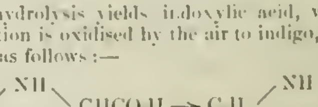
Identical or similar dyestuffs are manufactured by the firms of Kalle and Co. (Diazine Blues), The Berlin Actien Gesellschaft für Anilin Fabrikation (Indol Blue), L. Cassella and Co. (Naphthindone) (Eng. Pat. 3188 of 1895, this Journal, 1896, 110), The Elberfeld Farbenfabriken (Indigene), and The Hoechst Farbwerke (Janus Blue) (Eng. Pat. 5090 of 1896, this Journal, 1897, 233).

In calico printing the Diazine Blues are dyed in the usual manner on tissues mordanted with antimony tannate, and are printed with a discharge-mixture consisting of 6 litres of gum solution (10:10), 900 grms. of sodium chlorate, 250 grms. of potassium ferricyanide, 750 grms. of China clay, 750 grms. of zinc sulphate, 200 grms. of tartaric acid, and 1½ litres of water. The tissues are steamed for an hour, washed, and soaped at 50° C.—E. B.

*Indigo Dyestuffs, New Synthesis of.* R. Blank. Ber. **31**, [11], 1812—1817.

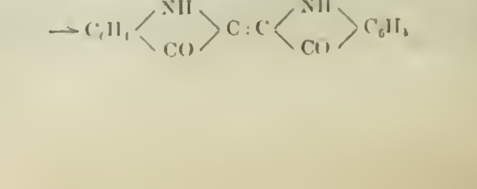
This synthesis, which has already been described in Eng. Pat. 19,946 of 1896 (this Journal, 1897, 734), consists in the inner condensation of the anilidomalonic esters, alcohol being split off simply on heating to 200° C. or above and an indoxylic ester resulting—

$$C_6H_5.NH.CH(CO_2R)_2 =$$



$$ROH + C_6H_4\left\langle\begin{matrix}CO\\NH\end{matrix}\right\rangle CHCO_2R$$

This on hydrolysis yields indoxylic acid, which in an alkaline solution is oxidised by the air to indigo, the various stages being as follows:—



$$C_6H_4\left\langle\begin{matrix}NH\\CO\end{matrix}\right\rangle CHCO_2H \longrightarrow C_6H_4\left\langle\begin{matrix}NH\\CO\end{matrix}\right\rangle CH_2$$
$$\longrightarrow C_6H_4\left\langle\begin{matrix}NH\\CO\end{matrix}\right\rangle C:C\left\langle\begin{matrix}NH\\CO\end{matrix}\right\rangle C_6H_4$$

As already described (*loc. cit.*), the alphylated amidomalonic esters are obtained by reacting with aromatic amines on the chloro- or bromomalonic esters. For example, a mixture of 239 grms. of bromomalonic ester and 184 grms. of aniline is allowed to stand for about 2 days. The melt which solidifies is washed with dilute hydrochloric acid, and the anilidomalonic ester separates as an oil. On further washing and standing in the cold, it solidifies and can be crystallised from dilute alcohol in colourless crystals melting at 45° C. When heated in a flask with agitation to 260°—265° C., condensation takes place, and on extracting the contents with alcohol, indoxylic ester is precipitated from this solution by adding water, and possesses all the characteristics described by von Baeyer.

The β-naphthylamidomalonic ethyl ester melts at 88° C., and, when fused at 230° C., it yields β naphthylindoxylic ethyl ester, which on hydrolysis and subsequent oxidation gives β-naphthylindigo (Wichelhaus, Ber. **26**, 2547; this Journal, 1894, 29). This substance in alcoholic or in aniline solutions is blue by reflected and green by transmitted light, whilst in concentrated sulphuric acid the colour is greenish-blue.—T. A. L.

*Dyestuffs, Natural and Artificial; Scheme for the Analysis of.* A. G. Rota. Chem. Zeit. **22**, [44], 1898, 437—442.

*See under* XXIII., *page* 798.

*Aniline, Quantitative Estimation of, Note on.* M. N. Schaposchinkoff. Rev. Gén. des Mat. Col. 1898, **2**, [18], 213.

*See under* XXIII., *page* 803.

## PATENTS.

*Logwood Colouring Matter, Impts. in the Treatment of.* J. E. and C. S. Bedford, Leeds. Eng. Pat. 14,043, June 9, 1897.

The patentees describe a method of oxidising fresh logwood by means of manganese dioxide. The oxidation is effected by allowing the logwood liquor, with or without the addition of acetic acid, to stand for some hours in contact with finely divided manganese dioxide at the ordinary or a slightly elevated temperature, agitating by means of a current of air or some stirring device. As soon as the liquors have developed a deep brownish-red colour, a sample tinting cold water cherry-red, the manganese is allowed to settle out and the supernatant liquors are concentrated in a vacuum pan to a density of 58° Tw. The oxidation may be effected simultaneously with extraction of the colouring matter by adding manganese dioxide to the chipped logwood in the extracting vessels, or the oxidising agent may be added directly to the dye-bath, in which case 4 per cent. of manganese dioxide on the weight of chipped logwood, is required.—R. B. B.

*Polyazo Colouring Matters [Blue], Production of New.* W. H. Claus, A. Rée, and L. Marchlewski, Manchester. Eng. Pat. 15,377, June 28, 1897.

An extension of Eng. Pats. 18,020 of 1896 and 4369 of 1897 (this Journal, 1897, 676; and 1898, 239). The new dyestuffs have the general formula N.P.G.H, where P is a *p*-diamine of the benzidine series or *p*-phenylene diamine, N is a naphthol sulphonic acid, G is γ-amidonaphthol sulphonic acid, and H is 1.1′.3.3′-amidonaphthol disulphonic acid. As a typical example, 24·4 kilos. of dianisidine are tetrazotised and poured into an alkaline solution of 22·4 kilos. of 1.4-naphthol sulphonic acid. The intermediate product obtained which still contains a free diazo group is added to an alkaline solution of the amido-azo compound obtained by combining the diazo compound from 23·9 kilos. of γ-amido-naphthol sulphonic acid with 31·9 kilos. of 1.1′.3.3′-amidonaphthol disulphonic acid in alkaline solution. The mixture is stirred for some time and is then raised to 50° C., the dyestuff being separated in the ordinary manner. It gives blue shades on cotton. If it be desired to employ *p*-phenylene diamine, *p*-amido-acetanilide is diazotised in place of the di-anisidine used above, the second amido group being diazotised after separating the acetyl group.—T. A. L.

*Colouring Matters [Yellow, Orange-red], The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 15,479, June 29, 1897.

By combining diazotised *p*-amidoazobenzene sulphonic acid with nitro-*m*-phenylene diamine, yellow to orange-red cotton dyestuffs are obtained which are very fast to acids, alkalis, and light. For example 27·7 kilos. of *p*-amido-azobenzene sulphonic acid are diazotised by means of 7 kilos. of sodium nitrite and 21 kilos. of hydrochloric acid (36·5 pure HCl). The diazo compound is then added to a dilute solution prepared by dissolving 16 kilos. of nitro-*m*-phenylene diamine in 2,000 litres of water and 21 kilos. of hydrochloric acid. The temperature is slowly raised to 50°—60° C., the dyestuff separating in orange-red flakes. After standing 12 hours it is filtered off and purified by dissolving in sodium carbonate and salting out. It gives bright orange-red shades on unmordanted cotton, fast to alkalis and acids, and also tolerably fast to light. All sulphonic acids of *p*-amidoazo compounds of the benzene series can be employed in the above process, and in place of nitro-*m*-phenylene diamine, nitro-*m*-tolylene diamine may be used.—T. A. L.

*Blue Mordant Dyeing Colouring Matters from Dinitro-anthraquinones, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Anilin and Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 16,495, July 12, 1897.

It is known that on treating dinitro-anthraquinones with fuming sulphuric acid and adding boric acid or sulphur to the melt, certain valuable results are obtained. The patentees now find that the addition of both sulphur and boracic acid simultaneously leads to the formation of new mordant dyestuffs. About 10 kilos. of 1·4′-dinitroanthraquinone, 10 kilos. of crystallised boracic acid, 2 kilos. of sulphur and 100 to 200 kilos. of fuming sulphuric acid (30—40 per cent. $SO_3$) are heated for 2—2½ hours at 120°—130° C. The melt is diluted with 2,000 to 3,000 litres of water, filtered, and the residue washed with cold water till neutral. Under the same conditions, the use of sulphur alone would have produced a reddish-blue dyestuff soluble in water, whilst boracic acid would have yielded brownish-violet dyestuffs. The new dyestuff is, however, a very green shade of blue, very slightly soluble in cold, but readily soluble in hot water. It dissolves to a yellow solution in concentrated sulphuric acid, whilst the solutions in ammonia, caustic soda and aniline are blue, the alcoholic solution is bluish-violet, and the solution in hydrochloric acid is brownish-yellow. The shades obtained are fast to fulling.—T. A. L.

*Colouring Matters [Blue] for Animal Fibre, The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Fabrik, Ludwigshafen, Germany. Eng. Pat. 16,496, July 12, 1897.

This is an extension of the Anthracene Blue patents (Eng. Pats. 19,588 and 19,589 of 1891, and 13,029, 13,147, 15,246, and 15,325 of 1892; this Journal, 1893, 257, 258, 921, 597, and 598). According to the foregoing specifications a nitro-anthraquinone or a reduction product thereof is treated with fuming sulphuric acid with or without a reducing agent such as sulphur, when intermediate dyestuffs are obtained which are soluble in water, and have the character of sulphuric acid esters. The patentees now find that on combining these esters with phenolic bodies or hydroxy carboxylic acids, new products are obtained which dye wool violet to blue shades from an acid bath. On subsequently treating them with chrome, greenish-blue to bluish-green shades are obtained. About 10 kilos. of 1.4′-dinitroanthraquinone, 50—100 kilos. of fuming sulphuric acid (12—40 per cent. $SO_3$) and 1·5 kilos. of finely divided sulphur are heated together to 130° C. for several hours until practically completely soluble in water. The melt is then allowed to cool to the ordinary temperature, when 8 kilos. of gallic acid are added and the temperature is raised with constant agitation to 50°—60° C. The reaction is complete in 5—6 hours, when the melt is cooled, poured into ice water, salted out, filter-pressed, and dried. It forms a black powder readily soluble in water dyeing unmordanted wool violet or bluish-violet, according to the strength of the anhydride employed, the shade, after chroming, becoming bluish-green or green. A table is appended giving reactions and properties of some of the new dyestuffs.—T. A. L.

*Dyestuffs [Blue, Black] for Cotton, The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 17,018, July 19, 1897.

These dyestuffs are obtained by heating with sulphur and alkaline sulphides 1.1′-amidonaphthol, 1.1′-dihydroxynaphthalene, or their sulphonic acids, and separating the products obtained by their difference in solubility in water. For example: 50 kilos. of 1.1′.4.2-dihydroxynaphthalene disulphonic acid is stirred into a mixture obtained by dissolving 200 kilos. of crystallised sodium sulphide ($Na_2S.9H_2O$) and 100 kilos. of sulphur. The whole is heated to 240°—250° C. until a test portion dissolves in water to a blue or bluish-black solution, and the colour does not increase in intensity. After cooling, the melt is powdered and repeatedly extracted with small quantities of hot water so long as the extract is of a blue or bluish-black colour. The dyestuff is then salted out from the combined filtrates, filter-pressed, and dried. It forms a bluish-black mass, and gives blue to bluish-black shades on cotton fast to alkalis and light, from hot and cold baths, containing reducing agents such as alkaline sulphides or sodium carbonate and grape sugar.—T. A. L.

*Yellow Azo Dyestuffs Fast to Washing and Light, Manufacture of.* O. Imray, London. From The Farbwerke vormals Meister, Lucius, and Bruning, Hoechst a M., Germany. Eng. Pat. 17,328, July 22, 1897.

These dyestuffs are obtained by combining certain diazo compounds with β-diketones, β-ketone aldehydes, and the ethers and anilides of β-ketone acids. The diazo compounds suitable for this process are those derived from dehydrothiotoluidine (or xylidine) sulphonic acid, primuline, amidoazobenzene sulphonic acids and the tetrazo derivatives from sulphonated diamidodiphenyl and diamido stilbene. The dyestuffs are chiefly for wool, but those from sulphonated thio bases and benzidine monosulphonic acid also dye unmordanted cotton from a neutral or alkaline bath. The diazo compound from 34·3 kilos. of sodium dehydrothiotoluidine sulphonate is run into 500 litres of water containing 12 kilos. of caustic soda lye (40° B.) and 13 kilos. of aceto-acetic ether, together with the amount of sodium acetate necessary to neutralise the excess of hydrochloric acid and to form the sodium salt. The dyestuff is then salted out and dissolves with a greenish-yellow colour. If benzidine be combined similarly with aceto-acetic ether, the yellow insoluble hydrazone can be sulphonated by stirring into five times its weight of fuming sulphuric acid (10 per cent. $SO_3$) and warming to 30° C. The melt is then poured on to ice and the dyestuff is salted out in the usual manner. —T. A. L.

*Green and Blue-green Colouring Matters from Triphenylmethane, Manufacture of.* J. Imray, London. From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, Paris, France. Eng. Pat. 18,002, July 31, 1897.

When *m*-nitrobenzaldehyde is heated with sodium sulphite it is converted into an amido sulphonic acid which, although it has not been isolated, will combine with simple or substituted secondary or tertiary aromatic amines to form leuco bases of the triphenylmethane series. A mixture of 15 kilos. of *m*-nitrobenzaldehyde, 100 kilos. of crystallised sodium sulphite, and 300 to 400 litres of water, is boiled for two hours, a current of carbonic acid being passed through, 25 kilos. of sulphuric (55° B.) are added, and then 20—30 kilos. of diethylaniline, and the boiling is continued under an inverted condenser for 15—20 hours. After making alkaline with sodium carbonate, the excess of diethylaniline is blown off with steam, the leuco base is precipitated by adding 15—20 per cent. of salt to the residue, and oxidised to the colouring matter by means of peroxide of lead. The lead is removed by sodium sulphate, and the solution of the dyestuff is evaporated to dryness. It dyes wool from an acid bath a bluish-green fast to alkalis.—T. A. L.

*Dyestuffs [Saccharo-rhodamines], Impts. in and relating to the Manufacture of.* G. B. Ellis, London. From La Société Chimique des Usines du Rhône anc. G. P. Monnet et Cartier, Lyons, France. Eng. Pat. 18,017, July 31, 1897.

The saccharo-rhodamines (Eng. Pat. 21,197 of 1896; this Journal, 1897, 795, see also 728) are not fast to alkalis, but according to the present specification they may be made so by treatment with mineral acids, acid anhydrides, or acid chlorides. The imido group is either replaced by oxygen or the hydrogen in it by an acid radicle such as $COCH_3$ or $POCl_3$. The shades vary but little from the original dyestuffs, whilst the products obtained are fast to alkalis. A mixture of 5 kilos. of diethyl-*m*-amidophenol saccharein and 50 kilos. of 70 per cent. sulphuric acid, is heated for 12 hours at 125°—130° C. The product is then poured into water, when the sulphate of the new dyestuff separates in greenish crystals. According to another example given, 5 kilos. of diethyl-*m*-amidophenol-saccharein and 20 kilos. of acetic anhydride are boiled for 2 hours. The excess of acetic anhydride is distilled off in a vacuum, and the new dyestuff, of which the base melts at about 232° C., may be recrystallised from water or alcohol.—T. A. L.

*Colouring Matters [Yellow, Brown, Violet, Black], The Manufacture or Production of New.* I. Levinstein and Levinstein, Ltd., Manchester. Eng. Pat. 18,376, Aug. 7, 1897.

The patentee oxidises *p*-nitrotoluene *o*-sulphonic acid with hypochlorites or hypobromites in presence of a caustic alkali, and reduces the dinitrostilbene disulphonic acid so formed (Eng. Pat 5351 of 1897; this Journal, 1898, 148) by means of weak reducing agents, such as invert sugar or ethyl alcohol in presence of caustic alkalis. The colouring matters so obtained dye cotton yellow to orange red shades. If the dinitrostilbene disulphonic acid be treated with strong reducing agents, a diamido disulphonic acid is formed, of which the tetrazo compound, when combined with amines or phenols, yields yellow to black dyestuffs. Moreover, when dinitrostilbene disulphonic acid is condensed with amines, diamines, nitramines, or amido phenols in presence of caustic soda, dyestuffs are formed giving yellow to red shades on unmordanted cotton, some of which can be diazotised and developed on the fibre, producing dark brown to black shades. The following quantities are given for the various workings. *Dinitrostilbene Disulphonic Acid:*—24 kilos. of *p*-nitrotoluene-*o*-sulphonic acid are dissolved in about 200 litres of hot water, and 70 litres of a sodium hypochlorite solution, containing 150 grms. of hypochlorite, and 30 grms. of caustic soda, are run in with constant agitation, the mixture being kept at 70°—80° C. When a test on acidifying no longer evolves chlorine, the whole is neutralised with hydrochloric acid, and allowed to cool. The neutral sodium salt crystallises out in yellow leaflets, and the remainder may be salted out, but in general it is not necessary to separate the acid, the liquid being used directly for the preparation of dyestuffs, or for reduction directly to diamidostilbene disulphonic acid. An orange-red dyestuff can be produced by dissolving 47·4 kilos. of the above sodium salt in hot water, adding 50 litres of a 30 per cent. caustic soda lye, keeping the solution at 60° C., and slowly running in 30 kilos. of invert sugar dissolved in hot water. When no further increase in the formation of colour takes place, it is salted out, filter-pressed, and dried. The shade may be varied by using a smaller proportion of invert sugar, a yellow being formed, for instance, by taking only 15 kilos. In order to obtain diamidostilbene disulphonic acid, 47·4 kilos. of sodium dinitrostilbene disulphonate are gradually added to a mixture of 135 kilos. of stannous chloride and 140 kilos. of 30 per cent. hydrochloric acid, and boiled. The diamidostilbene disulphonic acid separates out in white insoluble needles when the reduction is complete. As mentioned above it may be converted in the ordinary manner into its tetrazo compound, and combined with amines, phenols, and their derivatives. The following is a typical example of the condensation of dinitrostilbene disulphonic acid with a diamine:—24 kilos. of the sodium salt are dissolved in 300 litres of hot water, 60 kilos. of caustic soda lye 40° B., and 18 kilos. of *p*-phenylene diamine hydrochloride are added, and the mixture is boiled for 2—3 hours. The excess of caustic soda is neutralised, and the dyestuff is salted out, filter-pressed and dried. It dyes unmordanted cotton orange, and may be diazotised and developed on the fibre, giving dark brown to violet black shades.

—T. A. L.

*Anthraquinone Dyestuffs, The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 19,622, Aug. 25, 1897.

This is an extension of Eng. Pat. 12,011 of 1897 (this Journal, 1898, 572), and describes the sulphonation and simultaneous reduction of *p*-dinitro-anthrarufine (Ber. 29, 2940; this Journal, 1897, 135) and *p*-dinitrochrysazine (obtained by nitrating chrysazine) by treating with alkaline sulphites, bisulphites, and the like. In the first stage of the operation, intermediate products are formed, which dissolve in water with a brown colour. The transformation into the finished dyestuff can be more rapidly effected if these intermediate products are treated with caustic alkalis. For example, a mixture of 10 kilos. of a 20 per cent. *p*-dinitroanthrarufine paste and 100 kilos. of a sodium bisulphite solution (40° B.) is heated on the water-bath until almost the whole of the dinitroanthrarufine has been dissolved. The solution is then diluted with warm water, filtered, and caustic soda-lye is added to the violet solution until the colour turns greenish-blue, when the sodium salt of the diamidoanthrarufine disulphonic acid is precipitated by adding salt. In a similar manner, *p*-diamidochrysazine disulphonic acid can be obtained from *p*-dinitrochrysazine.

—T. A. L.

*Amido Base [Diamidodiphenyldisazoresorcinol] and New Direct Dyeing Polyazo Dyestuffs [Violet, Blue, Black] therefrom, Manufacture of a New.* O. Imray, London. From H. Schneider, Berlin, Germany. Eng. Pat. 19,634, Aug. 25, 1897.

Two molecular proportions of *p*-nitraniline are combined with one molecular proportion of resorcinol in an alkaline solution, and the resulting dinitrodiphenyldisazoresorcinol is reduced with sodium sulphide. This base, after conversion into its tetrazo compound, combines with various amidonaphthol sulphonic acids to form dyestuffs for cotton, fast to soap and light. The diazo solution from 28 kilos. of *p*-nitraniline diluted to 1,800 litres is run into 2,000 litres of water at about 10° C. containing 11 kilos. of resorcinol and 43 kilos. of calcined soda. The diazo body separates as a fine precipitate, and, after stirring for half an hour, the temperature is raised to 80°—90° C., and the product filtered off. It is stirred to a thin paste with about 800 litres of water, heated to 50° C., and 250 litres of a 42 per cent. solution of crystallised sodium sulphide are added with constant agitation. The temperature rises spontaneously, and is kept at 65°—70° C. until a clear solution is obtained, which is diluted to three times its volume and mixed with 90 kilos. of salammoniac to precipitate the new base, which is then filter-pressed, washed, and dried. It forms a brilliant metallic powder sparingly soluble in water, easily in caustic alkalis and in dilute hydrochloric acid, from which it is precipitated by strong hydrochloric acid as the hydrochloride. The tetrazo compound may be combined with the same or with different components, and a table is given in the specification of the various shades obtainable. The following is a typical example:—The tetrazo solution from 3·5 kilos. of *p*-diamidodiphenyldisazoresorcinol is combined with 2·4 kilos. of γ-amidonaphthol sulphonic acid in a weak acetic acid solution. The black, insoluble, intermediate product is then further combined with 3·2 kilos. of amidonaphthol disulphonic acid H. The dyestuff gives blue-black shades on unmordanted cotton.—T. A. L.

*Black Colouring Matters derived from Naphthazarine, The Manufacture and Production of New.* J. Y. Johnson, London. From The Badische Anilin and Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 21,297, Sept. 16, 1897.

Much bluer shades than from naphthazarin alone are produced on chrome mordanted animal and vegetable fibres by condensing naphthazarin with aromatic amines, with or without a condensing agent. These condensation products are only slightly soluble in water, but can easily be converted into soluble compounds, either by sulphonation with concentrated or fuming sulphuric acid, or by treatment with alkaline sulphites or bisulphites. About 1,000 kilos. of a 20 per cent. naphthazarin paste are treated with 200 kilos. of aniline in 600 kilos. of 30 per cent. acetic acid, the whole being constantly stirred. About 200 kilos. of boracic acid are added, and the temperature is kept at about 40° C. for 12 hours. When a test dissolves in caustic soda-lye to a reddish-violet instead of to a cornflower-blue solution (characteristic of naphthazarin), the melt is poured into water, filtered, and washed, when it can be employed as a paste in dyeing or printing. In order to sulphonate it, 100 kilos. are heated with 500 kilos. of sulphuric acid monohydrate for about five hours. The melt is then poured into 2,000 litres of water, boiled, filtered, salted out, filter-pressed, and dried. It forms a reddish-brown powder, dissolving in water to a red, and in caustic soda to a violet-blue solution. Another method for rendering the product soluble is to treat about 800 kilos. of the condensation product (in the form of a 20 per cent. paste) with 200 kilos. of a 40 per cent. sodium bisulphite solution, and heat to 95° C. for two hours. The paste so obtained dissolves to a brown solution in water, and the dry dyestuff also forms a brown powder. The solution in caustic soda is green, which becomes blue on exposure to the air, or more rapidly when boiled. The product dissolves to a dirty brown solution in concentrated sulphuric acid, which turns violet on heating.

—T. A. L.

*Trisazo Colouring Matters capable of Dyeing Unmordanted Cotton, and of Yielding Blue Shades by Development on the Fibre, Manufacture of New.* O. Imray, London. From The Society of Chemical Industry in Basle, Basle, Switzerland. Eng. Pat. 7659, March 30, 1898.

This is a further application of the method described in Eng. Pat. 4460 of 1894 (this Journal, 1895, 263). The azo dyestuffs, from diazonaphthol sulphonic acids and *p*-cresidine, *p*-xylidine, 1.3′- or 1.2-naphthylamine sulphonic acid, yield with tetrazo compounds intermediate products, which give new trisazo dyestuffs with one molecular proportion of a naphthol derivative. The dyestuffs give blackish-violet, grey, and black shades on unmordanted cotton, but do not possess any technical value. They are, however, of technical importance, in that when diazotised on the fibre and developed with β-naphthol, they yield violet and blue shades possessing a greater fastness to light, and being very fast to washing. These dyestuffs have the following general formula:—

$$\begin{array}{l}\mathrm{N.A.N{:}N.C}\\ \colon\\ \mathrm{N.P.N{:}N.D,}\end{array}$$

where C is an aromatic amine of the type of *p*-cresidine and D a naphthol or naphthol derivative not containing a diazotisable amido group, whilst A represents the radicle of 2.1′.3′-amido naphthol sulphonic acid or of 2.1′.3.3′-amidonaphthol disulphonic acid, and P the radicle of a *p*-diamine, such as benzidine, tolidine, ethoxybenzidine, diamidodiphenol ether, diamidodiphenylamine, *p*-amidoacetanilide, *p*-amido-acetnaphthalide, 1.4.2′- or 1.4.2-amido-acetnaphthalide sulphonic acid. For example, 34·1 kilos. of 2.1′.3.3′-amidonaphthol disulphonic acid (as acid sodium salt) are diazotised and combined with 13·7 kilos. of *p*-cresidine. After 12 hours, the azo dyestuff is dissolved by adding 40 kilos. of sodium carbonate, and the tetrazo compound from 24·4 kilos. of dianisidine is poured into the solution. The intermediate compound so obtained has added to it a solution of 15 kilos. of β-naphthol in 150 litres of water and 14 kilos. of 30 per cent. soda-lye. The whole is allowed to stand 12 hours, then raised to the boil, and the dyestuff salted out. It forms a dark powder soluble in water to a bluish-black solution, and gives bluish-black shades on unmordanted cotton. When diazotised on the fibre and developed with β-naphthol, the shade changes to marine blue. Tables are appended giving particulars of the various colours obtained.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### PATENTS.

*Vegetable Fibres in the form of Thread, Cord, Yarn, or the like, Impts. in the Process and in the Machinery for Mercerising.* R. Thomas and E. Prevost, Crefeld, Germany. Eng. Pat. 14,201, June 10, 1897.

This process consists of mercerising vegetable fibres, under tension, between the time of their being moistened with the mercerising fluid and their drying, either with:—(1) The mercerising liquid at the ordinary temperature. (2) The liquid reduced to a low temperature. (3) In addition to the above, squeezing the fibres so that an augmented gloss is obtained, (4) Passing the fibres, under tension, through rollers, alternately through mercerising and washing liquids, by which a permanent silk gloss is obtained.

Apparatus and machinery also are described by which the above processes can be performed.—C. M.

*Rhea Grass and Fibrous Plants, Impts. in the Treatment of, and in Apparatus therefor.* J. M. Macdonald and H. H. Boyle, Westminster. Eng. Pat. 14,868, June 19, 1897.

After decorticating, the plant is treated with various solutions as follows:—It is boiled in a solution of hydrate and silicate of soda, removed, washed, and re-boiled in a weaker solution. After removal and washing, it is now steeped in permanganate of potash solution, washed, treated with sodium thiosulphate, washed, then steeped in hydrochloric acid solution, thoroughly washed and digested in a soapy solution, and finally, after thoroughly washing and rinsing, passed between rollers and dried in any suitable way, when it is fit for combing.

These operations are carried out by placing the fibre in a cage fitted on to a carrier which travels across the various tanks. Between the tanks are perforated rinsing tables. After steeping in one tank, the plant is lifted out on to the table and thoroughly rinsed, then placed in the next tank, and so on.—C. M.

*Cotton and other Vegetable Fibres, Impts. in the Treatment of.* H. A. Lowe, Heaton Moor, Lancaster. Eng. Pat. 17,397, July 23, 1897.

Cotton fibres, sorted into parallel streams, and in a loose condition by carding or other operation, are treated with an alkaline hydrate of suitable strength.

The fibres, which may be used in the form of sliver lap or rove, are digested in the mercerising agent until they become almost transparent, and then, after removing the excess of alkali in a centrifugal machine, they are passed through a series of rollers, each succeeding pair of which travel faster than the preceding. The rollers are placed so close together that they grip each individual fibre and give it a glossy appearance. Immediately after leaving the rollers the fibres are washed with a water spray, and finally dried, when they are ready for spinning. It is claimed that by this method a more even gloss is given to the fibre than by the ordinary method of mercerising after spinning. It has also been found possible by this method to produce a soft weft cotton yarn with a silky lustre.—C. M.

*Waterproof Fabrics, Impts. in the Manufacture of.* T. Birnbaum, Bow, Middlesex. Eng. Pat. 22,615, Oct. 2, 1897.

Fabrics to be waterproofed are first treated with a coloured rubber ground, and upon this is placed a coloured dough striping, according to the method described in Eng. Pat. 29,556, Dec. 23, 1896. This surface is then finished with farina, mixed with various metallic powders, according to the shade desired.—C. M.

*Lining Stuffs with Paper Pulp [for Cement- or Chemical Manure Bags, &c.], A Process for.* F. Holtschmidt, Brunswick, Germany. Eng. Pat. 3,553, Feb. 12, 1898.

The process is claimed, which is distinguished by the mass of liquid paper being brought separately on the two sides of the stretched tissue, the intimate combination taking place by passing over the rollers of an ordinary paper machine. The contrivance is claimed for effecting the above process, which is distinguished by the upper apron for the mass of paper being inclined towards the lower apron, so that the stretched tissue covered with paper pulp is brought on the mass of paper on the lower apron, the covered tissue then passing between the roller of the paper machine.—A. C. W.

*Hanks and Skeins in the Stretched Condition, Impts. in the Method of and Apparatus for Mercerising.* W. E. Heys, Manchester. From Henri David, Paris. Eng. Pat. 10,246, May 5, 1898.

Yarns and threads, in hanks or skeins, are treated, in suitable machinery described, with mercerising fluids while under tension. The yarn is passed between a pair of rollers, one of which is perforated, and whose interior communicates with a vacuum chamber. In this manner the mercerising fluid is forced through the fabric, which can be mercerised and washed without removal from the roller.
—C. M.

*Fabrics from Nitrated Yarns, chiefly Designed for Filtering Purposes; A Process for the Manufacture of.* Emile Müller, Cologne, Germany. Eng. Pat. 12,867, June 8, 1898.

This process consists in nitrating the yarns intended for the warp and weft, with a mixture of nitric and sulphuric acids, and then completely freeing the yarn from adherent nitric acid by a centrifugal apparatus, and ample washing. The nitrated yarns are then woven in the wet condition.

In this way it is claimed that cloth can be made suitable for filtering strongly acid liquids.—C. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Dyeing, Theory of.* R. Gnehm and E. Rötheli. Zeits. für angew. Chem. 1898, [22], 501—505 (Conclusion). (This Journal, 1898, 660—661).

The palmitates are prepared similarly to the stearates, and a number of compounds of dyestuffs with amido acids are also described. Since the existence of compounds of colour bases with weak acids is to be regarded as evidence in favour of the chemical theory of dyeing, it should be possible to prepare compounds of acid dyestuffs with faintly basic bodies, and a number of these substances have been produced, as for example, Naphthol Yellow S and aniline, benzidine (only equimolecular proportions combine) and amidopalmitic acid.

In view of all the facts brought forward, the authors arrive at the following conclusions. The dyeing of different fibres is not a homogeneous process, and the dyeing of vegetable fibres does not depend upon the same processes (or only partially) as in the case of animal fibres. Even with these latter, different phenomena take place, mechanical effects accompanying chemical reactions. Hence, all the following cases must be taken into consideration:—1. Cotton dyeings on mordanted fibres; lakes formed from the dyestuff and the mechanical precipitate of the mordant on the cotton. 2. Pigments and azo colours formed on the fibre; purely mechanical precipitates on the fibre. 3. Indigo and basic substantive cotton dyestuffs; absorption of the dyestuff according to Georgevics. 4. Direct cotton dyeings with benzidine colours; solutions of the dye salts in the cell sap due to their small coefficient of diffusion. 5. Dyeings of mordanted wool and silk: colour lakes with the mordant fixed partly chemically and partly mechanically (the fibre taking no part in the fixing of the dyestuff so far as it does not combine chemically with the mordant).

6. Substantive dyeings on animal fibres; mixtures of chemical compounds with mechanically absorbed dyestuff.

—T. A. L.

*Colouring Matters, Dyed on Nineteen Metallic Mordants.* A. Scheurer and A. Brylinski. Bull. Soc. Ind. Mulhouse, 1898, April—May, 124—130.

The results of exposures to light of different colours (this Journal, 1897, 911) are seen below in a convenient tabulated form.

These exposures to light are performed in the way described on page 759 (see article "Action of Light upon Colours," A. Scheurer). That is, in comparison with a scale of vat-indigo blue colours, thus:—No. 1. Deep blue (four dips), type of *permanent* colour. No. 2. Medium blue (two dips), type of *moderate* colour. No. 3. Light blue (one dip), type of *fugitive* colour.

| Mordant. | Colouring Matter. | Fastness. |
|---|---|---|
| All mordants ........ | Tannin | No. 1. |
| „ ........ | Limawood | All colours changed to mode shades of fastness No. 2. |
| Cu.................... | Logwood | Very superior to No. 1 indigo. |
| Other mordants ..... | „ | Colours changed to dirty mode shades. |
| All mordants ........ | Cuba Wood | No. 1. |
| „ ........ | Sumach | No. 1. Fe, Ce, not so fast as the others, but faster than No. 1 indigo. |
| Ur, Cu, Cr, Fe, Y, Zr . | Quercitron Bark | No. 1. |
| Th .................. | „ | No. 2. |
| Al, Gl, Bi............ | „ | No. 3. |
| Cr, Fe. Ur, Zr ........ | Persian Berries | No. 1. |
| Al, Gl................ | „ | Changed to yellow mode colour. |
| Th.................... | „ | Colour not so full as on Zr. |
| Cu, Cr, Ur, Fe, Y, Zr, Bi. | Weld | No. 1. |
| Gl .................. | „ | Shade darker and more orange; not so bright. |
| Sn, Al ............... | „ | Changed to yellow mode shade. |
| Th.................... | „ | Not so fast as Zr. |
| Ur.................... | Turmeric | No. 1. Gl destroyed; the other colours more or less destroyed. |
| Ur ................... | Cochineal | Changed to yellowish colour, No. 1. |
| Zr, Th, Gl, Cr, Cu .... | „ | Turned greyish, No. 1. |
| Fe.................... | „ | Turned yellowish, No. 1. |
| Al ................... | „ | Turned purple, No. 2. |
| Ce.................... | „ | No. 2. |
| Sn, Bi................ | „ | Destroyed. |
| Sn, Ur, Fe ........... | Gambier Cutch | No. 1. |
| Al, Gl................ | „ | Changed to mode colour, No. 2. |
| Bi ................... | „ | No. 3. |
| Th, Zr, Cr............ | „ | Changed to mode. |
| Sn.................... | Pegu Cutch | Increased and brightened in shade. |
| Ur, Cu................ | „ | No. 1. |
| Th, Zr, Bi ........... | „ | No. 2. |
| Al, Y, Cr, Pb, Ni, Co.. | „ | Colours changed to greyish; No. 2. |
| Ce, Hg, Zn........... | „ | No. 3. |
| Cu.................... | Naphthol Yellow S (Badische). | Completely faded. |
| Bi.................... | Alizarin Yellow GG (M. L. & B.). | Changed to mode colour. |
| Cu, Cr, Ni. Co, Fe .... | „ | No. 1. |
| Zr, Th ............... | „ | Colours changed; No. 1. |
| Ni ................... | Sun Yellow (Leonhardt). | No. 1. Ground colour; No. 1. |
| Ni ................... | Naphthol Green (Cassella). | No. 1. |
| Bi ................... | „ | Fugitive. |
| Zr, Th, Cu, Cr, Ur, Gl | Alizarin Yellow A (Badische). | No. 1. |
| Bi ................... | „ | Fugitive. |
| Zr, Th, Gl, Cr, Ni .... | Alizarin Black (Badische). | No. 1. |
| Y..................... | „ | No. 2. |
| Ur ................... | „ | Colour changed to yellowish. |
| Bi, Ce................ | „ | No. 3. |
| Al, Fe. Cr, Cu, Ce, Pb. | Alizarin V. | Unchanged. |
| Zr, Th. Gl, Ur, Ni, Mn, Y. | „ | No. 1. |
| Bi, Sn, Hg, Zn, Cd, Co | „ | No. 2. |
| Al, Fe, Cu. Ce, Pb, Zr. Gl, Ur, Mn. | Alizarin R. | Unchanged. |
| Cr. Th, Y, Sn, Hg .... | „ | No. 1. |
| Ni, Zn, Cd, Co........ | „ | No. 2. |
| Al, Fe, Cu, Zr, Ur, Y . | Alizarin J. | Unchanged. |
| Ce, Pb, Gl, Mn, Cr, Th, Sn. | „ | No. 1. |
| Hg, Ni, Cd, Co ....... | „ | No. 2. |
| Zr, Y, Fe, Gl, Cu, Cr, Pb, Ni. | Anthraceno Brown. | No. 1. |
| Al, Th, Bi, Ce........ | „ | Between No. 1 and No. 2. |
| Ur, Sn ............... | „ | No. 2. |
| Bi, Th, Y, Zr, Al, Gl, Cr ................<br>Ce, Ur, Pb, Fe, Cu, Mn ................ | Nitro-alizarin | No. 1. |
| Sn, Ni, Zn ........... | „ | Between No. 1 and No. 2. |
| Th, Zr. Gl, Y, Al, Cr, Pb, Cu, Fe, Ur. | Alizarin Maroon (Badische). | No. 1. |
| Ce, Ni ............... | „ | No. 2. |
| Bi, Zn, Hg ........... | „ | No. 3. |
| Sn ................... | „ | Colour destroyed. |
| Fe, Al ............... | Alizarin Blue X (Badische). | Unchanged. |
| Gl, Zr, Th, Cr (colours only weak). | „ | No. 1. |
| Bi ................... | „ | No. 2. |
| Sn, Pb, Hg, Y........ | „ | No. 3. |
| All mordants......... | Alizarin Green S (Badische). | No. 1. Al, colour changed; Sn, colour changed to purple. |
| All mordants ........ | Malachite Green | Colours completely destroyed. |
| Ur.................... | Pyronine G (Leonhardt). | Colour changed to rosewood; No. 2. |
| All other mordants... | „ | Colours destroyed. |
| Ni ................... | Patent Blue (M. L. and B.), | Colour changed to light grey. |
| All other mordants... | „ | Colours totally destroyed. |
| Ni, Zr, Th ........... | Azo Green (Bayer). | Colours almost destroyed. |
| All other mordants... | „ | Colours destroyed. |
| All mordants ........ | Methyl Violet B (Badische). | Colours destroyed, leaving varied light grey tones. |
| Cu, Cr, Y, Th, Ce, Ni . | Chrome Violet (Geigy). | No. 1. |
| Gl Zr, Al ............ | „ | No. 2. |
| Co ................... | Eosin (Badische). | No. 1. |
| Ni ................... | „ | No. 2. |
| Bi, Y, Zr, Th......... | „ | Colours destroyed. |
| All mordants ........ | Rhodamine B (Badische). | Colours more or less destroyed. |
| Al, Y ................ | Gallein (Badische). | No. 1. |
| Sn, Ni, Cr, Zn, Pb, Th, Zr, Gl. | „ | No. 2. |
| Ce ................... | „ | No. 3. |
| All mordants ........ | Coeruleïn (Badische). | Colours unchanged. |
| Ur, Zr, Th, Pb, Co.... | Gallocyanine (Durand). | „ „ |
| Bi ................... | „ | Colour becomes flat; No. 1. |
| Al, Ni, Fe, Ce, Cd, Zn. | „ | No. 1. |
| Sn, Gl ............... | „ | No. 2. |
| Hg ................... | „ | No. 3. |
| Ur.................... | Methylene Blue | Colour changed to dark grey. |
| Bi, Hg, Sn, Ce, Cd, Zn, Zr, Th. | „ | Colours destroyed. |
| Ur.................... | Safranine (Badische). | Colour changed to reddish-wood. |
| All other mordants... | „ | Colours destroyed. |
| Ur.................... | Magdala Red (Durand). | No. 1. |
| Zr, Th ............... | „ | Colours destroyed. |
| Ur.................... | Benzoflavine (Oehler). | Changed to wood colour, and is darker than before exposure. |
| All other mordants... | „ | Colours more or less destroyed. |
| Zr ................... | Indigo Carmine | No. 3. |
| Ni ................... | Mikado Yellow (Leonhardt). | No. 1. Ground colour; No. 1. |
| Hg, Zr ............... | Soudan G (Berlin). | Colours totally destroyed. |
| Ni ................... | Erica B (Berlin) | No. 1. |
| Zr, Th, Cr ........... | Resorcinol Yellow (Berlin), | Colours totally destroyed. |
| Ni ................... | Acid Ponceau (Durand). | No. 1. |
| Y, Th, Zr ............ | Yellow M G (Poirrier). | Colours changed to mode shades. |
| Bi, Cd ............... | „ | „ „ |
| Cu (dirty violet)..... | Cloth Red (Bayer). | No. 1. |
| Co (mode violet) ..... | „ | „ |
| Ni ................... | „ | No. 2. |

| Mordant | Colouring Matter. | Fastness. |
|---|---|---|
| Ni .................... | Poneeau 6 R B (Berlin). | No. 1. |
| Cu, Cr .................... | Diamond Black (Bayer). | Colours scarcely altered. |
| Gl, Ni, Y, Zr, Th ..... | „ | No. 1. |
| Bi .................... | „ | No. 2. |
| Ur .................... | „ | Colour destroyed. |
| Mn, Bi, Ur, Zr, Th... | Bismarck Brown | Colours destroyed. |
| Ni .................... | St. Denis Red (Poirrier). | No. 2. |
| Bi .................... | „ | Colour destroyed. |
| Ni .................... | Chrysophenine (Berlin). | Colour unchanged. |
| Bi .................... | „ | No. 1. |
| Ni .................... | Congo G R (Berlin). | Colour changed and diminished. |
| Ni .................... | Benzo Orange (Bayer). | Colour becomes yellowish: No. 1. |
| Zr, Th, Y, Gl.......... | „ | Colours changed to cutch-brown of fastness nearly No. 1. |
| Ni .................... | Diamine Violet (Cassella). | No. 2. Ground colour; No. 1. |
| Gl .................... | Carbazol Yellow (Badische). | Colour increased to olive-yellow, which remains unchanged. |
| Zr, Th, Ni, Cu, Cr .... | „ | No. 1. |
| Hg, Ur .................... | „ | No. 2. |
| Ni, Co .................... | Diamine Blue (Cassella). | Colours turned violet; No. 1. |
| Th .................... | „ | Colour fugitive. |
| Fe .................... | Dinitroresorcinol | No. 1. |
| Co .................... | „ | No. 2. |
| Cu, Mn, Co, Ni, Fe, Y | Nitrosonaphthol | Colours unaltered. |
| Ur .................... | „ | Colour becomes cutch. |
| Cr, Pb .................... | „ | No. 2. |
| Bi, Zr, Th, Sn ......... | „ | Colours destroyed. |

—J. E. H.

*Substantive Colours Dyed upon 20 Metallic Mordants.* A. Scheurer and A. Brylinski. Bull. Soc. Ind. Mulhouse, 1898, April—May, 131—147.

The mordants selected for experiment were Ce, Th, Zr, Y, Gl, Al, Co, Ni, Ur, Cr, Cu, Zn, Cd, Mn, Sb, Bi, Pb, Sn, Ti, Fe.

An attempt made by the author to classify about 50 substantive dyes, according to the manner in which they behave towards the above mordants, is shown below:—

| — | Mordant. | Name of Colour. |
|---|---|---|
| Class I..... | Clear dyeings on Ni and Co. Zr, Ce, Th, Y, Ur, Bi, act more or less as resists. | Benzo Blue-Black (Bayer). Benzo Blue-Black G (Bayer). Benzo Blue-Black 5 G (Bayer). Curcumine G (Berlin). New Serge (Geigy). Mikado Orange (Leonhardt). |
| Class II. .. | Clear dyeings on Co, and Ni. Cu, Ur, act as resists. | Tolamine Blue (Gerber). Tolamine Green (Gerber). Diamine New Blue (Cassella). Diamine Blue 2 B (Cassella). |
| Class III. | Clear dyeings on Co, Ni, and Cr. Ur or Cu acts as a resist. Zr or Y acts as a resist, sometimes simultaneously. | Brilliant Azurine 5 G (Bayer). Direct Brown G (Geigy), almost belongs to Class I. except that it dyes upon Cr and that Cu acts as a resist. Sun Yellow (Geigy). Diamine Orange B (Cassella). Diamine Violet N (Cassella). |
| Class IV... | Clear dyeings on Co, Ni, and Cr. Cu, Ur, Th, Zr, Sn, act as resists. | Chloramine Brown (Bayer). |
| Class V.... | Clear dyeings on Co, Ni, Cd, and Cr. Ur, Zn, Th, Zr, Y, act more or less as resists | Direct Brown R (Geigy). Diamine Orange (Bayer). Chrysophenine (Leonhardt); no mordant acts as a resist for this colour. However Y is only very slightly dyed. |
| Class VI... | Clear dyeings on Co, Ni, Cr, Zn. | Benzo Sky-Blue (Bayer), also known as Pure Benzo Blue. |
| Class VII.. | Clear dyeings on Co, Ni, Cd, Mn, and Cr. Ur acts as a resist. Ti occasionally. | Erica N B (Berlin). Erica 2 N G (Berlin). Chicago Blue 6 B (Berlin). Chicago Orange R (Geigy). |
| Class VIII | Clear dyeings on Co, Ni, Cr, Gl, Y, and Al. Ur, Cu, Ti, act as resists. | Chicago Blue 4 B (Berlin). |
| Class IX... | Clear dyeings on Co. Ni, Cr, Cu, Zn, and Cd. Ti, Ur, Y, Zr, Th, act as resists. | Chloramine Brown (Bayer). Geranine (Bayer). Oxamine Blue (Badische). |
| Class X.... | Clear dyeings on Co. Ni, Cr, Gl, Zn, and Cd. Ur, Ti, act as resists. | Rose Aldazine (S C I). Diamine Scarlet B (Cassella). |
| Class XI... | Clear dyeings on Co, Ni, Cu, Cr, Gl, and Zn. Ur acts as a resist. | Titan Rose (R H). |
| Class XII.. | Clear dyeings on Ce, Th, Zr, Y, Gl, Al (feebly), Co, Ni, Cr, Cu, Mn, Ti, and Fe. Ur does not dye. | Benzo Chrome Brown G (Bayer). Benzo Chrome-Brown B (Bayer). Benzo Chrome Brown R (Bayer). Benzo Green G (Bayer). Benzo Green B B (Bayer). Direct Catechu Brown G (Rose). Direct Catechu Brown B (Rose). Diamine Green B (Cassella). Diamine Bronze (Cassella), dyes on Ur, but lighter than the ground colour, *i.e.*, colour without mordant. |
| Class XIII. | Clear dyeings on all mordants except Sn, Sb, Ur. | Chicago Orange R (Geigy). Congo Brown (Berlin). Benzo Dark Brown (Bayer). Diamine Fast Yellow (Cassella): Ti acts as resist for this colour. |
| Class XIV. | Clear dyeings on all mordants except Sn, Sb. | Yellow C R (Gerber). Toluylene Orange G (Berlin). Oriol Yellow (Geigy). Diamine Yellow N (Cassella). Diamine Fast Red F (Cassella). Chrysamine (Bayer). |

The numerous colours obtained on the various mordants were tried with regard to their fastness towards soaping either at 60° C. or at boiling point.

[In all cases the soap solution was made to contain 2 grms. of soap per litre.]

The following list of substantive dyes which are distinguished by the good resistance to soaping when dyed on certain mordants is given by the author:—

| Name of Colour. | | Mordant. | Resistance to Soaping. |
|---|---|---|---|
| Oxamine Blue (Badische). | Upon | Ni, Co, Fe ... | Good. |
| Chicago Blue 6 B (Berlin). | „ | Ni ............ | „ |
| Congo Brown (Berlin) .... | „ | Cu, Cr ........ | Very good. |
| | „ | Ni, Co, Gl, Y, Zr, Th, Ti, Fe. | Sufficient. |
| Diamine Orange (Bayer) . | „ | Co ............ | Very good. |
| Benzo Dark Brown „ . | „ | Cr ............ | Good. |
| Benzo Blue Black „ . | „ | Ni ............ | „ |
| Curcumine S (Berlin) .... | „ | Ni ............ | „ |
| Toluylene Orange G (Berlin). | „ | Cr ............ | „ |
| | „ | Gl ............ | Good enough. |
| Brilliant Azurine 5 G (Bayer). | „ | Ni ............ | Good. |
| Direct Brown R (Geigy) .. | „ | Ni, Co ........ | „ |
| Oriol Yellow „ ... | „ | Cr ............ | „ |
| Sun Yellow „ ... | „ | Ni ............ | „ |
| New Serge „ ... | „ | Ni, Co ........ | „ |
| Chicago Orange G „ ... | „ | Cr, Ni, Co.... | „ |
| Benzo Chromo Brown G (Bayer). | „ | Cr, Cu ........ | „ |
| Diamine Bronze (Cassella) | „ | Cr, Cu ........ | „ |
| Benzo Green (Bayer) ..... | „ | Cr, Cu ........ | „ |
| Direct Catechu Brown G (Rose). | „ | Cr, Cu, Ni ... | „ |
| Direct Yellow C R (Gerber) | „ | Cr ............ | „ |
| Tolamine Blue „ | „ | Ni ............ | „ |
| „ Green „ | „ | Ni ............ | „ |
| Diamine New Blue R (Cassella). | „ | Ni ............ | Very good to boiling soap solution. |
| Diamine Blue 2 B (Cassella). | „ | Ni ............ | Good to boiling soap solution. |
| Diamine Yellow N (Cassella). | „ | Cr, Ni, Gl, Zr, Th. | „ „ |
| Diamine Orange B (Cassella). | „ | Co ............ | „ „ |
| Diamine Fast Red F (Cassella). | „ | Cr ............ | „ „ |
| Chrysamine (Bayer)...... | „ | Cr, Gl, Cu.... | „ „ |

—J. E. H.

### *Action of Light upon Colours, Means of Measuring the.* A. Scheurer. Bull Soc. Ind. Mulhouse, 1898, April—May, 119—123.

In this paper the author after a few theoretical considerations relating to the action of light upon colours, refers to the several conditions to be observed in choosing a colour-standard by means of which other colours are to be compared.

(1.) The fibre chosen must be of constant composition and must be unaffected by exposure to light, that is, must undergo no chemical change whatever. The cotton fibre is the most suitable.

(2.) A colour must be had which is fixed on cotton directly. Mordants as a rule exert a very perceptible influence upon the fastness of colours to light, generally producing increased resistance; hence this factor must be eliminated.

(3.) The colour selected must possess moderate resistance to light. Indigo fulfils both these conditions, (2) and (3).

Having thus determined that cotton dyed with indigo is the most suitable standard for comparison, three shades of indigo blue, viz., *deep*, *medium*, and *light* are taken as standard types.

These are vat-dyed blues and correspond to 4 dips, 2 dips, and 1 dip respectively.

Further, the author states that this classification is based upon an experience extending over 15 years.

A colour is said to be of *good fastness* to light which corresponds to the fastness of the deep indigo blue; of *moderate* fastness, which corresponds to that of the medium blue; of *little fastness*, which corresponds to that of the light; *fugitive*, which is inferior to that of light blue.

Comparison with these types will allow of a limit, to the fading which each colour undergoes, being established, which must not be exceeded in practice.

Also the fastness of any colour, whatever its depth of tone, can thus be determined by comparing it with the indigo standards.

The experimental method adopted by the author for measuring the action of light upon colours comparatively, is essentially as follows:—

The colour to be tried is dyed in three shades, deep, medium, and light.

This scale is exposed by placing it alongside of the three indigo standards in a photograph frame under glass.

The action to light is continued until the light indigo standard is destroyed in shade to one half of its original depth.

This point is determined by comparison with a standard light indigo, the shade of which is considered, after numerous experiments in exposure, to be reduced to one half.

If the colour has not undergone any notable change, the exposure is continued further until the medium indigo standard is reduced to one half.

Although the deep indigo standard does not allow of any very distinct valuations being made, it nevertheless gives indications which are certainly useful. In this way, by comparison with the indigo types, the fastness of a colour is classed as good, moderate, small and fugitive.

This method can be varied by employing only one indigo standard, *viz.*, the light blue. In this case the standard must be replaced during the course of the experiments, so often as it is considered to have become reduced in depth of shade to one half.

Further, this mode of working will demand more care and attention, because the moment of semi-decoloration must be exactly observed.

Allowing for the fact that an error must result, since the eye will not always accurately estimate the exact point of of semi-decoloration, still the approximation is sufficiently near for all practical purposes. Indeed the author states that the error due to the eye amounts to some 3 per cent. in the blue and 8 per cent. in the red.—J. E. H.

### *Alizarin Yellow F S, Note on.* J. Brandt. Bull. Soc. Ind. Mulhouse, 1898, April—May, 115—118.

Alizarin Yellow F S (Durand) is sold in the form of a 20 per cent. paste. Like several of its congeners, *e.g.*, Alizarin Yellow R W and G G (M. L. and B.), it is not an anthracene derivative, but properly an azo colour obtained by the coupling up of the diazo derivative of Magenta and salicylic acid. It gives pretty brass-yellow shades, fairly fast to light and soaping, when dyed on cotton mordanted with chromium. Wool and silk are dyed directly, without the intervention of a mordant.

However, this colour, when printed on cotton, gives only dull shades. Nevertheless, by the employment of reducing agents, such as stannous chloride, or of certain oxidising agents, *e.g.*, potassium ferricyanide, very useful printed effects are obtained.

(*a.*) *Results with stannous chloride.*—Theoretically stannous chloride should split up the molecule of Alizarin Yellow F S into Magenta and salicylic acid. This is what actually occurs. By dyeing cotton mordanted with chromium with Alizarin Yellow F S, printing the dyed fabric with stannous chloride (100 grms. per litre of thickening), afterwards drying and steaming for three minutes, a bright puce shade (characteristic of magenta) is developed, which easily withstands soaping at 50° C.

The intensity of the puce obtained will be, of course, proportional to the depth of the dyed shade. By printing stannous chloride upon a light dyed shade, a rose-violet shade will be obtained instead of a puce.

An example will render clear the interesting applications of this process: Mordant the cotton fabric with weak alkaline chrome mordant. Print on a white and black discharge, thus giving a design in black and white (the black discharge is obtained by mixing the white discharge with Aniline Black). Print over thickened chromium acetate, steam, wash with chalk water, and dye with Alizarin Yellow F S. After dyeing, print with stannous chloride, dry, steam for three minutes, and pass through chalk water to neutralise excessof stannous chloride. A design in five colours is thus obtained; black and white discharged on the yellow ground, which is reduced to puce and rose-violet shades in the parts printed with stannous chloride, puce where the chromium acetate has been printed, rose-violet on the original yellow ground.

Further, the Alizarin Yellow F S can be combined with other colouring matters, *e.g.*, with Pherocyanin R, which gives a green, which is reduced by the stannous chloride to a purplish blue. Thus infinite variations of this process are possible. This reduction of Alizarin Yellow F S by means of stannous chloride is also applicable to wool and silk, when brighter shades are obtained than on cotton.

(*b.*) *Results with potassium ferricyanide.*—This oxidising discharge consists of potassium ferricyanide, an alkaline chlorate, and citrate, citric acid, and occasionally magnesium carbonate (which has no action upon Alizarin Yellow F S dyed on chrome mordant).

By printing this discharge upon cotton dyed green with a mixture of Alizarin Yellow F S and blue, *e.g.*, Celestine Blue, there is obtained, after steaming, a yellow discharge upon green, since the Celestine Blue is destroyed by the oxidising discharge.

By dyeing this green upon chrome mordant, printing on a discharge, say in black and white, and again printing with the above oxidising discharge, pretty effects are obtained.

Further effects are got by printing on stannous chloride, but in this case the quantity of stannous salt must be reduced to 50 grms. per litre of thickening, since prolonged steaming (one hour) is necessary to obtain good results with the foregoing oxidising discharge.

Finally, the property of this oxidising discharge to act as a resist to the stannous salt may be utilised for further new effects.

These different reactions have been employed by the author since September 1896.—J. E. H.

### *Indigo-Vat, Electrolytic.* A. Binz. Zeits. f. Elektrochem. 1898, **5**, [1], 5—9.

Previous attempts to effect the electrolytic reduction or hydrogenation of indigotin have not met with much success (this Journal, 1894, 387 and 388). The preparation of an indigo vat or the transformation of indigotin into indigo-white, by means of zinc dust and alkali, is usually represented by the equation—

$$C_{16}H_{10}N_2O_2 + H_2 = C_{16}H_{12}N_2O_2.$$

In confirmation of the results obtained by Goppelsroeder the author finds that electrolytic hydrogen can to a limited extent reduce indigotin to indigo-white. Zinc, however, acts much more rapidly, and he concludes that in the ordinary indigo vat (zinc and alkali), the colouring matter is attacked and transformed directly by the metal itself. A weak vat can be obtained on electrolysis by using a zinc anode, but very soon a point is reached when the sodium indigo-white itself begins to conduct, and this is accompanied by the re-formation of indigotin at the anode so that the process cannot proceed beyond a certain limit.

For practical purposes, therefore, it is necessary to employ a cathode vat which can be separated from the anode compartment by means of a porous diaphragm. But instead of using electrolytic hydrogen as the active agent for the production of an indigo vat it would seem as if better results could be obtained by liberating zinc directly at the cathode. Since the solution must be alkaline, the proper electrolyte for the purpose is sodium zincate. Details are promised in a future communication.—J. S.

### *Velvet Dyeing, Diazine Colours in.* E. Kutschera. Färber Zeit. 9, [11], 168.

In addition to Diazine Green for the dyeing of velvet (this Journal, 1898, 153) the author mentions Brilliant Diazine Blue and Diazine Black, which are applied in a similar manner.

They possess the same valuable properties as Diazine Green, being quite as fast to light and rubbing, and even superior in their fastness towards acids.

They are lacking in brilliancy to be used as self-colours, but are excellently adapted for shading.

Thus, Brilliant Diazine Blue will be useful for deepening the blue shades obtained with Methylene Blue, Methyl Violet, Brilliant Green, &c.; Diazine Black for darkening blue, green, and olive shades, which have been dyed directly with substantive dyes, or upon tannic acid fixed with tartar emetic.

Similarly, both the dyestuffs in question may be used (like Diazine Green), for shading mode and brown colours, whether the latter have been obtained with catechu and iron mordant, or by one of the above methods. Diazine Black alone gives with tannic acid and tartar emetic a deep grey shade, which, on account of its fastness towards light and rubbing, can be employed for the collars of mantles and overcoats.—J. E. H.

### *Furs, The Dyeing of.* E. Gruene. Färber Zeit. 1898, 9, [11], 215—216.

Two methods are practised in the dyeing of furs, *viz.*, immersion in the requisite solutions, and brushing these over the surface, the latter method being adopted when it is desired to keep the leather of the furs uncoloured.

The colours commonly dyed are black and brown. Yellow, red, violet, and blue are dyed on lambskins alone, suitable dyestuffs for the purpose being Cotton Yellow, Thiazine Red, Oxamine Violet, and Naphthyl Blue, which are applied from baths containing sodium sulphate and acetic acid.

For black, a mordanting mixture is prepared by grinding and mixing together litharge (35 grms.), copper oxide (25), ammonium chloride (15), crude potash (20), adding slaked lime (from 250 grms. of quicklime), and water (750), and diluting (to 1½ litres). This is kept for several days before use.

This mixture is twice brushed, as evenly as possible over the furs, which are then dried in the shade. After being beaten and brushed they are ready for dyeing.

The operation of dyeing is performed in the same manner as the mordanting. A mixture is used, composed of roasted gall-nuts (250 grms.), ferrous sulphate (40), copper acetate (20), alum (20), sumac (20), antimony trisulphide (20), and litharge (35), all finely ground and mixed with water (1¼ litres).

After the dyeing mixture has been applied, the furs are dried. Then a second application of the mixture is given them. Thereupon the furs are taken and laid in pairs, face to face, and left in the wet state for six hours. At the end of this time they are again dried, and the treatment is repeated, if necessary, to obtain the desired shade. Finally, the loosely adherent colour-lake is removed by brushing and beating (see this Journal, 1896, 510).—E. B.

## PATENTS.

### *Indigo-Dyed Cotton Fabrics, Production of Patterns on.* J. A. O'Loughlin, Mayfield, Manchester. Eng. Pat. 21,287, 1894.

White, yellow, green, or light blue patterns are produced on indigo-dyed cotton fabrics as follows:—The unbleached fabric, after doubling it or combining two plies by passing them in a wet state between rollers, is treated on the outer surface with the dye by continuous indigo-dyeing apparatus. The fabric dyed in this or in the usual manner is printed with a mixture of acetate, nitrate, and chromate of lead, and after drying, it is passed through a bath of sulphuric and oxalic acids and some glycerin. It is next washed, passed through weak alkali, preferably ammonia, again washed, and then it is raised by passing through chromate of potash or soda solution, whereby a yellow pattern is produced. If the indigo is not completely discharged, the fabric may be passed again through the acid, alkaline, and chromate baths. Green is produced by adding Prussian blue to the mixture for yellow. For white, acetate and nitrate are used with sufficient chromate to discharge the indigo in the acid-bath. The insoluble lead salts formed yield an opaque white. For light blue, Prussian blue is used in the process for white.

### *White and Coloured Patterns on Cotton Fabrics in Combination with Azo Dyes, Impts. in Producing.* J. Robb and J. Grime, Lanark. (Second Edition.) Eng. Pat. 256, Jan. 4, 1895.

The fabric is printed over with one or more resist compositions prior to the formation of the azo-dye on the fabric, which is afterwards produced in the usual way by padding with sodium naphtholate and developing with a diazotised aromatic amine. The resists, which must be applied in a heated state, are contained in steam-jacketed colour boxes. For a white pattern, the resist is prepared by taking 1 lb. of tartaric or citric acid, 4 lb. of British gum, 1 lb. of glue, 1 lb. of paraffin wax, and enough water to make up the mixture to 1 gall. For yellow, blue, and green patterns, nitrate of lead, Prussian blue, or a mixture of these two, is added respectively to the composition for white. Where a lead salt has been used, the fabric, after developing, is passed through potassium chromate. A feature of the patent is the employment of paraffin wax as an ingredient of the resist. A mixture of gum-water and oxalate of tin may be used for diminishing the intensity of the ground colour.—J. E. H.

### *White and Coloured Patterns on Aniline Black Grounds in Calico Printing, Impts. in the Production of.* E. Bentz, Manchester. Eng. Pat. 10,213, April 24, 1897.

This invention consists in fixing by precipitation upon the fabrics suitable resists, such as the oxides or hydroxides and carbonates of calcium, cadmium, zinc, or lead, the carbonates of strontium or barium, the oxide or hydroxide of magnesium, the silicates of calcium, magnesium, &c., the borate or sulphite of calcium and other compounds, or mixtures of these, if a white pattern is to be produced; or mixed with insoluble azo colouring matters formed on the fibre if coloured patterns are to be produced. The fabrics are then padded with an aniline black padding mixture, or printed over in pattern with an aniline black printing mixture, dried, aged, or steamed, rinsed in water, and soaped or chromed as usual. When lead salts are employed as resists, the fabrics are soaped and not chromed, unless it be desired to produce yellow resist patterns along with patterns of other colours.—R. B. B.

### *Indigo, Impts. in Dyeing Vegetable Fibre with.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen a Rhein, Germany. Eng. Pat. 12,940 (Second Edition), May 25, 1897.

It has been found that when pure refined indigo is used in vats other than fermentation vats, *e.g.*, the hydrosulphite

vat, the colours are less fast to washing than when the ordinary commercial indigo is employed. It is stated that this is due to the absence in the refined product of the substance known as "indigo glue," which is necessary for the production of the fastest shades. This patent describes the addition to the vat of some substance which acts as a substitute for indigo glue. Such substances are proteid bodies, albumin, casein, gelatin, and gelatinous bodies, glues such as bone-glue, hide-glue, and fish-glue, silk-gum, and similar bodies, and also Turkey-red oil, British gum, starch, soap, and resin soap. These bodies can be used in a variety of ways; they may be added in solution to the vat, the goods to be dyed may be previously impregnated with them, or they may be mixed with the refined indigo before use.
—R. B. B.

*Discharge Effects on Indigo, Impts. in Production of.* B. Willcox, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 20,210, Sept. 2, 1897.

By printing a reducing discharge (instead of the customary oxidising discharge) consisting of zinc dust, sodium bisulphite, British gum, and acetine, on goods which have been dyed in the indigo vat, a pure white is obtained upon an indigo ground. Coloured discharges can be produced by adding to the white discharge such aniline colouring matters as are stable against the reducing agents. Thus, to obtain a yellow effect, Primuline is employed. Where basic dyes are used, for instance, Safranine T extra, Methylene Blue, Nile Blue A, Phosphine, &c., the indigo-dyed goods must be impregnated with a 2 per cent. solution of tannic acid before printing, in order to obtain shades which are fast to washing. The process is essentially as follows:—Print the white or coloured discharge upon the dyed goods, dry, steam for one hour at half an atmosphere of additional pressure, pass through a weak solution of sulphuric acid (to which is added two parts of tartar emetic per 1,000 parts, in the case of basic dyestuffs), wash, and dry.—J. E. H.

*Fixing Insoluble Colouring Matters on Fibres or Fabrics, Impts. in.* B. Willcox, London. From S. Wallach and Co. and C. Schoen, Mülhausen, Alsace. Eng. Pat. 13,916, June 5, 1897.

The material is first printed with the colour previously mixed with size or casein, and is then exposed to the action of formaldehyde vapour or solution. As an alternative process, formaldehyde or its ammonia or bisulphite compound may be mixed with the colour in addition to size or casein, and the colour fixed by drying above 80° C. For complete fixation 4 per cent. of formaldehyde (or 10 per cent. of the commercial solution) on the weight of size or casein is required.—R. B. B.

*Dyeing, Sizing, and Washing Hanks of Yarn and Slubbing, Impts. in Machinery for.* W. Woodcock, J. Longthorpe, and W. Barque, Copley, Yorks. Eng. Pat. 14,639, June 17, 1897.

Upon the dye vessels several rollers are mounted which may be revolved by worms and wheels or other means. These rollers carry endless chains connected together by cross-bars or laths. The hanks to be dyed are slung over the chains and cross-bars, and after they have been made to revolve in the liquor in one direction, they are by an arrangement of clutches made to rotate backwards in the opposite direction. Machinery is also provided for expediting the discharging and refilling of the hank-carrying rollers.—R. B. B.

*Paranitraniline Reds and Analogous Colours, Impts. in Treating, when Applied on Vegetable Fibres or Silk, to render them Fast to Light.* H. Koechlin, Rouen, France. Eng. Pat. 15,587, June 30, 1897.

Paranitraniline red and allied colours are very fugitive, but they are rendered exceedingly fast to light, and their beauty and brilliancy of shade are increased by immersing the material, in the form of hanks or in the piece (after the red has been fixed on in the usual manner) in a dilute solution of nitric acid. By adding alkali bichromate or a copper salt to the nitric acid solution, the colour is rendered still more fast to light.

The result is further improved if, after treatment with nitric acid, the dyed material is immersed in a boiling solution of calcium or magnesium acetate. Finally the best result is obtained by submitting the dyed material before or after immersion in nitric acid solution, to a treatment with β-naphthol, with or without the addition of soap, or even with soap alone. The shade can be rendered more bluish by steeping the dyed material, before or after acid treatment, in a solution of concentrated caustic soda containing glycerin to prevent mercerising.—J. E. H.

*Dyeing of Textile Piece Goods, Impts. in the, and in the Machinery and Apparatus employed therein.* A. H. Wardle, Leek. Eng. Pat. 16,741, July 15, 1897.

Above the dye vessel is an adjustable horizontal shaft, and attached to this is a skeleton reel or winder, which can be raised or lowered from or into the dye-bath. The bottom of the dye vessel is semi-circular or concave in form.
—R. B. B.

*Bleaching Textile Fabrics, Impts. in the Method of and Means for.* W. Hadfield, J. J. Sumner, and H. Hadfield, Furness Vale; and E. K. Dutton and Co., Manchester. Eng. Pat. 19,260, Aug. 20, 1897.

The woven goods in the open or rope (preferably) form, after singeing, are passed into a bath of hot water. They are allowed to stand about three hours in a stillage washed with hot or cold water, and drawn through a series of six becks or cisterns (more or less than this number may be used) which are placed in communication by means of conduits along which the material passes. Each beck is provided with a pair of cast-iron squeezing-bowls, and contains a weak boiling solution of caustic soda (2° Tw.) which may also contain about 3 per cent. of petroleum.

After passage through the becks, the material is washed first in warm, and then in cold water. The bleaching process is completed according to former patent (29,645 of 1897; this Journal, 1898, 45), or in the usual manner. The advantages claimed are that the usual kiers are dispensed with, thus lessening the handling of the material, and shortening the bleaching process. Again, the material, by being constantly in motion, and passed continually between the various squeezing bowls, which express the size and other matters from it, is left in a more perfect condition for bleaching.—J. E. H.

*Printing on Fabrics, Improved Method of.* T. F. Stimpson, Providence, Rhode Island, U.S.A. Eng. Pat. 3775, Feb. 15, 1898.

The fabric is twice printed, the first time with a black or other dark ground pattern, and afterwards with a pattern in a comparatively bright colour. The figures of the second pattern do not coincide with those of the first, and portions of the bright colour figures are obliterated or concealed to a greater or less extent by the dark pattern previously printed.
—R. B. B.

*Dyeing Vats, Impts. in or relating to.* J. and L. Budgens, Aix-la-Chapelle, Germany. Eng. Pat. 5475, March 5, 1898.

This improvement relates to dye vats, which consist of a dye-bath and a mixing chamber, the two being separated by a perforated partition. In order to prevent dyestuff from passing into the dyebath before it is completely dissolved, a movable cover is provided, by means of which the openings in the partition can be opened or closed at will.—R. B. B.

*Wood, Impregnating and Dyeing; Impts. in or relating to Apparatus for.* G. F. Lebioda, Paris, France. Eng. Pat. 7105, March 23, 1898.

The beams of wood are placed vertically in a cylindrical receptacle closed below by a perforated plate. The holes left free by the wood in the sieve plate, are stopped with plugs, and a thin layer of cement or other substance which hardens when cold, is poured over the plate. The liquid is conveyed under high pressure into the receptacle and forced through the wood, after which it passes out through the holes in the plate, which are under the wood. The liquid can also be forced from below upwards, through the wood, if desired. When impregnation is complete, steam is passed into the cylinder in order to soften the cement, &c., and the wood is then removed.—R. B. B.

*Bleaching or Dyeing Spanish Cane (Calamus Rotang), Bamboo, and other Plants which have a Silicious Coating; Impts. in Processes for.* R. C. W. A. Sieverts, Hamburg, Germany. Eng. Pat. 8029, April 4, 1898.

The cane is first steeped in a cold dilute alkaline solution, with or without a previous exposure to the action of hot steam, and afterwards treated with a suitable bleaching liquid, *e.g.*, potassium hypochlorite, or the alkali and bleaching agent may be applied simultaneously. The novelty of this process consists in the application of the alkali in a cold condition, whereby, it is claimed, the silicious coating is unaffected, and the cane is left in a suitable condition for bleaching or dyeing.—R. B. B.

*Mordanting Fibres or Fabrics, An Improved Process of.* F. J. Oakes, New York. Eng Pat. 8108, April 5, 1898.

The textile material, whether composed of either animal or vegetable fibres, or a mixture of both, is immersed in a solution of tannic acid, *e.g.* extracts of hemlock, chestnut, nut-galls, sumac, &c.

The surplus tannic acid liquor is squeezed out, and the material is worked in a neutral or acid bath of potassium bichromate, when a rapid change occurs, and chromium is deposited evenly and in an insoluble form upon the fibre. The material thus mordanted has great affinity for dyestuffs. —J. E. H.

*Apparatus for Dyeing and Similarly treating Textile Rovings.* R. Brandts, Munchen-Gladbach, Prussia. Eng. Pat. 10,205, May 1, 1898.

In this apparatus, the bobbin shafts upon which the textile rovings, are wound, are perforated. Upon each bobbin is fitted a copper cylinder having smaller perforations than the bobbin-shaft, so that the whole is air-tight. A bottom-plate having conical holes into which are fitted the corresponding conical ends of the bobbin-shafts, is filled with bobbins thus arranged, and lowered into a tank by means of a crane-chain and hook until it rests on two projections on the inside walls of the tank, near the bottom. The joint between the bottom-plate and tank is made air-tight with packing. The dye or other fluid is injected by a pump to the space below the bottom-plate, whence it passes through the perforations of the bobbin-shaft to the rove and away through the smaller perforations of the outer copper cylinder. The chief feature of this apparatus lies in the fact that when the fluid passes through the rove, it cannot escape freely through the holes of the cylinder, and thus a back pressure is set up which adds to the momentary pressure of the fluid throughout the rove. In this way, spots and unevenness are prevented, and risks of damage to the rove during the process are minimised.—J. E. H.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Manganous Sulphate, Water of Crystallisation of.* P. Schieler. Monatsh. fur Chem. 1898, 19, [5], 280—297.

Manganous sulphate crystallises from water with 7, 5, 4, or 1 mols. of $H_2O$ according to temperature. The form with 4 molecules is dimorphous. The supposed sulphates having 6, 3, and 2 molecules are non-existent.—J. O. B.

*Calcium Chloride Solutions, Electrolysis of.* [*Production of Chlorates.*] F. Oettel. Zeits. f. Elektrochem. 1898, 5, [1], 1—5.

*See under XI. A., page 769.*

*Perchlorate, Determination of, in Sodium or Potassium Nitrate.* N. Blattner and J. Brasseur. Bull. Soc Chim. 1898, 19, [12], 539.

*See under XXIII., page 795.*

*Formic Acid, Determination of, in presence of easily Oxidisable Organic Substances.* A. Leys. Bull. Soc. Chim. [3], 19, 472.

*See under XXIII., page 804.*

*Nitric Oxide, The Absorption of, by Ferrous Salts.* Thomas. Bull. Soc. Chim. 1898, 19, [10], 419.

*See under XXIV., page 807.*

*Perchlorate in Saltpetre, Estimation of.* F. Freytag. Zeits. für öffentl. Chem. 4, 326; Chem. Centr. 1898, 1, [23], 1203.

*See under XXIII., page 795.*

### PATENTS.

*Flowers of Sulphur, Impts. in and Apparatus for the Manufacture of.* R. H. Davidson and The United Alkali Company, Limited, Liverpool. Eng. Pat. 17,476, July 24, 1897.

Two pots are used, one in which the sulphur is melted, and the other in which it is boiled for the production of flowers of sulphur, the pots being connected by a tube, so that as fresh material is added to the outer pot, continuous flow may take place into the inner pot as the sulphur volatilises. The flues are so arranged that the sulphur in the outer pot is melted by waste heat from the fireplace in which the inner pot is set. The chamber containing the pot for boiling is so partitioned that, by means of a fan, the gases of the condensing chamber are caused to pass or circulate over the surface of the boiling sulphur.—E. S.

*Cyanides, their Derivatives, and other Nitrogen Compounds, A Process for the Economic Production of.* D. Lance, Paris, and R. L. E. de Bourgade, Enghien-les-Bains, France. [Under International Convention.] Eng. Pat. 26,326, Nov. 11, 1897.

On passing a mixture of nitrogen, hydrogen, and ammonia over intensely heated carbon, an acetylenic carbide of ammonium ($C_2(NH_4)_2$) is transitorily formed, in which the free nitrogen becomes fixed, forming ammonium cyanide $NH_4CN$, which volatilises. To obtain the maximum yield, it is necessary that an excess of hydrogen should be used.

Instead of pure hydrogen, hydrocarbons resulting from the distillation of organic matters may be used, and nitrogen as in the air. In practice, a mixture is made in a gas-holder of 2,000 litres of hydrocarbons with 200 litres " of nitrogen of the air," and this mixture, as well as 80 litres of ammonia gas are pumped into a mixing cylinder provided with a stirrer, and after drying by passage over lime, the gases are passed through a cylinder containing carbon, heated to about 1,100° C. The effluent gases are led through an alcohol-absorption apparatus, in which the ammonium cyanide produced is retained, and the excess gases are collected to be reconstituted in composition and re-used.

To obtain sodium or potassium cyanide, the ammonium salt is decomposed by an alcoholic solution of soda or potash, when the desired cyanide separates as a crystalline powder. A process for obtaining Prussian blue is claimed, consisting in passing the ammonium cyanide as it is formed into a dilute solution of iron in hydrochloric acid, maintained at 60° C., the process being made continuous by further additions of iron and acid as the blue deposits. The unabsorbed gases are passed to a gas-holder.—E. S.

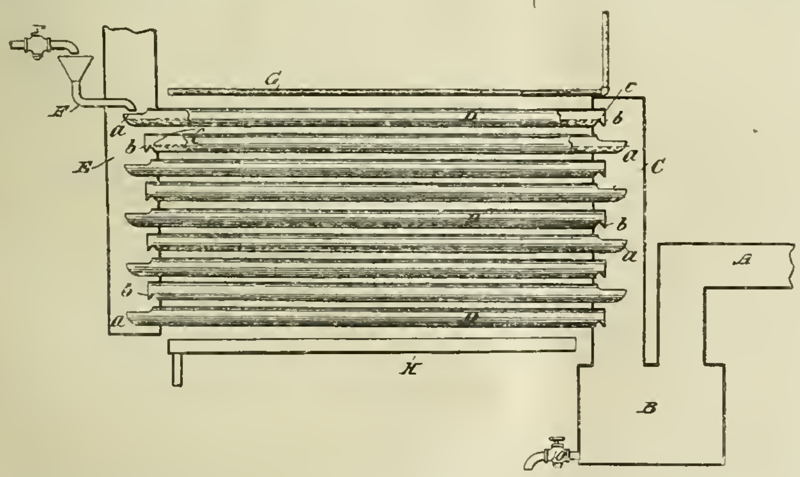
*Treating Solids with Gases, Impts. in and relating to Rotary Furnaces and Apparatus for.* P. Dunn, Birmingham. From P. Naef, New Brighton, U.S.A. Eng. Pat. 30,196, Dec. 21, 1897.

The furnace is primarily intended to be used with heating flues independent of the reaction chamber, so that the gases of combustion may not be mixed with gases arising in the decomposition of the ore or other material subjected to treatment. It may be used in the decomposition of various chlorides, such as magnesium oxychloride, for the production of chlorine, or for the production of sulphuric acid by treating sulphurous acid gas with metallic oxides, &c.

The apparatus consists of a revolving iron cylinder, lined internally with thin tiles or asbestos, next to which are tile flues, with a system of gas burners in combination with a supply pipe connected by a movable joint. The internal ore chamber is divided into compartments by longitudinal partitions, with firebrick radial ore-lifting partitions meeting at the central axis of the cylinder, and having the outer edges of the partitions seated in the longitudinal grooves. There is a stationary exit flue for the fire-gases registering with the ends of the heating flues, and an end plate at the discharge, having a central air opening for the furnace chamber. The ore or other material is supplied to the furnace by a mechanical feed. The discharge openings are preferably fitted with the automatically controlled covers described in Eng. Pat. 2736, 1895 (this Journal, 1896, 359). —E. S.

*Acids and similar Substances, Apparatus for the Manufacture of.* P. A. Newton, London. From The Baker and Adamson Chemical Company, Easton, Pennsylvania U.S.A. Eng. Pat. 7565, March 29, 1898.

The invention relates to "the manufacture of hydrochloric acid and similar substances which are formed by the absorption of gases by liquids." In the apparatus figured, a series of open horizontal tubes, D, are arranged one below the other, the ends of which, on one side, enter the tube C, communicating with the cistern B, to receive the liquid acid, and with the pipe A, which conveys the hydrochloric or other gas to be absorbed; while the ends on the other side enter the vertical tube E, into which any unabsorbed gases pass. The tubes, D, are kept cool by water sprayed from the perforated pipe G, any excess of which is received in the trough H. Water for absorbing the gas is delivered into the top tube from the funnel and pipe, F, and overflows at the opposite end into the projecting trough of the next lower tube, and so through the series, the water flowing reverse ways in alternate tubes, the discharge of the saturated solution being from the lowest tube into the cistern B. A special feature of the invention is that each of the tubes D is provided at one end with a V-shaped upward projection, c, to secure the retention of the water to the level required, and the outer side of the dam forms a spout, b, from which the water flows from the top of the dam into the trough a of the next lower tube. It will be seen that each tube has a dam at one end and a trough at the other end, so that the flow is continuous throughout the series.—E. S.



*Alkaline Silicate, Solid but easily Soluble; Process for the Manufacture of.* F. Henkel, Dusseldorf, Germany. Eng. Pat. 9730, April 27, 1898.

According to the first process given, "six parts of silicate of sodium or potassium, as it is obtained from the furnace, and containing more or less alkali, are pulverised, intimately mixed with one part of water, and the mixture is then strongly heated for a short time until the water has disappeared and a homogeneous substance is obtained, which cakes to form a solid mass" that may be disintegrated or pulverised, and is easily soluble in cold water. The alternative process consists in dissolving the alkali silicate, as obtained from the furnace, by means of hot water or steam, and adding to the hot solution, "powdered sodium water-glass or powdered potassium water-glass." The mixture cakes, and may be disintegrated.—E. S.

*Acids and other Corrosive Substances, An Improved Apparatus for Distillation of.* P. A. Newton, London. From The Baker and Adamson Chemical Company, Easton, Pennsylvania, U.S.A. Eng. Pat. 10,132, May 3, 1898.

The distilling apparatus consists of a pair of stoneware pipes arranged horizontally and parallel to one another over the fire-space of a furnace, a series of glass tubes, similar to test tubes, being secured, by asbestos or otherwise, at their open ends, to openings in each pipe, so as to depend vertically into the furnace. The furnace walls are contracted above, to receive the tubes, the bottoms of which are thus shown in the drawings to be protected from the direct impingement of flame. Each stoneware pipe or acid receiver is provided with an inlet for the acid to be distilled, a vapour outlet for the distillate, and with an outlet near the bottom of the pipe for running out the undistilled liquid or tailings, since the apparatus is not intended for distilling the acid to dryness. It is a claimed feature of the invention that the vertically depending tubes shall be removable, so that in case of fracture any of them may be readily replaced. —E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Glass Test Tubes, &c., Alkaline; Warning Against.* C. Liebermann. Ber. 31, [11], 1818.

*See under* XXIII., *page* 795.

### PATENTS.

*Glass and other Articles or Materials, Impts. in the Production of Coatings or Films on, and in Apparatus therefor.* J. Wheeler, Ilfracombe. Eng. Pat. 12,017, May 14, 1897.

Tablets are formed from powdered birch bark, mixed with an oxygen-carrier, such as potassium nitrate, which, when burnt by slow combustion—the flames when they are first lit being immediately extinguished — yield fumes of "pyrobetulin" or of pyrobetulin anhydride, which when deposited upon the surfaces of glass or other articles give either, in the case of pyrobetulin, porous opaque films; or, in the case of pyrobetulin anhydride, such as are "reticulated transparent." The apparatus employed consists of a closed chamber having a cover below and centrally, to which is arranged a lamp, and having agitating apparatus furnished with a cylinder over the lamp for mixing the fumes together and with the air of the chamber, so that the fumes from the lamp will rise up within the cylinder and the cover.

The opaque film on glass is pervious to hydrofluoric acid, so that the acid may impart to it "a finely pitted translucent surface, such as to render it suitable for use as focussing-glass or other purposes." The transparent film offers resistance to the acid, and, deposited on various articles, secures them against dampness and the attack of insects and fungoid growths. When paper is to be filmed, to receive printed impressions or otherwise, asphaltum or dragon's blood is added in compounding the tablets. Lint may be filmed to enable it to act as a preventive to the formation of pus in wounds, and wound-dressing materials generally may be similarly treated. Also, finished photographs and prints may be filmed to heighten effects.—E. S.

*Glass-melting Plant, Impts. in.* E. F. W. Hirsch, Radeberg, Germany. Eng. Pat. 16,062, July 6, 1897.

A pot furnace and a tank furnace are combined in juxtaposition, the mixture of materials being introduced into and melted in one or more pots, and the molten "metal" flowing from the pots into the working tank. The apparatus as shown consists of a pot furnace separated by a wall, up to the level of the bottom of the pots, from a tank furnace. There are two rows of pots, the larger of which, and the farthest from the tank furnace, receive the mixture of materials through separate supply pipes. A side tube rises from the bottom of each pot and extends over the smaller pot next it, so that as the material melts, it flows into the smaller refining pots, and thence in like manner overflows on to a channel, whence it falls into the tank furnace. Both furnaces may have free intercommunication for the purpose of the radiation of the flame, or for leading the products of combustion from one furnace to the other; or a perforated partition wall may be provided between the two.—E. S.

*Kilns for Burning Earthenware and the like, Impts. in.* J. Lengsholz and C. Lengsholz, Porz, Prussia. Eng. Pat. 15,777, July 2, 1897.

The outer walls of the kiln are raised to a greater height than the summit of the arch, to support a thick layer of earth on the top. Tube pieces fit into a number of openings in the arch, so as to be flush with the under side, but projecting outwardly. Iron cowls of about twice the diameter of the tubes are set, one over each tube, in such manner that the surrounding earth lutes them air-tight. Each tube is valved, the handles to the rods controlling the valves being, when the firing is commenced and the valves closed, buried in the earth covering. When the baking is over, the valves are opened so that the heated air may pass into the relatively capacious cowls. It is claimed that this method of dry-damping permits of the use of "the heat stream out from the kiln for drying purposes," besides affording other stated advantages.—E. S.

*Glass-annealing Furnaces or Leers, Impts. in.* W. Preston, St. Helens, Lancaster. Eng. Pat. 19,303, Aug. 20, 1897.

The object of the invention is to provide a furnace or leer in which glass goods, and particularly plate glass, may be annealed by a continuous process. The kiln and graduated leer are arranged in the form of an L, and two receiving chambers, one on each side, are placed at the receiving, and hotter, end of the kiln, the cooling-off leer being at the other end. According to the third claim, the floor or bed of the leer is formed with longitudinal grooves or channels, in each of which rails are mounted on grooved rollers carried by levers or radial arms fixed on a series of rock-shafts arranged transversely in or beneath the floor or bed, the rock-shafts being coupled together to have a similar and equal movement, and having suitable means for oscillating them in both directions to raise and lower the rails, and therewith the glass lying on the floor or bed. The rails are connected at one end to a cross-head or bar, and are provided with means for moving them, and therewith the glass, over the rollers when lifted by the rock-shafts, and for returning them (the rails), but not the glass, when lowered into the grooves by the rock-shafts, so that the glass may be shifted step by step through the leer. There is also claimed an arrangement of worm-gearing for oscillating the rock-shafts in both directions.—E. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Continuous Kiln, Improved, for Baking Pottery or Burning Cements.* E. Gobbe, Jumet, Belgium. Eng. Pat. 15,217, June 25, 1897.

This is a continuous-firing gas kiln, consisting of a battery of chambers communicating with each other by means of flues constructed in the walls, allowing of the passage of the hot air from the bottom of each chamber to the top of the next. The gas is formed in producers, from which it passes successively into a collector, vertical columns, and horizontal distributing channels. Bells permit of any one of the chambers being put into communication with the gas, which enters through a series of mouths, forming burners. The floors of the chambers are provided with small openings, communicating either with the flue or with the adjacent chambers through the flues.—H. H. B. S.

*Kilns for Burning Lime and other Similar Material, Impts. in the Construction of.* J. Foster, Carnlough, County Antrim, Ireland. Eng. Pat. 18,046, Aug. 3, 1897.

A chamber is constructed according to the usual open vertical type common in lime kilns, the top being dome-shaped and leading to a second chamber of an inverted cone shape. Across this is constructed an arched wall provided with a central feed shoot, by means of which fuel may be fed directly to the centre of the kiln, and also a series of vertical chambers, whereby the material to be burned is fed evenly into the kiln and at the same time heated during its descent. There are also a number of annular shoots through the dome-shaped top, for feeding part of the fuel directly into the principal chamber.—H. H. B. S.

*Imitation Marbles, Stones, and Gems, An Improved Process of Making; applicable also for Hardening Stone and rendering it Impervious.* J. M. Danielli, Paris. Eng. Pat. 14,970, June 21, 1897.

Alabaster or any calcareous earth, or an artificial agglomerate, is taken as the basis of the material. It is calcined at a high temperature, and simultaneously saturated with alum, and is then reduced to powder. The appropriate colours being added, the whole is made into a paste and moulded. The moulded objects are polished and dried, and are then immersed while hot in a hot solution of borate of soda, sulphate of alumina and potash, and common salt. They are now again dried, exposed to the air for a few days, and immersed in a bath of melted pine resin, Dammar gum, Carnauba wax, and paraffin. Finally the articles are cleaned with carbon bisulphide or other solvent.—H. H. B. S.

*Hydraulic Cement, A New or Improved Manufacture of.* N. T. Ashton and J. Crompton, both of St. Ives, Cornwall. Eng. Pat. 17,419, July 23, 1897.

Twenty parts of granite, 1 part of serpentine, 10 parts of china-clay, and 100 parts of calcareous sand, are ground to powder, mixed to a slurry and burnt to a clinker, which is then crushed and ground to an impalpable powder.
—H. H. B. S.

*Cement, Artificial Stone, Preservative Composition, Concrete or the like, Impts. in the Manufacture of.* C. Heap and T. Oddy, both of Rochdale. Eng. Pat. 21,456, Sept. 18, 1897.

Solutions of chloride of magnesium and silicate of soda are mixed together, and the gelatinous precipitate produced is mixed with lime, silica, and an inert filling material.
—H. H. B. S.

*Artificial Stone, An Improved, and Method of Manufacturing same.* W. A. Oakley, Bondi, Sydney, N.S.W. Eng. Pat. 684, Jan 10, 1898.

Disintegrated silicious rocks, marls, shale earths, kaolins, sands or clays, in a powdered condition, are mixed with their natural fluxes or reducing agents, such as calcareous earths, alkaline earths, fluor spar, felspathic granite or clays, also in a powdered condition, and the mixture is then burnt so as to flux or chemically unite the ingredients. In some cases, the materials of these two series are found in nature already mixed, so that they only require fusing or chemically uniting by the aid of heat. For example, disintegrated felspathic granite or clay may be mixed with an ordinary aluminous earth or clay. On heating, the two materials will flux and form a stone. Should, however, the disintegrated granites or clays or aluminous earths be deficient in fluxing materials, it is necessary to add such material as according to the nature of the ingredients, would cause fluxing to take place. The mixed material may be used for making moulded articles, which may further be lined with plumbago or fire-clay, or they may be hardened by adding powdered charcoal or other reducing agent to the materials before burning.—H. H. B. S.

*Fire-proof Composition suitable for Building Purposes, An Improved.* H. Rous, Middlesex. Eng. Pat. 1209, Jan. 15, 1898.

3 LB. of size, 6½ oz. of liq. ammon. fort., and 13 oz. of a saturated solution of potash are dissolved in 5 galls. of water to which 4 lb. of washing soda, 3½ lb. of powdered alum and 1½ lb. of tungstate of soda are added. The whole is mixed with about a bushel of hay chaff and the mixture allowed to stand for 24 hours, after which about half a bushel of powdered grey lime is stirred in, the mixture allowed to stand for three or four hours and dried. It is said to be particularly applicable for filling in walls, ceilings, &c.—H. H. B. S.

*Wood, Impregnating and Dyeing, Impts. in or relating to Apparatus for.* G. F. Lebioda, Paris, France. Eng. Pat. 7105, March 23, 1898.

*See under* VI., *page* 761.

## X.—METALLURGY.

*Sulphide Ores at Broken Hill, Treatment of.* Eng. and Mining J. 1898, **65**, [23], 666.

IN the treatment of sulphide ores at Broken Hill, the system of mechanical separation of the zinc and lead sulphides has always been considered inapplicable on account of the intimacy of the mixture, but as none of the numerous fire and wet methods which have been tried, could be worked at a profit, the principal companies are now using and endeavouring to improve the first-named method of separation. The difficulty of separating the sulphides is increased by the fact that the relative percentages of lead, zinc, and silver continually vary, as also the proportions of silver occurring in the lead, and in the zinc respectively; and that there is not much difference in specific gravity between the blende, and the rhodonite and garnet which form part of the gangue.

The average ore contains about 22 per cent. of lead, 22 per cent. of zinc, and 15 ozs. per ton of silver. In the concentration system now employed, there is first produced an argentiferous lead concentrate sufficiently low in zinc to make it available for the ordinary lead furnace. There is also obtained about 25 per cent. of the original ore in the form of "zinc middlings," 5—15 per cent. as slimes, and the remainder as worthless tailings. The lead concentrate assays 55—70 per cent. of lead, 8—14 per cent. of zinc, and 20—30 ozs. per ton of silver; and the zinc middlings, 8—20 per cent. of lead, 25—40 per cent. of zinc, and 8—18 ozs. per ton of silver. The slimes contain 25—35 per cent. of lead, 15—20 per cent. of zinc, and 20—28 ozs. per ton of silver, but the formation of a large percentage of these should be avoided, as only one-half of the lead, one-third of the silver, and no appreciable portion of zinc are recovered. The "zinc middlings" are further treated to obtain lead concentrates and a useful zinc ore. They are first crushed and then the concentration effected in jigs of special construction, in which the ore is given a lateral shake in addition to the usual up and down motion. In these jigs the sieves are usually arranged with five "hutches," and the jigging so carried out that lead concentrates are obtained in the first three "hutches," and zinc middlings in the fourth and fifth, whilst the tailings pass over the tailboard. The amount of ore treated is about 7 or 8 tons per hour, with a consumption of 30 galls. of water per ton. The plungers make 150—160 revolutions per minute. The cost of concentration by the above system averages about 4s. per ton of ore, including the cost of the handling of the ore and products to and from the plant.—A. S.

*Limestone and Lime in Blast-Furnace Practice.* C. Cochrane. Read before Iron and Steel Institute, May 1898.

THE results are given of a comparison of limestone and lime (1896—97) employed as flux at a furnace 90 ft. high, of 31,659 cb. ft. capacity (as originally constructed), enlarged by wear to about 34,500 cb. ft., at Ormesby Ironworks, Middlesbrough. The chief points of difference noted were:—

1. Reduced consumption of coke and low percentage loss of carbonic acid when calcined flux was employed. The coke saved amounted to 1·03 cwt. per ton of iron. With regard to the loss of carbonic acid, 13·96 cwt. of limestone yielded 5·99 cwt. of calcined flux, and 5·04 cwt. of raw limestone, and thus, although the calcined flux was obtained from 13·96 − 5·04 = 8·92 cwt. of limestone, with a loss of 32·81 per cent. of carbon dioxide, yet its admixture with the 5·04 cwt. of raw limestone reduced the real loss of carbon dioxide on the 13·96 cwt. total of limestone to only 20·99 per cent.

2. Reduced waste of raw ironstone per ton of iron from 2·06 cwt. in the period when raw limestone was employed to 0·66 cwt. when imperfectly calcined flux was used. Over a period of 16 months, ending August 1896, while using partially-calcined flux, this average waste of ironstone per month was found to be 0·21 cwt., this being less than the average waste of the four months September and October 1896, and May and June 1897, to the extent of 0·45 per cent. The furnace was blown in April 1895, with an original capacity of 31,659 cb. ft., which in July 1896 had enlarged to 34,814 cb. ft. In January 1897 it was slightly reduced, to 34,320 cb. ft.; and in April 1897 possessed a capacity of 34,396 cb. ft. The gross loss or waste of ironstone was only 0·23 cwt. for the eight months of 1895, May to December, and 0·19 cwt. for eight months of 1896, January to August, so that the difference in capacity does not appear to account for the increase of the gross waste to 0·66 cwt.; but the low waste may probably be accounted for by the fact that during the period in question, the limestone was calcined to the extent of 27·37 per cent., while the furnace also enjoyed the advantage of new internal "lines." The increase of the waste of raw ironstone to 2·06 cwt. per ton of iron when raw limestone alone was employed, appears to be due to the circumstance that raw limestone descends much further down into the furnace than does partially-calcined flux. There is thus a large reduction of the cubical space of "boshes" in which perfect admixture of ironstone and lime can take place for perfect fusion into iron and slag. Where the flux is introduced calcined wholly or in part, the breaking up of the friable lime quickly commences, and becoming intimately mixed with the ironstone in a longer period of descent than can possibly be the case with limestone introduced raw, a more perfect chemical reaction ensues, with correspondingly less waste of ironstone.—A. S.

*Blast-Furnace Gas, The Use of, for Motive Power.* A. Greiner. Iron and Steel Inst. Spring Meeting, 1898.

AFTER reviewing the comparatively scant literature of the subject and referring to the practical trial of blast-furnace gas with an 8 horse-power gas engine in which only 4 horse-power was obtained until the gas passages and admission ports were enlarged to admit of a more rapid supply of gas, the author describes the recent successful six months trial at Seraing of a 200 horse-power engine specially constructed for the purpose. The gas from the blast furnace mains is led through three pairs of coke scrubbers to the engine or to a gas meter and thence to the engine. The latter has a single horizontal cylinder, 31·5 ins. in diameter, and a stroke of 39·37 ins., whilst the speed is 100 revolutions per minute. The 15 ton fly-wheel is 13 ft. in diameter and the compression in the cylinder is carried to 114 lb. per sq. in., the ignition being electrical and adjustable. The objections offered are many, the first being the dust which impregnates blast-furnace gas. At Seraing it amounts to over 12 grms. per cubic metre. The heavier dust, consisting chiefly of ferric oxide, is deposited near the furnaces, a lighter dust, becoming richer in silica, lime, alumina, &c., towards the stack, whilst about 2 grms. per cubic metre of very fine dust remains in the gas after every possible means of washing. This, with a 200 horse-power engine, means 88 lb. of dust per day, but practically the whole of it passes out again through the exhaust, and it is apparently not at all imperative that a thoroughly clean gas should be used in a gas engine, since a small engine ran four months with this gas without a clean-up of the cylinder.

A second objection is the corrosive action of the acid matters in the dust, but no corrosion has been observed, the lime, &c. also present, probably preventing it. The ignition appliance requires occasional cleaning, but not through corrosion, and the time taken for this compares favourably with that for cleaning and repairing a boiler. The third objection is the irregularity of the composition of the gas, but this is not so serious as might be expected, since the gas-engine accommodates itself to the variation, and there is no more trouble than if the gas is burnt under the boilers. If the gas becomes bad and non-inflammable, coal has to be used on the boiler-grates, and it could be used in gas-producers for the gas-engine. The great advantage of gas is its easy and cheap transmission to the spot where power is required.—A. W.

*Iron and Steel, the Solution Theory of.* H. Jüptner v. Jonstorff. Iron and Steel Inst. 1898, 1—38.

This is an attempt to apply the laws of solution to iron and steel, on the assumption that these metals at ordinary temperatures represent solidified solutions.

*I. Conditions of Solubility.*—The solubility of carbon in iron at 3,500° C. is 10 per cent. (Moissan), at 1,030° C. it is 1·5 per cent. (Royston), and at 700° C. it is 0·9 per cent. (Arnold); and the maximum percentage, 4·63 (Percy), of carbon found in otherwise pure iron agrees with the formula $Fe_{4\cdot4}C$, and does not, therefore, indicate the existence of a chemical compound. The absorption of carbon is affected by the presence of other substances. It is increased by manganese and chromium, but tables are given as evidence that in a series of ferro-manganese alloys, ranging from 0 to 90 per cent. Mn, and from 95·4 to 2·5 per cent. Fe, and from 4·63 to 7·5 per cent. C, the manganese and the iron are each accompanied by proportionate quantities of carbon: thus, if each part of iron present represent 0·04855 part of carbon, each part of manganese in any alloy will represent 0·078 (0·075 to 0·082) part of carbon (in the series examined). Similarly with a ferro-chrome series, if each part of iron represent 0·04855 part of carbon, each part of chromium will correspond to 0·113 (0·105 to 0·117) part of carbon. The saturating powers of iron, manganese, and chromium for carbon are therefore in the ration 1:1·6:2·4.

*II. Lowering of the Melting-Point by Carbon and Silicon.*—The molecular weight of the dissolved carbon and silicon is calculated in the case of certain alloys of known fusing-point, from the equations $M = \frac{Em}{t}$, and $E = 0\cdot0198 \frac{T^2}{w}$, where M is the weight sought, E is the molecular freezing-point diminution, *m* is the quantity of substance dissolved in 100 grm. of the solvent, *t* is the lowering of the melting-point, $T_c$ is the melting temperature of the solvent in absolute temperature, and w is the latent heat of fusion of the solvent. T is taken as 1,500 + 273 = 1,773° C. for pure Fe (Osmond) and 2,173° C. for Mn (Carnelley) and w is taken at 20 calories. The following inferences are drawn: In the absence of Mn and Si, the molecules of the dissolved C consist of at least 2·53, and probably of 3 atoms. With increasing percentages, either of Si or Mn, the average size of the (C + Si) molecule also increases as a rule. In the case of a silicon spiegel with 24 per cent. Mn and 13 per cent. Si, the average number of atoms per (C + Si) molecule is calculated to be 7·16.

*III. The Absolute Size of the Dissolved Carbon and Silicon Molecules.*—Dissolved in iron, it is calculated that the carbon and silicon molecules correspond respectively to $C_3$ and $Si_7$, but that the molecules of the dissolved Si increase in size much more rapidly with increasing percentages of manganese than is the case with carbon. There are not sufficient data for the determination of the molecular size of C and Si when dissolved in Mn.

*IV. Calculation of the Melting Temperature.*—Accepting that the molecules of C and Si contain, respectively, 3 and 7 atoms when dissolved in iron, and taking 3,112 for the molecular freezing point diminution for iron as solvent, the melting-point of pure iron containing 4·63 per cent. C, should be 1,100° C, and that of hæmatite pig-iron, containing 3·29 per cent. C and 2·45 per cent. Si, should be 1,169·5° C. Similar calculations for manganiferous irons cannot yet be made owing to the uncertainty which exists as to the size of the carbon and silicon molecules dissolved in such materials.

*V. Calculation of the Osmotic Pressure.*—For want of suitable observations, especially of the specific gravity of iron at its fusing point, this calculation can only be approximate. But taking the specific gravity at 7·47 (deduced from the linear expansion of iron from 0° C. to the fusing point), and using the equation $p = \frac{1000 \times S \times w}{21\cdot17} \cdot \frac{t}{T_0}$, where P is the required osmotic pressure, S is the specific gravity of the solvent, and the other letters are interpreted as in section II., the osmotic pressure of a 1 per cent. solution of C in Fe at the melting point is 335; whilst the numbers obtained from the theoretical vapour-pressure of carbon gas at 1,500° and 1,085° C. respectively are 296 and 274. The pressure is therefore approximately 300; and from this the osmotic pressure of a 1 per cent. solution of Si in Fe at the melting point is found to be 56. The osmotic pressure for the solution saturated at the melting point in the case of iron and carbon (4·63 per cent. C) would be 1,389, and in the case of ferro-silicon (2·38 per cent. C and 11·46 per cent. Si) would be 1,355; from which it would seem probable that the osmotic pressure of saturated solutions may, at the melting point, be independent of the nature of the dissolved substance and constant for the same solvent. The maximum osmotic pressures for saturated solutions of carbon in iron at different temperatures may be found by multiplying the percentage of carbon required to saturate the iron at each given temperature by the osmotic pressure of a 1 per cent. C solution at that temperature. Thus $P_{max.}$ at 3,500° C. = 40 × 768·4 = 30,736; at 1,100° C. = 4·63 × 279·6 = 1,294·5; at 1,030° C. = 1·5 × 265·3 = 398; and at 700° C. = 0·9 × 203·6 = 183·2 atmospheres. The sudden drop between 1100° and 1,030° C. seems to indicate a change in the molecular condition (formation of carbide) at that temperature.

VI. *Latent Heat of Solution of Carbon in Iron.*—From formulæ given, the heat of solution of 1 grm. of $C_3$ in iron is 1595 grm. calories, or for pig iron with 3 per cent. graphite, for 1 kilo. pig iron it would be 47·85 calories. But the difference between the latent heats of fusion of gray and white pig iron is 33 − 23 = 10 calories, instead of 47·85. The form of carbon ($C_3$) dissolved in iron cannot therefore be identical with graphite, and it is shown that 1,261 grm. calories are rendered latent in the conversion of $C_3$ into graphite. Hence the graphite molecules must consist of many atoms, an inference which is confirmed by the formulæ given to the various graphitic acids.

VII. *General Review of the Behaviour of Iron Alloys.*—Just as the crystals deposited from concentrated aqueous solutions of salts contain varying proportions of water according to the temperature at which they are formed, a 50 per cent. ferro-chrome and a chrome-steel with less than 13 per cent. of Cr, have been shown by Behrens and Van Linge (this Journal, 1895, **14**, 274), to deposit crystals having respectively the composition $Cr_3FeC_2$ and $Cr_2Fe_7C_3$, the former containing far less of the solvent (Fe and C) than the latter. Iron containing carbon behaves in cooling from fusion in a manner analogous to that of an aqueous solution (*e.g.*) of ferric chloride, and Roozeboom's researches with this latter solution (Zeits. Phys. Chem. **10**, 477), are quoted for comparison. A saturated solution of carbon in Fe, gives up 4·63 − 1·5 = 3·13 per cent. of graphite in cooling from fusion to 1,030° C.; and then from 1,030° to 700° C., 1·5 − 0·9 = 0·6 per cent. of carbon must again separate, this time, however, not as graphite, but as cementite ($Fe_3C$), and during the same period $Fe_3P$ and $Mn_3P_2$ separate in the form of small granules. Below 700° C. the $Fe_{24}C$ (with 0·9 per cent. C) becomes converted into two or three constituents, *viz.*, ferrite, cementite, and sorbite. With low-carbon steels, ferrite begins to separate at 700°—750° C. and the residue is the mother-liquor, which at high temperatures is martensite, and at low temperatures becomes a mixture of ferrite, cementite, and sorbite. Troostite is another constituent intermediate between ferrite and martensite. Campbell has found that the carbide has various polymers $C_nFe_{3n}$, and that those in which $n = 2, 3, 4$, or 5 are commonest; those existing at the highest temperatures being, of course, least complex. In steel, with less than 1·3 per cent. C, $C_4Fe_{12}$ predominates. Polymerisation appears to take place in the mother-liquor (martensite) and this constituent is considered to be a compound $C_nFe_{3n}Fe_{18n}$ (resembling a salt with water of crystallisation), which may occur as such or dissolved in an excess of iron. Little is known yet of the phenomena observable while the metal is passing through the zone of temperature, after that at which graphite separates, and before that in which iron carbide occurs. It seems probable that an alloy of carbon (not carbide) and iron may then exist, and austenite which, according to Osmond, can be recognised by the microscope in iron alloys containing more than 1 per cent. of C, when quenched from a very high temperature, may be such a solution of $C_n$ in Fe. This is confirmed by the extreme softness and yet richness in carbon of the material. If this be so, iron rich in carbon would, at the melting point, consist of austenite and graphite, possibly with martensite; and, as the temperature falls, martensite would gradually increase at the expense of austenite. With a 1·5—1·6 per cent. C steel, cementite would begin to separate at 1200° C., and at 700° to 800° C., all the austenite would have disappeared. Thus, at high temperatures a rich carbon-iron alloy would separate into two solutions, martensite containing dissolved carbide, which would separate by degrees as cementite; and austenite, which would deposit first graphite and then temper-carbon. A table is given showing graphically the supposed behaviour of various carbon-iron mixtures at all temperatures from 0° to the fusing point.

VIII. *Application of the Laws of Solubility to the Separation of Ferrite, Cementite, and Pearlite in Solid Steel.*—The result of the examination is that iron and steel contain polymeric carbides $C_nFe_{3n}$, depending on the carbon percentage and the temperature; any one sample of steel contains at high temperatures (and therefore in the hardened form at low temperature), also iron carbides of simpler constitution than those existing at low temperatures (or after slow cooling); and calculation shows for high temperatures (and for hardened steel), a molecular size for the dissolved carbide that increases with the carbon contents, at least in steels containing less than 1 per cent. C.
—W. G. M.

### *Allotropic Iron and Carbon.* E. H. Saniter. Iron and Steel Inst. Spring Meeting, 1898, 1—16.

The author publishes some more photomicrographs of the etched surfaces of iron obtained by his own method of etching at high temperatures with calcium chloride (this Journal, 1897, 801). He now increases the time of etching from 15 to 25 seconds. The structure of carbonless iron at a bright red heat loses its normal cubical condition and shows signs of crystals with definite angles, none of which, however, are right angles. This shows iron to be dimorphous, and supports Osmond's allotropic iron, but has no bearing on his hardening theory. With 0·1 and 0·18 per cent. of carbon the large grain structure is broken up, but there is still an indication of allotropy, whilst with more carbon the mass becomes amorphous until the acicular crystals of high carbon metal appear.

Comparing these results with the cooling curves of iron and steel, there is an agreement as to the expected duration of arrest due to allotropic iron and to its gradual reduction with increasing carbon until it disappears as a separate point. Apparently carbon is inimical to the presence of allotropic iron, since the allotropic arrest disappears with the displacement of the allotropic crystal indications, in these high temperature etchings, by the acicular structure. This supports the carbide theory of hardening steel. Further experiments show that small quantities of other elements also modify the allotropic structure, such as sulphur, phosphorus, and manganese. The author then replies at length to the questions and criticisms by correspondence which were brought forth by his previous paper on this subject.—A. W.

### *Gold, Aqueous Solutions of Metallic.* R. Zsigmondy. Annalen, 1898, **301**, [1], 29—54. (Compare this Journal, 1898, 669.)

The author now gives details for the preparation of a red solution of the so-called colloidal gold. The following method generally succeeds:—25 c.c. of a solution of crystallised gold chloride (hydrogen aurichloride), containing 0·6 grm. substance per litre, are diluted with 100—150 c.c. of distilled water and 2—4 c.c. of 0·2 N-potassium carbonate or bicarbonate added. After the slightly alkaline solution so obtained has been heated to the boiling point and the source of heat removed, 4 c.c. of a solution of 1 part of freshly distilled formic aldehyde (b.p. 97°—109°) in 100 parts of water are added a little at a time, but pretty rapidly, whilst the liquid is vigorously stirred. The reaction is complete in a few seconds when the originally colourless liquid assumes a bright red colour. A great deal, however, seems to depend on the purity of the distilled water used. Traces of phosphates especially are objectionable. Very often the liquid obtained has a violet or blue-black colour and a turbid appearance. Large quantities (2—3 litres) of the red solution cannot be prepared in one operation on account of the difficulty of adding the formic aldehyde sufficiently rapidly. Solutions prepared as above, and containing 0·005 per cent. of gold, can be boiled or preserved for months without undergoing any change.

By subjecting the solution to dialysis in a warm place (40°—50° C.), the foreign substances can be removed and the concentration of the gold increased to 0·12 per cent.

The author then proceeds to describe at length the properties of the colloidal solution of gold, and arrives at the conclusion that the gold is actually dissolved by the water and is not merely a suspension of very finely divided particles of gold or a solution of an oxide of gold.

Most mineral acids and salts precipitate the metal; agitation with mercury does not remove it from solution; fungi grow readily in the solution, &c. The absorption spectrum of the solution is also described and compared with those of ruby glass and thin films of gold. On subjecting the solution to electrolysis the gold is deposited on the positive electrode, but may be intercepted and deposited on a parchment partition between the electrodes.

In 1857 Faraday prepared similar coloured liquids containing gold, but considered that the gold was merely suspended in the form of excessively fine particles.

After the above experiments were completed the author became aware of Faraday's work, and discusses it in an appendix. He still maintains that the red liquid consists of allotropic or colloidal gold dissolved in water.

Another paper is promised, in which the relationship between the coloured liquids and the purple of Cassius, will be discussed.—J. S.

### *Tin, Action of Nitric Acid on, in the Presence of Metals of the Iron Group.* F. van Leent. Rec. trav. Chim. Pays Bas, 1898, **17**, 86. (Through Chem. Zeit. Rep. 1898, [17], 142.)

On treating tin in the presence of iron with moderately concentrated nitric acid and evaporating the residue to dryness, a substance soluble in water is left. The same result is obtained by treating the metals separately and uniting the products of the reactions. By adding to this solution twice its volume of concentrated nitric acid, a pale yellow precipitate results, which, when washed twice with nitric acid (sp. gr. 1·2), is completely insoluble in nitric acid. From this precipitate iron cannot be completely extracted either by long-continued washing or by repeated precipitation of the substance from aqueous solution. On long-continued drying *in vacuo* over lime, the solubility is greatly diminished. The aqueous solution of the substance which has been repeatedly washed with nitric acid gives a precipitate with nitric acid in excess, with dilute sulphuric acid, and with oxalic acid. A deposit also occurs on cooling the solution, in which particular it is distinguished from the solution of the product of the original reaction. With this latter solution, sulphuretted hydrogen gives a greenish-brown precipitate, potassium bichromate a yellow precipitate, and alkaline hydroxides a white precipitate incompletely soluble in excess of the reagent. The soluble tin compound has been found by the authors to consist of meta-stannic acid, and by boiling it with hydrochloric acid in excess, it can be converted into the ordinary modification. When kept *in vacuo*, the substance, consisting of metastannic acid with iron and nitric acid, constantly loses water and nitric acid, and it has been established that there is no definite relation between the amounts of nitric acid and iron. The sesquioxides of chromium and aluminium behave in an analogous manner when treated with nitric acid in the presence of tin, yielding a residue soluble in water; or, in other words, converting the stannic acid into a soluble modification.—C. A. M.

*Ternary Alloys, Constitution of.* G. Charpy. Comptes Rend. **126**, [23], 1645—1648.

When a fused mixture of tin, lead, and bismuth is allowed to cool, a pure metal separates first (which of the three is determined by their proportions in the mixtures), then an alloy of two of the metals (one of which is always that which first separated alone) and, lastly, a eutectic alloy of the three. Microscopic examination of the surface of a section of the solidified alloy, which has been slightly etched by weak hydrochloric acid, shows these three distinct portions. The author has examined the copper-tin-antimony, lead-copper-antimony, tin-lead-antimony, and zinc-tin-antimony alloys, as well as many binary alloys. He finds that in the case of binary alloys, a pure metal (or in some cases a definite compound of the two metals) separates first, and then a eutectic alloy of the two; while ternary alloys behave like the particular alloy quoted above, save that sometimes the alloy behaves as though it were a mixture of definite compounds of two of the metals, rather than of the metals themselves — the tin-copper-antimony alloys rich in copper, for example, behave as though constituted of $Sn, Cu_3$, $SbCu_2$, and copper. The natures and relative amounts of the successive deposits depend only on the composition of the original alloy, but their physical properties are influenced by many circumstances, especially by the rapidity of solidification.—J. T. D.

*Silicon in Steel, Note on Drown's Method of Determining.* G. Auchy. J. Amer. Chem. Soc. 1898, **20**, [7], 547.

*See under* XXIII., *page* 796.

*Manganese in Iron and Ores, Use of Hydrofluoric Acid in the Determination of.* A. P. Ford and J. M. Bregowsky. J. Amer. Chem. Soc. 1898, **20**, [7], 504.

*See under* XXIII., *page* 796.

*Iron from Manganese, Aluminium, &c., Electrolytic Separation of.* M. Engels. L'éclairage électr. 1898, **14**, 106.

*See under* XXIII., *page* 796.

*Bismuth, A New Method of Determining.* L. Vanino and F. Treubert. Ber. 1898, **31**, [9], 1303.

*See under* XXIII., *page* 797.

*Lead in Alloys, Determination of.* W. E. Garrigues. Eng. and Mining J. 1898, **65**, [26], 761.

*See under* XXIII., *page* 796.

## PATENTS.

*Gold and Silver Ores, Improved Method of, and Means for the Preliminary Treatment of.* C. Wetherwax, Best, N.Y., U.S.A. Eng. Pat. 14,392, June 14, 1897.

The ore is treated with steam at high pressure and suitable solvents, in a vertical digester with a sloping grid near the bottom and a discharge cock passing through the side of the vessel at the lowest point of the grid, and with a manhole at the top for introducing the ore and another at the bottom for removing ore-particles that pass through the grid. Tanks containing respectively the solvent and water are connected with an inlet pipe at the top, whilst an outlet pipe at the bottom is connected with a circulating pump, which may be used, if necessary. Steam is introduced below the grid, or the whole vessel may be heated by steam coils or external firing. At the top of the apparatus is an outlet connected with a worm condenser, which is only used at the end of the operation, and serves to condense noxious vapours. The solvent is circulated upwards over fine, and downwards over coarse, ore. The final discharge of the ore into an outside sluice with ripples is assisted by a steam jet placed near the discharge pipe.—W. G. M.

*Gold Ores, An Improved Process of Treating.* C. Wetherwax, New York, U.S.A. Eng. Pat. 15,674, July 1, 1897.

The ore is submitted to the action of a hot mixed solution of caustic soda and sodium silicate for about an hour under a steam pressure of about 80 lb. per sq. in., previous to extracting the gold by the usual methods. This treatment is said to be advantageous with refractory ores, by liberating the gold from its combination or association with the other elements, and thus rendering it easy of attack by the solvent.—A. W.

*Gold and Silver, Impts. in and Apparatus for the Treatment of Ores or the like containing Gold or Silver, or both, for the Obtainment of the Precious Metals therefrom.* The General Gold Extracting Co., Ltd., London. From L. Pelatan, Paris. Eng. Pat. 22,256, Sept. 28, 1897.

The apparatus consists of a circular vat, on the bottom of which is a layer of mercury or an amalgamated plate, which constitutes the cathode. The anode consists of horizontal radiating arms fixed to a revolving shaft, suspended and worked from above. These arms, which have a lower surface of iron or carbon, are parallel with, and travel equidistant a few inches above the cathode surface. They are provided with projecting wooden or non-conducting pins, which, in slowly moving, carry the sludge gently over the surface of the mercury. Baffle plates on the sides of the vat are provided to prevent whirling and centrifugal action. The sludge contains the ore and water mixed with salt and a solvent, such as cyanide, and, after passing between the anode and cathode, is drawn off through a low-level tap. Other substances are added according to the ore treated, and in the case of argentiferous ores the temperature is raised to favour the formation of the chloride, which then dissolves in the cyanide previous to its reduction to metallic silver.—A. W.

*Dephosphorisation of Metals, An Improved Composition for the, and Process and Ingredients for producing the same.* A. Böttger, Düsseldorf, Germany. Eng. Pat. 14,909, June 19, 1897.

To remove phosphorus from cast metals, a mixture of sodium carbonate, lime, red lead, and petroleum is placed in the mould with the molten metal, or is fed, with the air-blast, into the cupola furnace containing the molten metal. —A. W.

*Slag or Cinder from Steel-melting Furnaces, Impts. in Utilising.* J. Riley, Glasgow. Eng. Pat. 19,338, Aug. 21, 1897.

Iron ore in small pieces, or iron scale, or purple ore, or blue billy, is mixed with the slag as it flows from the steel furnace. This mixture may then be used in the blast-furnace, so that a means is found for smelting both powdered ore and waste slag. Lime may be added with the ore if desired. Where acid-steel slag is to be treated, the ore should not be too silicious. If preferred, the steel-furnace slag may be solidified in the usual way and then re-melted in a cupola and mixed with the ore.—W. G. M.

*Ores, Impts. in Roasting and Drying, and Apparatus therefor.* G. F. Redfern, London. From P. Argall, Denver, U.S.A. Eng. Pat. 29,751, Dec. 15, 1897.

Four or more long, slightly inclined, but nearly horizontal, tubes are so mounted symmetrically around a common axis that they are parallel to one another. The tubes are lined with fire-clay, and are surrounded near either end by a ring resting on rollers, so that the combination may be rotated about its inclined axis. The mouths of the tubes open at either end into a short wide cylinder, which rotates with the tubes. The lower of these cylinders communicates with a furnace, in which liquid fuel is burned, and it is also provided with outlets for removing the roasted or dried ore. The upper cylinder leads to a smoke-chamber terminating in a flue, and has, supported within it, but out of contact with it, a bent chute, which passes through the roof of the smoke-chamber and is connected to a hopper, through which the raw ore is supplied to the calcining tubes. Running the whole length of each tube is a long box or hollow rabble, divided lengthwise into two compartments, of which the upper has numerous perforations along one side. These boxes are bolted firmly to their respective cylinders, and communicate at the flue end with a central blast-box and an axial air-supply pipe, passing through an iron sleeve outside the smoke-chamber. Air is forced by a fan through this tube and through the imperforate compartment of each rabble to the furnace end of the cylinders, and is there returned through the perforated compartment, which thus serves to distribute fresh hot air in the midst of the ore from end to end of the cylinder. The rabble rotates with the tubes, and is provided with a projecting ledge or flange which protects the perforations from being blocked by ore at that period of each rotation in which they are beneath the charge. The masonry of the furnace and the smoke-chamber is provided with elliptical castings, through which the ends of the rotating cylinder pass freely, an apron being attached to the latter to prevent escape of heated gases; and an arrangement is made by which the inclination of the tubes may be varied. In a modification of the furnace, the cylinder at the lower end is fitted with a conical prolongation communicating with the furnace, whilst the ore is discharged through one of a series of openings in the periphery, all, excepting those at the lowest point, being closed by a fixed band which nearly surrounds the cylinder, and within which the cylinder rotates. The tubes may be from 25 to 30 ft. in length for roasting operations, and from 15 to 20 ft. for drying ores, and the diameter, inside the lining, may vary from 20 to 26 ins. It is stated that, as compared with single-cylinder calciners, there is a higher heating efficiency (60 per cent. less fuel having been used), whilst, owing to the narrowness of the tubes, the heating may be more gradual, and also that, owing to the superior balance of the system, less power (by 80 per cent.) is required for driving. There are eleven claims.—W. G. M.

*Furnaces, Impts. in or connected with Smelting, for Metallic Alloys.* F. W. Minck, Berlin, Germany. Eng. Pat. 9717, April 27, 1898.

A movable hopper is suspended in the fireplace above the crucible in the ordinary pot furnace. In making alloys, the chief metal is first melted in the crucible, and the alloying metals then added through the hopper, the fumes, &c., being drawn off through the space between the crucible and hopper into the flue at the back.—A. W.

*Steel Armour Plate, Impts. in Art of Manufacturing.* J. T. Wainwright, Chicago, U.S.A. Eng. Pat. 30,463, Dec. 23, 1897.

Molten steel is treated by exhausting the dissolved gases (as described in Eng. Pat. 2293 of 1898), and is then manufactured into a case-hardened armour-plate.

—W. G. M.

*Perchloride of Iron, An Improved Process for the Recovery of, from the Solutions of Perchloride of Iron used for Etching Purposes.* [*Metals.*] O. Luppe, Munich, Germany. Eng. Pat. 18,417, Aug. 7, 1897.

Iron is put into the spent solution, whereby the copper dissolved in it is precipitated and recovered. The ferrous chloride solution remaining is then converted into ferric chloride for re-use, by treatment with hydrochloric acid and potassium chlorate.—E. S.

*Antimonial Ores, containing Precious Metals, A New or Improved Method of and Process for Treating.* N. W. Edwards, London. Eng. Pat. 15,791, July 2, 1897.

The ore is treated with cold solutions of calcium sulphide (about 7 per cent.) and washed, whereby the antimony sulphide is dissolved, leaving the gold in the residue in a condition suitable for extraction by the ordinary methods. The double sulphide solution is decomposed by carbon dioxide, which first precipitates calcium carbonate with production of sulphuretted hydrogen. After settling and filtering, a further treatment with carbon dioxide precipitates antimony sulphide in a commercial form. The sulphuretted hydrogen is passed into milk of lime to yield fresh calcium sulphide.—A. W.

*Brass and Silver, Manufacture of a Compound Metal of.* E. Martin, Paris. Eng. Pat. 938, Jan. 12, 1898.

Upon the surface of plates of brass is deposited a thin layer of copper, and then a coating of zinc, tin, or silver, and the whole is pressed between steel plates. Thin sheets or foil of silver are then laid on the plates of brass, and these are put together in series alternately with steel plates, and again submitted to hydraulic pressure whilst heated to about 200° to 300° C., whereby the silver and brass are united together to form sheets of the double metal.—A. W.

*Alloy for Dental Purposes, An Improved.* C. A. R. Samsioe, Stockholm. Eng. Pat. 9069, April 19, 1898.

An alloy of tin, 84 per cent.; silver, 15 per cent.; gold 0·5 per cent.; and platinum, 0·5 per cent., is more durable than tin, and is therefore better suited than that metal for bridge work, or for use without other addition for securing artificial teeth in place where no plate is used. The alloy is used in the form of cubes or balls, which, before use, are melted over a Bunsen flame.—W. G. M.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (*A.*)—ELECTRO-CHEMISTRY.

*Calcium Chloride Solutions, Electrolysis of.* [*Production of Chlorates.*] F. Oettel. Zeits. f. Elektrochem. 1898, **5**, [1], 1—5.

Some hitherto unpublished results dealing with the electrolysis of calcium chloride solutions obtained by the author confirm those recently published by Foerster and Bischoff (this Journal, 1898, 666). The smallness of the loss by reduction is ascribed to the formation on the cathode of the film (calcium hydrate), which acts as a diaphragm. The electro-chemical activity of this film, however, appears to have nothing to do with its thickness, since excellent results have been obtained with a very thin membrane, whilst another 2 mm. thick was practically worthless. The production, and what is more important, the preservation of a good membrane requires much skill and experience.

The following table is given as representing a normal run, during the electrolysis of a solution of calcium chloride (130—150 grm. per litre) to which a little milk of lime had been added to precipitate any magnesia present. The current density on both cathode and anode was 900 ampères per sq. metre; potential 3·8—4·0 volts; and temperature 75° C.:—

| Time from Commencement. | Current Efficiency. | Water Decomposition. | Reduction. |
|---|---|---|---|
| Hours. | Per Cent. | Per Cent. | Per Cent. |
| ¼ | 71·8 | 4·1 | 24·1 |
| ½ | 76·3 | 11·4 | 12·3 |
| 1 | 78·2 | 12·5 | 9·3 |
| 3 | 79·7 | 16·5 | 3·8 |
| 5 | 83·1 | 12·9 | 4·0 |
| 8 | 81·1 | 12·5 | 6·4 |
| 10 | 81·9 | 11·4 | 6·7 |
| 22 | 83·3 | 12·5 | 4·2 |

It will be seen that the current efficiency improves during the first few hours, and then remains nearly constant at about 80 per cent. The distribution of the remaining 20 per cent. between water decomposition and reduction is, however, very variable. In a precisely similar experiment, the electrolyte only being changed, the following results were obtained after a 10 hour run:—

| | Per Cent. |
|---|---|
| Current efficiency | 79 |
| Water decomposition | 9 |
| Reduction | 12 |

These results being considered very favourable, an attempt was made to utilise them by—1st, electrolysing the $CaCl_2$ to produce $Ca(ClO_3)_2$; 2nd, adding KCl to precipitate difficultly soluble $KClO_3$ and regenerate $CaCl_2$, and then continuing the process. A totally unexpected result was obtained. The presence of KCl in the solution caused the separation of thick crusts on the cathode, which gradually broke away, and at the same time free chlorine (not HClO) was evolved along with the hydrogen. The author considers that the character of the deposit on the cathode was such that it could not redissolve rapidly enough to fix the chlorine. The behaviour of the deposit on the cathode was very uncertain. At one time a very thin film was sufficient to prevent the evolution of any chlorine, whilst at another time the film would entirely disappear, and its place be taken by the above thick crusts, chlorine being at the same time evolved. An addition of more calcium hydrate sometimes temporarily improved matters; the amorphous crusts disappeared only to reappear again after some time.

Crystals of calcium oxychloride in the form of hard long needles were frequently obtained from the electrolyte on cooling, and the author considers that this substance plays an important part in the electrolysis. It is possible that the curious behaviour on the addition of KCl may be connected not with the formation, but with the solubility of the oxychloride in KCl, and that the cathode crusts are composed, not of calcium hydrate, but of insoluble oxychloride.

The electrolysis of calcium chloride solutions is subject to many other disturbing influences. On one occasion some copper chloride which had formed on one of the copper conductors, got detached, and fell into the decomposing cell, where it was converted into copper oxide, which then acted catalytically, decomposing the hypochlorite with liberation of oxygen. Many other substances, including the oxides of nickel, iron, and lead, act in the same way, so that it is necessary to exercise caution in judging the process from the results of gas analysis. This is clearly shown by the results which were obtained when copper oxide was present:—

| — | Current Efficiency. | Water Decomposition. | Reduction. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Apparent | 30·9 | 57·2 | 11·9 |
| Real | 73·3 | 14·7 | 12·0 |

The second or true value was obtained from the same data after determining and allowing for the oxygen which was evolved by the action of the copper oxide in the same time (3 min.) when no current was passing through the cell.

From the results of his investigation the author considers that the electrolytic production of chlorates takes place by two different processes, which occasionally operate together.

(*a*.) Production of chlorate through the hypochlorite. The products first obtained, *viz.*, alkali and chlorine, form hypochlorite, which, by the further action of chlorine and partially also by direct electrolytic oxidation, is then converted into chlorate.

(*b*.) Production of chlorate by the direct union of chlorine and oxygen whilst they are being simultaneously evolved at the anode.

In neutral solution (*a*) is the chief reaction; in strongly alkaline solutions (*b*) predominates; whilst in weak alkaline solutions the two reactions take place simultaneously, provided that the current density at the anode remains the same in all three cases.

During the electrolysis of calcium chloride solutions (*a*) is the principal reaction. Such solutions show a tendency to become slightly acid owing to the formation of free hypochlorous acid; whilst in the case of the alkali chlorides it depends on the position of the electrodes, and on the current strength whether the stationary condition is accompanied by the presence of a slight excess of free alkali or of free chlorine.—J. S.

### *Aluminium as an Electrode in Cells for Direct and Alternating Currents [Aluminium Throttling Cells].* E. Wilson. Proc. Roy. Soc. 1898, 63, 329—347.

When an electric current is passed through a cell containing an aluminium anode dipped into a solution of alum or sulphuric acid, the aluminium speedily becomes coated with a film of basic aluminium sulphate, and the current practically ceases unless the voltage be raised to about 22 volts, or more (compare this Journal, 1897, 807). This forms the basis of a method for transforming alternating into direct currents. If a cell, charged with a saturated solution of alum, be provided with an aluminium and a carbon electrode, then, on coupling up with an alternating current, the aluminium plate will alternately become anode and cathode, and thus the passage of the current in one direction, namely, when the aluminium is anode, will be "choked" or "throttled," provided that the potential of the alternating current is just equal to or preferably a little less than that which is necessary to overcome the resistance of the film of basic sulphate. By a suitable arrangement an alternating current can thus be transformed into a direct current.

In the first part of the paper the author deals with the behaviour of direct currents on such throttling cells, and comes to the conclusion that temperature has an effect upon the apparent high resistance of an aluminium plate and its film. In one particular case, for example, the potential difference between the electrodes fell from 30 volts to 3 volts when the temperature was raised from 13·5° to 70 C. This points to the necessity of circulating or otherwise cooling the electrolyte in practice.

The second part of the paper treats of alternating currents, and the conclusion arrived at is, that the choking or throttling effect takes time to develop, and is not fully developed with alternating currents of frequencies sixteen and ninety-eight complete periods per second. It can be increased by increasing the current density for a given film, and is greatly influenced by temperature. The metal aluminium with its film is suitable for use as the plates of condensers, if due regard be given to current density and temperature.

It might, in some cases, be found useful as an equivalent to a metallic resistance.—J. S.

## PATENTS.

### *Electric Batteries, Impts. in and connected with.* E. A. Jahncke, London. Eng. Pat. 7958, March 27, 1897.

The object is "to produce a constant battery with a maximum of energy." An example is shown which takes the form of copper-zinc couples in a solution of copper sulphate. "During the working, the sulphate of zinc produced rises to the surface of the electrolyte and runs into channels or gutters." As the zinc sulphate ascends, the copper sulphate descends. "In this manner," says the inventor, "it will be obvious that I am enabled to get rid of the sulphate of zinc almost as fast as it forms, which is of great importance."
—J. C. R.

### *Batteries, Impts. in Secondary.* D. D'Arbel, Paris, France. Eng. Pat. 3368, Feb. 10, 1898.

The electrodes are formed of metallic boxes, grated on their two large side faces and open at the top, which are filled with pastes composed respectively of minium or litharge mixed with sulphuric acid and water. Chloride of lead or chloride of lime dissolved in a solution of sodium chloride, sulphuric acid and water forms the electrolyte.
—G. H. R.

*Batteries, Accumulator, Manufacture of Active Material for.* H. Tobler, Muttenz, and J. H. Graeber, Bâle, both of Switzerland. Eng. Pat. 12,019, May 26, 1898.

The claim is for "a manufacture of active material, free from non-conducting binding material for accumulators, produced by mixing a paste of finely powdered lead oxide and graphite with dilute sulphuric acid, moulding the mass to the desired form, and alternately drying the material, and treating it with dilute sulphuric acid, until the mass is hard and coherent, so as not to require a supporting frame."
—J. C. R.

*Electric Batteries, Impts. in.* E. Edwards, London. From P. A. Emanuel, Aiken, State of South Carolina, U.S.A. Eng. Pat. 12,321, June 1, 1898.

The object is to provide means, which are shown, for heating the electrolyte, forcing the electrolyte (whether hot or cold) through the battery, by pressure of steam, gravitation, or by mechanical means, as by a pump, and keeping the elements of the battery the same distance apart at all times and under all conditions of use, whereby the internal resistance of the battery is kept constant. There are 17 claims. Claim I. "The method of producing a strong and substantially constant electromotive force in electric batteries by heating an electrolyte and causing it to flow under pressure through an electric battery and through the elements thereof, the said elements being kept at a uniform distance apart under all conditions."—J. C. R.

*Electrode Plates, An Improved Process for the Manufacture of, for Storage Batteries [Binding Material].* C. Marschner, Berlin, Germany. Eng. Pat. 18,029, July 31, 1897.

This process for the manufacture of electrodes, which is an improvement on that described in Eng. Pat. 12,250, 1896, consists in dissolving 1 kilo. of amber in 5 kilos. of epi- or dichlorhydrin, and mixing 1 part by volume of this solution with 5 parts by volume of alcohol, this mixture being used as the binding medium in forming the active material into a paste.—G. H. R.

*Electric Conduits, A New Material for the Manufacture of.* C. Hannay and P. J. Thom, Glasgow. Eng. Pat. 18,666, Aug. 12, 1897.

The diatomaceous material known as "Barbadoes earth" is moulded into shape with paper pulp, fired, and the porous product impregnated with asphalte, &c.—F. H. L.

*Chemical Compounds, Impts. for the Production of, by Electrolysis [Alternating Current].* J. W. Richards and C. W. Roepper, Pennsylvania, U.S.A. Eng. Pat. 9637, April 26, 1898.

An alternating current is passed between two electrodes immersed in an electrolyte, one or more of whose constituents forms alternately at each electrode, by electrolytic attack thereon, a compound or compounds partially derived from the consumption of that pole, and which are insoluble in either the electrolyte or the products formed at the opposite pole for the time being, which may be gaseous, and which compound or compounds are precipitated from the electrode of consumption.—G. H. R.

*Sulphides, Process for Manufacturing Metallic, Electrolytically.* J. W. Richards and C. W. Roepper, Pennsylvania, U.S.A. Eng. Pat. 9638, April 26, 1898.

The process consists in electrolysing a solution containing a thiosulphate salt between an anode composed of or containing the metal or metals whose sulphide is desired and any suitable cathode, thereby obtaining, as a precipitate, the sulphide of the metallic base present in the anode. The temperature of the bath depends on the nature of the sulphide being formed.—G. H. R.

*Plates for Electric Accumulators, Impts. in [Gas Chambers].* M. Bernstein, Berlin, Germany. Eng. Pat. 9778, April 28, 1898.

The improved electrode consists of two lead plates secured in a lead frame, each having dovetailed grooves formed on one side to receive the active material, and numerous small perforations communicating with the central chamber, into which gas may be conducted during the working of the cell, for the purpose of continuous regeneration of the plate.
—G. H. R.

*Plates for Electric Battery Purposes, Impts in the Art of Making [Spongy Lead].* A. C. Croftan, Chicago, U.S.A. Eng. Pat. 10,094, May 3, 1898.

Spongy lead plates are formed by forcing molten lead, by air pressure, through a series of small orifices, and receiving it on a horizontally-vibrating support at a temperature intermediate between its solidifying and melting points. The stringy spongy mass thus formed is compressed in parts, so as to produce comparatively solid strengthening ribs or frames.—G. H. R.

*Electric Resistances, Impts. in.* W. P. Thompson, Liverpool. From The Chemisch-Elektrische Fabrik "Prometheus" Gesellschaft mit beschränkter Haftung, of Bockenheim, Frankfort-on-the-Main, Germany. Eng. Pat. 10,585, May 9, 1898.

"Electric resistances, consisting of layers of copal varnish burnt on metallic surfaces as insulating material, and layers of precious metals burnt on these copal layers, as resistance material, the said copal layers being such as will not carbonise at the same temperatures at which metallic lustres or enamel colours are deposited."—J. C. R.

## (*B.*)—ELECTRO-METALLURGY.

*Zinc Chloride Solutions, Electrolysis of, and the Nature of Spongy Zinc.* F. Foerster and O. Günter. Zeits. f. Electrochem. 1898, **5**, [1], 16—23.

Starting with the assumption that the deposition of spongy zinc is caused by the presence of precipitated zinc oxide on the cathode, the authors have made some experiments which tend to confirm this view.

A zinc cathode was suspended in a perfectly neutral purified zinc chloride solution containing 54·6 grms. of zinc per litre, between two anodes of pure electrolytic zinc.

With a current density of 1·4 ampères per sq. dcm. a smooth compact deposit of zinc was obtained on the cathode for the first six or seven hours, but after 20 hours spongy zinc was being deposited, and the solution was turbid from the presence of suspended basic zinc chloride.

On filtering off the precipitate and inserting a new cathode, a good deposit was obtained during the first few hours The electrolyte then again became turbid, and, at the same time the deposit showed signs of becoming spongy. Schnabel (Handbuch der Metallhüttenkunde, **2**, 114) states that the formation of spongy zinc may be prevented by the addition of chlorine or bleaching powder to the solution. The authors, therefore, tried the method adopted by Mylius and Fromm for keeping a solution of zinc sulphate constantly acid. A small quantity of free chlorine was continually evolved in the cell from two small platinum anodes, which were connected with the positive pole of two accumulators, whilst the negative pole was attached to the large zinc plate, which thus served as cathode for both pairs of anodes. The formation of spongy zinc, however, could not be prevented in neutral solutions by this arrangement.

The oxygen of the air easily oxidises zinc in presence of zinc chloride, and a certain quantity of the oxide formed is dissolved by the solution forming basic zinc chloride. Deposition of spongy zinc only occurs when the excess of oxide begins to render the electrolyte turbid, and if this precipitation is prevented by the periodic addition of hydrochloric acid, good deposits of zinc can be obtained. The authors, therefore, recommend that the electrolyte should contain a quantity of free hydrochloric acid corresponding to a $\frac{1}{20}$—$\frac{1}{30}$ normal solution. The acid may be increased until it represents $\frac{1}{10}$ normal acid, but it should not be allowed to fall below $\frac{1}{200}$—$\frac{1}{300}$ normal.

If this slightly acid electrolyte is used in conjunction with the arrangement for liberating free chlorine, still better deposits of zinc are obtained. When the solution contains 0·2 grm. of free chlorine per litre, the hydrogen evolved at the cathode does not remain sticking to the electrode long enough to impair the surface of the deposit.

Although the main body of the electrolyte remains slightly acid, it sometimes happens that bad (spongy) deposits of zinc are obtained. This is accounted for by the fact that the layer of electrolyte in contact with the cathode becomes deprived of its free acid, but this difficulty may be overcome by sufficiently agitating or circulating the electrolyte.

On substituting anodes of commercial zinc for the pure zinc anodes, the zinc deposited on the cathode was found to be entirely free from arsenic.

When a 40 per cent. boiling solution of zinc chloride, is saturated with zinc oxide (the cadmium subsequently removed by treatment with pure zinc) electrolysed at 90° C., with insoluble (platinum) anodes, good deposits of zinc are obtained.

The quantity of zinc thus deposited, when compared with the amount of copper deposited by the same current in a copper voltameter was found to be in good agreement with Faraday's law. Ashcroft (Schnabel's Handbuch der Metallhuttenkunde **2**, 214—215), on electrolysing a similar solution, found that the zinc deposited in the first two hours was 19 per cent., during the next two hours, 37 per cent., and during the third two hours, 14 per cent. in excess of that required by Faraday's law, whilst in the last two hours the theoretical amount was deposited The authors conclude by discussing the nature of spongy zinc and the effect of adding various oxidising and reducing agents to the electrolyte. The addition of 1 per cent. of a perfectly neutral solution of ammonium persulphate to a neutral solution of zinc chloride furnished good deposits of zinc on electrolysis, whilst on the addition of 0·025 per cent. of hydrogen peroxide, the formation of spongy zinc was observed after electrolysing 10 minutes. —J. S.

*Copper. Analyses of Slimes deposited during the Electrolytic Refining of.* A. Hollard. Bull. Soc. Chim. [3], **19**, 470.

*See under* XXIII., *page* 797.

## PATENTS.

*Electric Furnaces and Processes of Treating Ores, Impts. in.* G. D Burton, Boston, U.S.A. Eng. Pat. 15,472, June 29, 1897.

Relates to methods of and apparatus for treating metallic ores in electrical furnaces. The specification supplies a mass of detail relative to temperatures, physical conditions of the ores the use of suitable fluxes, reducing agents, and the like, which are not usefully abstractable. It is accompanied by drawings of apparatus, and concludes with 25 claims. Claim I. is: "The process of expelling impurities and foreign substances from metallic ores containing them, by placing the ore in a partly-closed furnace or receptacle, and subjecting the ore while in said furnace or receptacle to the action of an electrical current of sufficient volume and pressure to eliminate the gases." II. . . . "and then increasing the volume of the current to a sufficient degree to fuse the ore" . . . III. . . . to desulphurise them, "and then increasing the current so as to melt and liquify the remaining metal contained therein," and so forth.

—J. C. R.

*Metals, Electro-deposition of, Impts. in Apparatus for the.* J Holloway, Westcombe Park, Kent. Eng. Pat. 17,857, July 2[illegible], 1897.

"Relates to the process of coating surfaces in which the surface is the cathode, whilst the anode is an instrument carrying the plating solution and moved over the surface. The apparatus forming the subject of this invention consists of a hollow vessel having a slot in its operative face, the anode being fixed centrally in the slot. The vessel is filled with the plating solution, the slot being covered with felt or the like to prevent the solution from escaping too quickly and to strain it. Preferably the vessel is made with a hollow handle, through which the conductor to the anode is led and to which a pipe can be connected to supply the solution."—J. C. R.

*Metal, Manufacture by Electrolysis of Sheets or Objects of Polished, Impts. in the.* L. E. Dessolle, Epinay-sur-Seine, France. Eng. Pat. 18,272, Aug. 5, 1897.

"In a process of direct manufacture by electrolysis of sheets or objects of polished metal: I. The method of preparation of the cathodes consisting of depositing on them a thin layer of a metal which is not attackable by the solution in which the cathode will be subsequently employed; subsequently saturating with hydrogen the surface of the thin layer of the metal, with which one has previously coated the cathodes in order to prevent the adherence of the metallic deposit to be afterwards made, and lastly, polishing the surface of the cathode in order to obtain deposits equally polished. II. The process, which consists in first coating the cathode with the precious metal, with which the produced objects shall be plated, and subsequently depositing the baser metal on said precious metal. III. A vat, having the cathodes suspended on cross-bars, &c. . . . moving the cathodes to and fro to disengage gas, &c. IV. "The preparation of cathodes of fusible metal or alloy and covered with a copper coating covered itself with nickel or any other metal not attackable by the baths to be subsequently used for the manufacture of hollow bodies, from which the cathodes are removable by melting."—J. C. R.

*Copper or other Metals, Impts. in and connected with the Electro-deposition of; on Rotary Cathodes.* W. E. Heys, Manchester. From The Electrical Copper Co., Ltd., Paris, France. Eng. Pat. 20,486, Sept. 7, 1897.

The impregnators, which are composed of or contain gelatinuous, albuminous, or analogous matters free from excess of grease, used in contact with rotary cathodes in electro-depositing baths, are rendered insoluble by treatment with alkali bichromates; for example, with a 5 per cent. aqueous solution of potassium bichromate, or other similar treatment.

—G. H. R.

*Metals or Metallic Alloys, Carbides; Process for the Extraction of Pure, and the Production of, by Electric Heat.* H. Aschermann, Cassell, Germany. Eng. Pat. 7423, March 26, 1898.

"By adding carbon in sufficient quantity to the mixture of an oxide and a sulphide, the carbon will absorb by formation of carbide that one of the two liberated metals which has the greater affinity for the carbon, while the other metal, according as it is fixed or volatile, collects at the bottom of the matter in fusion, or in the canals by which the gas escapes." The inventor introduces this as an important modification of his prior patent, Eng. Pat. 859 of 1897 (this Journal, 1897, 146), for the reduction of metallic oxides, inasmuch as it allows of obtaining at the same time carbides with the metal to be treated. "In principle, the modification consists in subjecting, to electric heating, a mixture of carbon with an oxide and a sulphide of metals having different affinities for the carbon. If, for example, a mixture which is approximately formed of the desired theoretic quantities of sulphide of iron ($FeS_2$), lime, and carbon, be treated in the ordinary electric furnace, metallic iron is obtained on one side; on the other side, carbide of calcium, which gives an acetylene as pure as the carbide prepared in the usual way, while the expenditure of electrical current is diminished at least 40 per cent."

The claim is: "A modification of the process patented by Patent No. 859 of the 12th January 1897, consisting in the addition to the mixture of oxide and sulphide of carbon, with a view to obtain at the same time carbide, &c."

—J. C. R.

# XII.—FATS, OILS, AND SOAP.

*Elæococca Oil.* F. Jean. Rev. de Chim. Ind. 1898, **9**, [102], 161—163.

Under this title the author gives a description of the properties and uses of Tung-oil (Japanese Wood-oil). (This Journal, 1897, 195; 1898, 671.) He states that it is derived by cold expression from the seeds of the *elæococca vernica*, a member of the *euphorbiaceæ*, which grows in China and Cochin China, 100 parts of the decorticated nuts yielding about 35 parts of oil. It is not to be confounded with the oleoresinous "wood-oils" of Cochin China or the oil of Gurjun. Of these wood-oils there are three qualities—white, dark, and black,—the densities of which vary from 0·960 to 0·966. Their drying properties are inferior to those of Tung-oil, nor do they solidify under the influence of heat.

The author has obtained the following results with specimens of Tung-oil imported into France:—Density at 15° C., 0·940; refractive index, 1·80°; flashing point, 260° C.; acidity as $H_2SO_4$, 0·784 per cent. Mention is also made of the remarkable drying properties of the oil, and the curious polymerisation which it undergoes on heating (*cf.* Jenkins, this Journal, 1898, 674).

On account of its siccative properties, this oil can be employed without preliminary oxidation in the manufacture of varnishes. When boiled with oxide of lead, and dissolved in turpentine, it yields an excellent varnish without the addition of any resin. It can also be used for rendering paper, &c. impervious. Paper thus treated is transparent and can be written upon with ordinary ink. The substitution of this oil for boiled linseed in oil paints would offer many advantages, especially where rapid drying is of importance, as for instance in the painting of ships and of metal work.—C. A. M.

### *Wool-Fat, The Composition of.* E. Schulze. Ber. 31, [8], 1290—1202.

Darmstädter and Lifschütz (this Journal, 1898, 253) obtained by precipitation of the purified alcohols of wool-fat with methylic alcohol, a compound which from its melting point and reactions they considered might be isocholesterol, notwithstanding the fact that it differed from that substance, as described by others, in its elementary composition (C, 81·59, and H, 11·79 per cent.), as well as in appearance and solubility. Pending their further investigation, the author reserves his opinion on the subject, but points out that the isocholesterol prepared by him (Journ. Prak. Chem. **9**, 325) contained 83·44 per cent. of carbon and 11·86 per cent. of hydrogen, figures in agreement with those obtained by Urich (*ibid.*, 325), and now confirmed by Winterstein. The melting point of the benzoate of isocholesterol given in the former paper as 194°—195° C., is said to have been too high, owing to the presence of a little cholesterol, and is now given as 190°—191° C. The author has been unable to isolate from wool-fat as many constituents as Darmstädter and Lifschütz, but attributes this to the varying composition of the fat. Thus he has found a great variation in the quantity of cholesterol, &c., in different specimens. For instance, of four samples examined by him and his co-workers, three on saponifying the portion readily soluble in alcohol, yielded a large amount of cholesterol, whilst the fourth yielded hardly any. Great variations were also observed in the relative proportions between the alcohols and the acids.—C. A. M.

### *Wool-Fat, The Cholesterols of.* L. Darmstädter and J. Lifschütz. Ber. 31, [8], 1122—1127.

This paper contains an account of the authors' further work upon the composition of wool-fat (this Journal, 1896, 206, 460, 548; 1897, 150; and 1898, 253). The semi-liquid fraction of the softer portion of the wool-fat alcohols separated by means of methylic alcohol, as described in the last communication, was dissolved in from two to three times its volume of ether and the solution cooled in ice for several hours. The white needle-shaped crystals which deposited melted at 76°—77° C., solidified at 72°—70° C., and did not give the characteristic reactions of cholesterol. To this substance the authors gave the provisional name of "Alcohol 2*a*." On evaporating the ethereal filtrate, taking up the residue in petroleum spirit, and diluting, there was a white flocculent deposit, which, on drying, left a substance resembling colophony. This was termed "Alcohol 2*b*." Finally, on evaporating the petroleum spirit of the filtrate, there was left the main bulk of the fraction, a neutral yellow, honey-like mass, amounting to from 50—54 per cent. of the alcohols of the softer portion of the fat. This substance, termed "Alcohol 2 *c*," could not be further fractionated. It was insoluble in water, but readily dissolved in the other usual solvents. At the ordinary temperature it was transparent, becoming more opaque and solid on cooling, though without crystallising. It gave all the reactions for cholesterol, although solid cholesterol could not be isolated from it. It did not show the spectrum of the so-called iso-cholesterol in the "cholestol" reaction. On dissolving a few mgrms. of the substance in glacial acetic acid and adding four or five drops of concentrated sulphuric acid, no heat was evolved, but a reddish-yellow tint was produced, which on standing became intense green and gave a characteristic spectrum with a small absorption band in the red between C and *d*, and a second fainter band at D. The colour lasted for from 10 to 15 hours, gradually passing through greenish-yellow to brownish-yellow, but the spectrum could still be clearly observed after 24 hours. Neither cholesterol, isocholesterol, nor the natural wool-fat gave this reaction. The authors arrived at the conclusion that this substance was a hydrated cholesterol. Cholesterol itself dissolved in it with difficulty when hot and crystallised out on cooling in transparent rhombic crystals.

This alcoholic body readily combined with organic acids and anhydrides to form esters. Heated in an open vessel with benzoic anhydride for two hours at 120° C., it formed at least three esters, two of which were solid and of high melting point, whilst the third was a clear yellow fatty substance. The mixture of the solid esters was readily soluble in acetone and ether, soluble with difficulty in alcohol, and insoluble in methylic alcohol. It dissolved easily in boiling glacial acetic acid, crystallising on cooling in microscopic hexagonal tables, which, on saponification with alcoholic potash, diluting with water, and shaking out with ether, yielded a white crystalline mass consisting of cholesterol and isocholesterol in the approximate proportion of 2 : 1.

The fluid ester was readily soluble in glacial acetic acid, and soluble with difficulty in alcohol and methylic alcohol. On saponification it yielded benzoic acid and the original "alcohol 2 *c*," giving the "cholestol" reaction and the characteristic reaction with sulphuric and acetic acids, as described above. It was noted as remarkable that the corresponding ester obtained by treating "alcohol 2 *c*," with benzoic anhydride under pressure at 190°—200° C., yielded, on saponification, an alcohol which did not give this test.

On adding an equal volume of acetic anhydride to the solution of "alcohol 2 *c*" in the mixture of acetic and sulphuric acids after it had assumed the green coloration described above, there was a liberation of heat, and after an hour the solution showed the characteristic bands of the isocholesterol spectrum in addition to those peculiar to the "cholestol" reaction. From this the authors inferred that isocholesterol must have been formed, as well as cholesterol, by the action of the dehydrating agent.

The intimate relationship existing between cholesterol, isocholesterol, and "alcohol 2 *c*" was further elucidated by the following experiments:—On boiling pure isocholesterol with glacial acetic acid containing a few drops of concentrated sulphuric acid or a little zinc chloride until a permanent reddish violet tint appeared, and adding alcohol when cold, a green solution was obtained which showed the spectrum of cholesterol and gave Liebermann's "cholestol" reaction. A parallel experiment with pure cholesterol yielded a solution giving an identical spectrum and the same colour reactions.

On heating a solution of pure isocholesterol in acetic anhydride containing sulphuric acid nearly to the boiling point, the resulting solution gave the characteristic tests for cholesterol.

Pure cholesterol boiled under a reflux condenser with strong alcoholic potash for several hours, and the solution diluted with twice its volume of 70 per cent. alcohol, and filtered when cold from the unaltered cholesterol, furnished on extraction with ether a small quantity of a yellowish substance giving all the reactions of "alcohol 2 *c*." In this way the authors succeeded in transforming from 20 to 25 per cent. of the cholesterol into the other alcohol.

The proportions in which these different constituents were found in the alcohols of the softer portion of the wool fat were : alcohol 2 *c*, 50—54 per cent.; isocholesterol, 11 per cent.; alcohol 2 *a*, 3—4 per cent.; alcohol 2 *b*, 6—7 per cent.; and non-cholesterol substances, which are being further examined, 24—30 per cent.—C. A. M.

*Cholesterol and "Phytosterin," Extraction of; from Animal and Vegetable Fats.* E. von Raumer. Zeits. angew. Chem. 1898, [24], 555—556.

The method recommended by Bomer (Zeits. fur Untersuch. Nahr. 1898, 31) requiring a large quantity (1200—1350 c.c.) of ether, the author attempted to reduce the volume of this inflammable solvent by proceeding as follows:—

50 grms. of fat were saponified with 100 c.c. of (Meissel) caustic potash, the resulting soap being evaporated to dryness, reduced to powder, and placed in a Soxhlet apparatus, where it was extracted with 50—75 c.c. of ether, plugs of cotton washed free from grease, &c. being placed above and below the layer of soap. The residue was saponified again with 10 c.c. of (Köttstorfer) alkali, evaporated to dryness with sand and re-extracted as before, during 2 hours.

The results obtained—0·7919 of crude "phytosterin" and cholesterol per 100 grms. of cottonseed oil; 1·3146—1·5256 grm. in the case of sesamé oil; and 0·2030—0·2176 grm. in the case of hog fat—showed that when the work is carefully done, a second saponification, &c., is unnecessary, the amount left behind after the first operation being negligeable.

As an alternative method, the dried soap from 50 grms. of sesamé oil was subjected to cold extraction in a separating funnel, by exposure for ½ hour to 100 c.c. of ether, this operation being repeated twice with fresh quantities (100 c.c.) of the solvent. In this manner, 1·3510 grms. of crude "phytosterin" and cholesterol were obtained per 100 grms. of oil, thus demonstrating the practicability of the method and the needlessness of employing large volumes of ether.—C. S.

*Fatty Acids, Separation of Saturated from Unsaturated.* K. Farnsteiner. Zeits. Untersuch. Nahr. u. Genussmittel. 1898, **1**, [6], 390.

*See under* XXIII., *page* 801.

*Wax, Examination of; by the Hot and Cold Methods.* K. Dieterich. Helfenberger Ann. 1897, 218.

*See under* XXIII., *page* 805.

*Fats and Oils, Cold Saponification of.* K. Dieterich. Helfenberger Ann. 1897, 124; Chem. Centr. 1898, **1**, [23], 1209

*See under* XXIII., *page* 805.

*Earthnut Oil (Arachis), New Method of Determining in other Oils.* F. Jean. Rev. de Chim Ind. 1898, **9**, [102], 162.

*See under* XXIII., *page* 801.

*Cod Oil, Spontaneous Combustion through.* Der Gerber, **24**, [571], 141.

*See under* XIV., *page* 776.

*Rosin and Rosin Oil in Linseed Oil, Note on the Estimation of.* L. de Koningh. Chem. News., June 1898.

*See under* XXIII., *page* 805.

PATENTS.

*Soap containing Ammonia, An Improved, and Process of Manufacture therefor.* W. S. Thomson, London. Eng. Pat. 7651, March 30, 1898.

The object of this invention is to manufacture a soap containing ammonia in such a way that it shall neither injure the perfumes, combine with the fats, nor escape from the composition through bubbles formed after the milling. A "compound," composed of 24 oz. of ammonia, 4 oz. of rectified spirit of wine, and 26 oz. of mixed essential oils, is prepared 18 hours before it is required for use. An ordinary soap is milled, coloured, and superfatted, &c., as may be desired; then 84 lb. are quickly mixed with 2 oz. of ammonia and 4 oz. of spirit of wine; next 32 oz. of the "compound" are added; the whole is milled again, and finally finished in the usual manner.—F. H. L.

*Purification of Oils, Syrups, and other Liquids, and for Deodorising and Decolorising Purposes; Impts. in the Manufacture of a Material for Use in the.* R. A. L. Hill, Wimbledon, Surrey. Eng. Pat. 7808, April 1, 1898.

Plastic clay, as free as possible from calcium and magnesium sulphates, is dried in a kiln, ground sufficiently to pass a ¼-in. sieve, and passed through a sifting mill to remove any dust. 32 galls. of the granulated clay are then mixed with 16 galls. of fine, hard-wood sawdust, previously steeped in one quart of crude petroleum and 1 quart of linseed oil. After 24 hours, the materials are thoroughly mixed and put into circular iron or clay retorts, heated to redness. After closing the retorts, the temperature is maintained for two hours or more until the contents are thoroughly carbonised. The substance is then taken out of the retorts, avoiding contact of air as much as possible, and allowed to cool in closed vessels.—N. H. J. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—*PIGMENTS, PAINTS.*

PATENT.

*Paint [Water-Colour], Impts. in the Manufacture and Use of.* C. E. Lee and C. F., A. L., and A. W. Lawton, New York. Eng. Pat. 24,178, Oct. 19, 1897.

By this process a water-colour paint is produced which becomes insoluble when it is dry, and presents greater resistance to rain, frost, and sun than ordinary linseed oil paints. Two compositions are required. The base in (A) consists of some compound or compounds of lead or (and) zinc, copper, barium, calcium, &c., which is (are) almost insoluble, but will react with an alkaline silicate to form perfectly insoluble bodies, and which is (are) preferably selected, so that 10 to 50 per cent. is in the form of an hydroxide or hydrated silicate. A typical mixture is composed of white lead, 10 lb.; zinc hydroxide or hydrated silicate, 5 lb.; well ground calcium carbonate, 20 lb.; with or without colouring matter. This is stirred into 10 or 15 lb. of water, then 15 to 30 lb. of a 3 to 5 per cent. aqueous and neutral solution of silica prepared by dialysis are added, and the whole is ground on a paint mill. When compounded in this manner, the silica solution gelatinises but slowly; and its function is only to hold the insoluble ingredients in place, without rendering the coating impervious to the liquids in (B). (B) is made by mixing 20 lb. of an 8 or 16 per cent. solution of water-glass with ½ or 1 lb. of a strong solution of zinc oxide in ammonia; stirring the precipitate till it dissolves again. If the surface to be painted is porous, B must be "bodied up"; and if the paint is to have a colour, it must be incorporated with a pigment. Both substances must be chosen so as not to induce premature insolubility; the "body" may consist of calcium carbonate, barium sulphate, &c.; and the pigment of iron oxide, red lead, blue or green ultramarine, "smalts," &c.

A and B are of course kept separate, and the former is stirred up before use. When a thin coating is desired, A is diluted with water to proper consistency, and is laid on first with a brush, or sprayed over the surface and allowed to dry; B is next applied. If a thick covering is required, A is not diluted so much, and the compositions are employed in the order B, A, B. In either case the alkali silicates in B, which hold the zinc silicate in solution, are decomposed by the hydroxides in A, and various complex treble or quadruple silicates are produced, causing the whole to become insoluble and impervious to moisture. When preparing the aqueous solution of silica, it is not necessary to dialyse out the sodium chloride; for if lead oxide is present in A, lead chloride and caustic soda are formed, which also take part in the final reactions.—F. H. L.

### (*B.*)—RESINS, VARNISHES.

*Resins, Acetyl Products and Acetyl Numbers of Some.* K. Dieterich. Helfenberger Ann. 1897, 39—45; Chem. Centr. 1898, **1**, [23], 1205.

It has been stated in the works of Tschirch and his pupils that a large number of resins contain oxyacids. It was found on carrying out acetylisation experiments, that all the resins examined were not completely soluble in acetic anhydride, but that some, as copal and dammar, were only partially dissolved. Both portions, the soluble as well as the insoluble were acetylised. The acetyl products are quite different from the original materials, and the acetyl numbers obtained are characteristic for the respective resins. In general, the acetyl numbers of resins are lower than those of fats and oils. All acetyl products decompose on keeping, even in absence of air, acetic acid being split off, and they are all soluble in alcohol, with the exception of the acetylised soluble portion of copal. The author gives, in a table, all the values found by him since 1896 of the acid, ester and saponification numbers of acetylised and non-acetylised resins.—A. S.

### PATENTS.

*The Production of Chemical Compounds from a certain [Tung] Oil or Oils or the Fatty Acids of same, Impts. in, and the Employment of such Compounds in the Manufacture of Drying Oils, Varnishes, Enamels, Lacquers, Antifouling Compositions, and the like.* S. G. Rosenblum and S. Rideal, London. Eng. Pat. 16,147, July 7, 1897.

The lead and manganese salts of the acids contained in tung oil (prepared by any of the processes adopted in similar cases) are far superior as driers for linseed oil to the resinates or linoleates of the same metals, 0·7 per cent. of the tung oil driers giving the same or better results than 2·5 per cent. of the older materials. The best method of employing the "tungates" is to dissolve them in the linseed oil, and then to "atomise" the solution in a jet of ozone (ozonised air or oxygen) at a temperature of 54° C. The same products can also be dissolved in turpentine and used as (coloured or plain) spirit varnishes, or lacquers; the aluminium salt can be employed to render wood, &c. fireproof; and the mercury salt as the active ingredient in antifouling compositions. The present specification, however, refers chiefly to the tungates as driers in conjunction with ozonised oil; and the sole claim, therefore, is for "dissolving the manganese or lead salts of tung oil or mixtures thereof in oil such as linseed oil, and atomising the solution in a jet of ozone. . . ."—F. H. L.

*Waterproof, A Composition to be used for Rendering Garments and other Articles.* G. F. Newman, Peel Forest, Canterbury, New Zealand. Eng. Pat. 11,264, May 17, 1898.

Boiled linseed oil, three gallons; castor oil, 1 gallon; turpentine, three pints; patent driers, three oz.; powdered sulphur, ¼ oz.; and yellow ochre or other pigment. Three coats are applied hot for the purposes indicated in the title.—F. H. L.

### (*C.*)—INDIA-RUBBER, &c.

### PATENTS.

*Compositions containing Casein for Electric Insulating and other purposes, Impts. in.* P. H. Hansen and J. K. Westengaard, Copenhagen. Eng. Pat. 14,272, June 11, 1897.

Fifty to 60 parts of casein, 20 to 35 of india-rubber, and 10 to 15 of asphalte, with or without pigments and loading material, such as ground bone, &c., are mixed together in hot water and moulded to the desired shape under high pressure. The rubber is employed as a 23 per cent. solution in benzene; and the casein is prepared by kneading milk curd or cheese for 15 minutes in water at 90° to 100° C. till the fat is removed, or else by the process given in Eng. Pat. 1550, 1897 (this Journal 1897, 450).

—F. H. L.

*Gutta-Percha, Impts. in or connected with the obtaining of.* R. J. Friswell and Brooke, Simpson, and Spiller, Ltd., London. Eng. Pat. 19,727, August, 26, 1897.

The leaves, seeds, or other parts of the gutta-percha tree are first crushed, if necessary, then heated and agitated under pressure in a solution of caustic soda until the softer organic tissues are decomposed and only the gutta-percha and the cellulose are left. The product is washed free from alkali, dried, and extracted with hot or cold toluene for 24 hours. The residue is also agitated with fresh portions of the solvent, if desirable; and the solutions are concentrated till a convenient quantity of the toluene has been recovered. The liquor is cooled, run into a large vessel provided with a stirring apparatus, and mixed with 1½ or two volumes of (roughly) 90 per cent. alcohol to precipitate the gutta-percha. The insoluble matter is either washed in spirit and water or dried in a current of air; and it is finally macerated in hot water, rolled and finished in the usual manner.—F. H. L.

*Gutta-Percha, Impts. in or connected with the obtaining of.* R. J. Friswell and Brooke, Simpson, and Spiller, Ltd., London. Eng. Pat. 12,728, August 26, 1897.

Compare preceding abstract. In this process, after the raw material has been softened in caustic soda, the gutta-percha is dissolved in carbon bisulphide; but as alcohol is of no use here as a precipitant, the solution is either evaporated or mixed with acetone to throw down the desired product. The gutta-percha is then finished as before.

—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Sicilian Sumac and its Adulteration.* Friedr. Andreasch. Der Gerber, **24**, [571], [572], 139—140 and 151—153.

In recent years it has become more and more difficult to obtain a pure sample of this, perhaps the most important tanning material known, owing to the fact that the practice of adulteration has become more and more common. This year, however, the Italian Government have passed some very stringent laws which it is hoped will tend to improve the product. In order to study the matter thoroughly the writer spent some months in Sicily by permission of the Austrian Government. It was found that the quality of the sumac depended upon (1), the position of the plantation, its height above the sea level, and distance from the sea; (2), the weather; (3), the composition of the soil; (4), the operations during the harvest, drying, powdering, and storing.

1. Sumac was introduced into Sicily by the Arabs, and is mostly planted in the south-east part of the province of Catania from which the best quality is produced. It should not be planted too far from the sea, and must be well exposed to the sun. It can be grown at a height of 700 or 800 metres.

2. Frequent changes in temperature or frequent rains, especially when the product is almost ready for harvesting greatly deteriorate the quality and strength of the sumac. If rain should come while the leaves are drying previous to powdering, it is sometimes sufficient to render the sumac completely worthless.

3. Sumac may be grown in a poor soil, which may be stony, or volcanic, or even calcareous, but it must be a dry soil and in such a position as to get as large a supply of sunshine as possible.

4. The sumac plant is always produced from shoots, never from seed. In the first year the plants are not cut, in the second year they are pruned to within two inches of the ground, the same in the third year when the harvest really begins. At harvest time the branches with the leaves are cut off close to the stem, and are laid flat upon the ground to dry; this takes from two to six days. The sumac plantations are generally mixed with plantations of Indian figs, beans, or even olives.

In Sicily there are two distinct varieties of sumac, the original *rhus coriaria*, and an inferior variety of the same known as *sommacco fimeneddu*. The leaves of the latter are larger, the plants stronger, but the percentage of tannin contained is much smaller, and of inferior color. When the leaves and twigs which have been laid upon the ground exposed to the sun are dry, the whole twig, leaf and stem are piled up in a heap upon a stone bed and roughly ground by stone mills. This forms what is known as "foglie sumac," and is worth from 8 to 8½ lira per 100 kilos, and in this state is usually sold by the growers to the larger sumac refiners and exporters. Some is sold in this condition direct to the tanners, but far the greater amount is ground a second or even a third time. By the second grinding it is powdered to a coarse powder, and by passing it through coarse sieves, the coarser stems, wood, &c., are separated from the finer sumac leaf. After this second grinding it is known under the name of "mezza macecinata," and is sold at from 14 to 15 lira per 100 kilos. Up to this point no adulterants are ever added as they would be easily detectable. Then comes the third powdering, which is still done with the stone mill. The sumac is left in until it has become a fine powder; it is then ventilated to remove the sand, which at this stage is often as high as 5 per cent. This product is then the sumac of commerce, and is known as ventilated sumac "polvero," and sold from 16 to 18 lira per 100 kilos. In this powdered form it is packed in sacks and ready for export, and at this stage the adulteration usually takes place.

It is important that during the whole of the grinding process, which is largely conducted in the open, that the weather should remain fine, as, if the sumac gets damp, a fermentation sets in by which the tannic acid is transformed into gallic acid.—J. G. P.

### *Bates and New Bateing Materials.* Der Gerber, 24, [570].

The bran bate (bran drench), like the dung bate, is still by some leather manufacturers erroneously looked upon as merely a means of ridding the hides and skins of lime. By the action of the dung bate the skins have been reduced from their swollen condition and have become thin and slippery. The intercellular substance having been dissolved out, the fibres and tissues of the hide lie closely one upon another, the pores and cavities thus becoming closed. The action of the bran bate is to open them up, and to prepare the skins for the subsequent process of tawing or tanning.

By the fermentation of the bran liquors, gas is evolved, not merely in the liquor outside, but also in the interior of the skins. This gas separates the individual tissues and opens up the fibre of the skin, cleanses the pores and other channels, so as to render the skin hollow and open, ready to suck up the tawing paste or tanning liquors. In practice the bran is covered with hot water and then put into a pit of water at a temperature of 30° C. The alcoholic fermentation which is induced soon passes on to acid fermentation with the generation of gas. This gas is apparent by small blisters forming on the skin, causing them to rise to the surface. If the process is allowed to go on too long the blisters become large, and are liable to burst, thus damaging the skin.

The action of the bran bate cannot be considered a chemical one, although if any traces of lime are left in the skin they are neutralised by the formation of the acid. Nor can the action of the bacteria be considered as important. It is to the dynamic action of the gas developed to which we must attribute the results obtained on the skins. The gas developing in the interior of the skin causes it to expand in all directions—length, breadth, and thickness, and at the same time expels any extraneous matters from the interior of the pores or hide cavities. The rising of the skins in the pit is usually looked upon by the practical man as an indication that the goods have been sufficiently long in the drench liquor. On no account must the blisters formed be allowed to become large, otherwise they are liable to burst or to separate the skin into two layers.

For good results a fresh drench should be made for each pack of goods, although a small portion of an old liquor may be used to start the fermentation. Old drenches are dangerous because of the presence of numerous bacteria which have a detrimental action upon the pelt.—J.G.P.

### *Cod Oil, Spontaneous Combustion through.* Der Gerber, 24, [571], 141.

Every year numerous fires take place in tanneries and leather factories the cause of which remains unexplained.

These mostly occur in the currying shops and especially in the rooms where the oiled leather is allowed to lie in pile. During the past winter in the Vienna Leather Industries' Laboratory, in the room which is used for experimental work on curried leather, some woollen cloths which had been used for oiling the leather with cod oil, and were therefore in a semi-saturated condition, took fire by spontaneous combustion, with little warning. Those standing in the room were only warned by the penetrating odour. A few minutes later the woollen cloths burst into flame.

In the currying of leather, the leather is heavily impregnated with cod oil, and with fats, and hung or laid in pile in heated rooms. Undoubtedly the same oxidation takes place in the leather as took place in the woollen cloths, and no doubt this spontaneous combustion caused by the oxidation of the fat may be put down as being the cause of many of the unaccountable fires in leather factories, especially in currying shops.—J.G.P.

### *Glue, Comparative Value of Commercial.* R. Kissling. Chem. Zeit. 1898, 22, [45], 150.

In this note, the author maintains the view he has already expressed (this Journal, 1898, 384), that more insight can be gained into the commercial value of any specimen of glue by determining the tenacity or gelatinising power of its jelly than by estimating the viscosity of its solution. In addition to the samples he has previously described, he quotes three others given in the annexed table, which show that whilst the viscosity rises slightly with the quality of the materials, the consistency of the jellies increases in far greater proportion.

| Variety. | Price in Marks per 100 Kilos. | Relative Tenacity. | Viscosity. |
|---|---|---|---|
| Leather glue........ | 92 | Over 100 | 2·80 |
| Powdered glue...... | 70 | 15 | 1·94 |
| Bone glue........... | 52 | Liquid | 1·88 |

—F. H. L.

## PATENTS.

### *Tanning, Impts. in or connected with.* A. Mario. Malonna di Campagna, Italy, Eng. Pat. 15,352, June 26, 1897.

The improvement consists in attaching each skin to a separate roller, moving in bearings at the head of the tanning vat, and weighting the skin by means of a bar attached to the lower edge, so that the skin is suspended vertically in the liquor. On setting the rollers in motion, the skins become wound thereon, the operation causing the one side of the skin to stretch, thus opening the pores and facilitating the absorption of the tanning materials. The same effect is produced on the other side of the skin when the movement of the rollers is reversed.—C. S.

### *Tanning (Rapid) of Skins and Hides, Improved Process and Apparatus for.* C. Michel, Paris. Eng. Pat. 16,804, July 15, 1897.

The objects of this invention are to provide a means for automatically ascertaining the strength of the tanning liquor in rotary tan-pits, and for replenishing same, according to requirements, without opening or stopping the machine; furthermore, to cause the hides, when lifted out of the liquor by the internal pegs, to fall back with a jerk, so as to increase the rapidity of absorption; and, finally, an improved tanning liquor.

To effect the first-named purpose, the rotating vessel is fitted at one end with a coiled pipe which, as the vessel moves, collects a portion of the liquor and conveys it through the hollow trunnion into a test glass provided with a tannometer or densimeter for registering the strength of the liquor, which is then returned to the vessel through a pipe debouching through the opposite trunnion. The automatic recuperation of the liquor is effected from a number of storage chambers fixed around the walls of the main vessel and provided with spring valves, the projecting stems of which come in contact with an external cam which depresses the valves and allows a portion of the recuperative solution to run into the vessel. The pitch of this cam can be altered so as to admit a smaller or larger quantity of solution as required.

The jerky motion is imparted to the hides by fitting the internal lifting pins with "mushroom" heads in place of the usual pegs, this arrangement allowing the hides to fall back suddenly into the liquor.

The tanning liquor employed consists of a "tannic juice or solution" of 1° to 5° B. strength to which is added ½ kilo. of ammonia and ½ kilo. of sodium carbonate for each 100 kilos. of hides, in the case of soft leather, or ½ kilo. of boric acid when hard leather is to be produced.—C. S.

*Whalebone, Improved Artificial.* W. A. C. Hunkemöller, Amsterdam. Eng. Pat. 12,599, June 4, 1898.

The whalebone substitute is prepared from animal sinews, bone gelatin, &c., by impregnation in a solution of metallic salts—preferably chrome alum—and (or) some organic body—*e.g.*, formaldehyde, tannin, &c.,—endowed with tanning properties. The material so treated is then dried, subjected to hot pressure, and cut into strips of suitable dimensions.—C. S.

## XV.—MANURES, Etc.

*Bone Meal, Experiments on the Action of the Phosphoric Acid of.* J. Kühn. D. Landw. Presse, 1897, **24**, 62 and 63.

Bone meal is a sparingly soluble manure, and consequently not very effective when applied for crops which occupy the land for short periods. Even the after-effect in such cases is inconsiderable, as, with ordinary soils, the phosphoric acid of bone meal remains, after the harvest, unchanged in the sparingly soluble, tribasic form. The effect of bone meal is, however, essentially different in the case of crops such as winter wheat, perennial clovers, &c., and the appropriative power of plants, such as winter rye, is of importance.

With heavy soils, and with crops of long vegetative periods, it is best to employ superphosphate; with good, warm, average soil, both steamed bone meal and superphosphate may be used, whilst superphosphate alone should be applied to cold loams, or cold sandy loams. In the case of good sandy soil superphosphate should not be used; both basic slag and steamed bone meal are suitable. There will, perhaps, in future, be more competition between degelatinised bones and basic slag, for light sandy soils, than has hitherto been the case.—N. H. J. M.

*Distillery Waste Waters, Utilisation or Disposal of.* Saare. Zeits. Spiritusind. 1898, **21**, [28], 249.

*See under* XVII. *page* 783.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar-Beet, The Growth of, and the Manufacture of Sugar in the United Kingdom.* Sir J. B. Lawes and Sir J. H. Gilbert. J. Roy. Agric. Soc. 1898, [3], **9**, 344—370.

In the year 1869 a factory was started at Lavenham, in Suffolk, for the purpose of manufacturing sugar from home grown beets. The venture, however, was not a success and only lasted until 1873. The quality of the beets, it is true, improved from year to year, but the farmers did not properly modify their rotations to secure a sufficient supply of beets for working the factory at a profit. The price of raw beet sugar was 25*l.* per ton in 1873, as compared with 9*l.* at the present time. About the same period, the authors of this paper conducted experiments on beet-growing at Rothamsted and found large variations in the crops obtained, according to the extent of the manuring, the sugar ranging from 1,704 lb. to 5,976 lb. per acre.

Since the year 1884 the duty in France has been levied on the roots and not on the sugar; consequently the efforts of the cultivators have been expended on producing moderate crops, with the highest proportions of sugar obtainable. By manuring with nitrate of soda, instead of sulphate of ammonia as formerly, and by careful selection of seeds, a very high degree of perfection has been attained.

M. Tisserand, formerly director of the Ministry of Agriculture at Paris, has issued a most valuable summary of the conditions of beet culture in France. The rotation of the crops is triennial: beetroot, wheat, oats. From 14 to 16 tons per acre of farmyard, or other natural manure, must be applied as soon as the previous crop has been removed, and well incorporated with the soil by the autumn. In the spring, 3 cwt. to 5 cwt. of superphosphate, 2¼ cwt. to 3¼ cwt. of nitrate of soda, and if necessary, 1¼ cwt. to 1¾ cwt. of chloride of potassium are put on. The seed is sown in rows, 16 inches apart, and when the young plants have developed four leaves, they are thinned out to a distance of 10 or 12 inches apart. The price of the roots varies with the locality and the strength and purity of the juice. The weather has a very considerable influence on the resulting crop, especially during the months of September and October, and it is of great importance that vegetation should be continuous and suffer no check during the hot days of July and August. In France, beet roots average 13 to 15 per cent. of sugar, and 9·5 to 12 per cent. is extracted, according to the purity and the method of working. The average crop during recent years, in France, is 11 tons 3 cwt. per acre, and in Germany, 12 tons 7 cwt. The average price ranges from 19*s.* to 21*s.* per ton, for a juice-density of 7° B., equal to about 14 per cent. of sugar. The capital value of beet lands varies from 40*l.* to 64*l.* per acre, and the rental from 22*s.* 6*d.* to 42*s.*

A pamphlet has recently been published by Mr. Sigmund Stein, which describes the prospects of sugar-beet cultivation in England in somewhat glowing terms, which are not confirmed by the opinions and experience of the authors.

On the whole, the authors are rather sceptical as to the suitability of England as a beet-growing country and, although satisfactory crops could, no doubt, be obtained in certain districts, the results on the whole country and on an average of seasons, are not likely to be profitable.

The perfected beetroot, grown on the Continent, is supposed to require an average summer temperature of 70° F., whilst statistics prove that the mean temperature for July, the hottest month in the year, is, in England, only 62·5° F., and for September and October is 50° F. and 43·2° F. respectively. These latter are the critical months, during which the beet matures and ripens and, in our country, October is nearly always rainy and often frosty. Nevertheless, in favourable districts such as the south and east of England, it is probable that, in the majority of seasons, a fair quality of beet for the manufacture of sugar could be grown, if proper care were taken to gather the harvest before the early frosts appeared. In this connection the authors strongly recommend the planting of the roots close together, whereby the luxuriance of foliage is limited, the purity of the juice increased and earlier ripening secured. Should the present revival of the subject of beet culture induce capitalists to invest money in the industry, Norfolk or Suffolk would probably be chosen as the most favourable district for the new enterprise. The climate is suitable and the soil not too light and not too heavy. It is of the highest importance to place the factories where a full supply of roots can be readily procured; taking a yield of 15 tons per acre, a factory, working 40,000 tons, would require a feeding area of about 3,000 acres, or, with biennial rotations 6,000 acres.

The authors conclude that, whilst sugar cultivation in England is doubtless possible, its sphere must of necessity be limited to certain favourable areas. Even if the continental sugar bounties are reduced or abolished, beet-growing can never be commercially profitable for the agricultural districts of Great Britain as a whole. Whilst protesting against the indiscriminate advocacy of beet-growing as a universal panacea for agricultural depression, the authors by no means wish to discourage an enterprise which would be of immense benefit to the districts in which it was successfully established.—J. F. B.

*Sugar Cane Juice, Note on the Purification of.* Hignette. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1136–1137.

On defecating juice with lime, the liquid between the scum or blanket and the precipitate appears clear and limpid, but still contains an appreciable quantity of suspended impurities. No efficacious means are known for filtering this juice; still less for scums and deposits, as mechanical filters are quickly obstructed, and begasse filters give a very imperfect purification.

The author has found at the sugar factory of San Pedro Alcantara in Spain that by special centrifugal machines, both mill and diffusion juices may be continuously filtered either hot or cold, irrespective of the quantity of lime used. In an experiment on 60 hectolitres of juice, treated with 0·2 per cent. of lime and centrifugalised cold, carbonated cold, and then heated to 90° C. and filtered, the co-efficient of apparent purity was raised from 85 to 93, whilst the ordinary process of defecation gives no increase of purity. The process appears to be equally applicable to both cane and beetroot juice.—L. J. de W.

*Soluble Starch, Properties and Preparation of.* A. Wróblewski. Chem. Zeit. 1898, **22**, [38], 375–376.

Soluble starch is the first product of the hydrolysis of common starch and is soluble in water. The solution may be filtered through porous porcelain. It does not reduce Fehling solution and is coloured a pure blue with iodine. All observations point to the existence of only one soluble starch, which may be regarded as the highest dextrin or as the first product of the breaking down of the starch molecule.

To prepare it on the small scale 20 grms. of Hoffmann's rice starch are triturated in a mortar with 100 c.c. of cold water and the cream poured into a flask having a capacity of two litres. About a litre of 0·5 per cent. boiling potash lye is then added, and the mixture boiled under an inverted condenser for 1½ to 2 hours when it becomes quite fluid and of a pale yellow colour.

After neutralising with dilute acetic acid, the soluble starch is precipitated with an equal volume of 95 per cent. alcohol, filtered and washed successively with 50 per cent., 95 per cent., absolute alcohol and finally with ether, and dried in a vacuum. It is thus obtained as a snow-white powder, containing 0·4 to 0·6 per cent. of ash, is soluble in about 3 per cent of water, and precipitated by 35 per cent. alcohol in the cold and by 45 per cent. when hot. The precipitation with alcohol is dependent on the quantity of ash, purer products being precipitated with greater difficulty.—L. J. de W.

*Starch, Soluble, No. II.* W. Syniewski. Ber 1898, **31**, [11], 1791—1796.

The author has further examined the soluble starch obtained by treating ordinary starch with sodium peroxide (this Journal 1897, 1029). He finds that it has a specific rotatory power of $[\alpha]_D = 195·3$ at 20° C. in 10 per cent. solutions. Solutions containing more than 12·5 per cent. of starch cannot be read on account of the deposition of a white precipitate having properties dissimilar to soluble starch. This substance is insoluble in water and when washed with water, alcohol, and ether, and carefully dried, it gave numbers on analysis, which showed it to be derived from soluble starch by the removal of the elements of water. The insoluble body always results when the concentration of a solution of soluble starch exceeds a certain degree and this explains how in the incautious preparation of diluted solutions of soluble starch a turbidity occurs.

When soluble starch is added to boiling water, clear solutions of high concentration may be obtained. These solutions become opalescent on cooling and soon set to a gelatinous mass identical with an ordinary starch paste. When warmed, the mass dissolves and sets again on cooling. A substance similar to that above described separates from the gelatinous mass.

The reversion product is probably identical with the substance resulting from the action of high pressure on potato starch (torrefaction dextrin?), the starch first goes into solution and on cooling separates in a gelatinous condition.

A solution of soluble starch when treated with baryta water yielded a product of the composition $C_{18}H_{32}O_{16}$ BaO. On acetylation a white derivative was obtained of the composition $C_{18}H_{25}O_{16}(C_2H_3O)_7$. An analogous benzoyl compound was also isolated. Soluble starch could not be regenerated by the hydrolysis of the acetyl derivative.

When inverted with hydrochloric acid under conditions which are described, an amount of glucose was obtained equivalent to 99·3 per cent. on the assumption that soluble starch has the formula $C_{18}H_{32}O_{16}$. The invertive action of water under pressure on soluble starch is slight, but a freshly prepared malt extract acting at 65° C. for 90 minutes produced 82·7 pec cent. of maltose.—J. L. B.

*Massecuites, Determination of Crystallised Sugar in.* H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1137.

*See under* XXIII., *page* 805.

*Invert Sugar, Estimation of Colorimetrically.* D. Sidersky. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898. **15**, [11], 1134.

*See under* XXIII., *page* 806.

*Sugar Analysis, The Correction for Unfermentable Reducing Substances in.* T. A. Glendinning. J. Fed. Inst. Brewing, 1898, **4**, 363.

*See under* XXIII., *page* 805.

## PATENTS.

*Sugar, Impts. in or connected with the Manufacture of.* W. Powell, Liverpool. Eng. Pat. 13,357, May 31, 1897.

The sugar in the moulds is liquored with a coloured liquid so as to produce loaf or lump sugar of a light and transparent shade or tint.—L. J. de W.

*Sugar, Impts. in the Process of and Means for Refining.* R. A. Chesebrough, New York, U.S.A. Eng. Pat. 8561. April 12, 1898.

The use of carbonised porous clay is claimed as a new medium for refining saccharine solutions by filtration. The natural clay, to replace bone-black, is found in Florida and Georgia in the United States of America; it is called "Florida fuller's earth" and "infusorial earth." Its valuable filtering property may be mainly attributed to its porous structure. All attempts hitherto to refine sugar by its use failed, because the water in which the sugar is dissolved reduces it to a mud. The invention is based on the discovery that, by carbonising the pores of the clay, after heating it to 320° F. to expel moisture, by mixing it hot with petroleum residue and heating slowly and carefully to about 800° F., the disintegration is entirely prevented.—L. J. de W.

*Adhesive Compounds, Impts. in.* A. J. Boult, London. From C. M. Higgins, New York, U.S.A. Eng. Pat. 11,753, May 24, 1898.

In preparing a mucilage by combination of dextrin with an alkaline solution and boric acid, it is preferred to so proportion the ingredients that the composition shall be nearly neutral, or only slightly alkaline, in any case much less alkaline than borax. To obtain a liquid mucilage, sufficient caustic soda (or other alkali) is dissolved in a gallon of water to bring it to a density of 1½—2 lb., and 7 to 7½ lb. of yellow dextrin are stirred into the solution, without applying heat, which would cause discoloration; 6 or 7 oz. of boric acid are then added, and the mixture is stirred until complete solution is effected, when it may be bottled for use. To prepare a dextrin paste, 3 oz. of caustic soda solution of 40° B. are added to 1 gallon of cold water, and from 5 to 6 lb. of white dextrin are stirred in, from 5 to 6 oz. of moistened boric acid being then added. After mixing, a suitable flavouring or antiseptic substance may be added. A white, semi-fluid pasty mass is thus produced, with a slightly alkaline reaction, very adhesive, and smooth-spreading under the brush. "The gallon mentioned in the formulas is the American gallon of 128 oz." It is preferred to add from 1 to 5 per cent. (by volume) of alcohol to the liquid mucilage, to increase its viscosity, and enable it to dry more rapidly when spread.—E. S.

### *Decolorisation and Purification of Liquids* [*Saccharine*], *Impts. relating to the.* P. Pessé, Marcq-en-Baroeul, France. Eng. Pat. 7561, March 29, 1898.

The liquid is digested with powdered animal-charcoal, at any suitable temperature, in a mixing apparatus to maintain the charcoal in constant suspension The mixture is then passed through a filter press, and the cake from the press dried by steam after being slightly washed with water, or even without having been washed. The charcoal still possesses considerable purifying power, without being revivified, and may be used a second, or even a third time for liquids more impure than that for which it was first employed. In this manner the purifying effect of the charcoal is more than doubled, because the first action, whilst effecting decolorisation itself, renders the syrup more susceptible to the action of the charcoal in the second operation.—N. H. J. M.

### *Sterilisation or the Disinfection of Waters of all kinds, Treatment of Sweetened Syrup, and other Analogous Applications, by means of Ozone, Air, or other Gases, Apparatus for.* H. H. Leigh, London, from La Société Anonyme pour les Appl. Ind. de l'Ozone, Paris. Eng. Pat. 6061, March 11, 1898.

The apparatus consists of an air-tight cylinder filled to a certain height with pieces of pumice, brick or other inert matter. The water is introduced at the top in fine jets, by means of a perforated feed pipe, whilst ozonised air, or other gas is introduced at the bottom. The cylinder is provided with perforated partitions at different heights to prevent the water from running off directly down the walls. The cylinders may be used singly or in series.

A modification is described in which the column is placed horizontally. In this case the receptacle is completely filled with inert matter, and is provided with baffle-plates to check the ozonised air as it passes through.—N. H. J. M.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### *Alcoholic Fermentation without Yeast Cells.* H. Lange. Wochenschr. für Brau. 1898, **15**, 377.

It has been urged against Buchner's views on the nature of fermentation, that the fermentative activity of the liquor expressed from yeast, suffers a considerable decrease on passing through a Berkefeldt filter, and that part of the action, at any rate, is therefore due to the living cells present in the extract. By counting the cells present and by calculating from the known mean weight of a yeast cell, the author finds that 100 c.c. of pressed extract of yeast, after filtering twice through paper, contains 0·09 grm. of yeast cells or about 0·1 per cent. He has treated sugar solutions, ranging from 30 to 60 per cent. in concentration, with quantities, varying from 0·1 to 2·5 per cent. of three different varieties of yeast and has measured the extent of the fermentation set up, by means of the carbon dioxide evolved. The yeasts employed were a bottom-fermentation beer yeast, a pressed yeast and a distillery yeast. The concentrations, at which the yeasts cease to set up fermentation, vary with the different varieties and the proportions used. The bottom-fermentation yeast had the least power of fermenting strong sugar solutions, whilst the pressed yeast had the greatest; but the small quantity of 0·1 per cent. of yeast, such as is found in Buchner's pressed extract, had in no case any considerable effect on solutions of 40 per cent. of sugar and over, and the extract filtered twice through paper, may safely be used for experiments on the fermentation of concentrated solutions.

The author has also prepared extracts from the three varieties of yeast, mentioned above, by pressure according to Buchner's method and has obtained them perfectly free from cells by passing them through a Berkefeldt filter. It is essential that the yeast used for this purpose should be quite fresh, as the pressed extracts from yeast, which has been kept, have little fermentative power, owing probably to the peptonising ferment, which Hahn has shown to be present in pressed yeast made from grain. One kilo. of yeast was treated in each case and yielded about 440 c.c. of pressed extract.

The extract from the bottom-fermentation beer yeast was a bright, strongly opalescent, yellow liquid, which contained 9·24 per cent. of nitrogen on the dry solids, and when allowed to act on a 37 per cent. solution of cane-sugar for $1\frac{1}{2}$ hours, liberated 1020 c.c. of carbon dioxide.

The liquor from the pressed yeast was only slightly opalescent, contained 6·9 per cent. of nitrogen on the dry solids and liberated 550 c.c. of carbon dioxide after $1\frac{1}{2}$ hours action on a 37 per cent. sugar solution. The extract from the distillery yeast was scarcely opalescent and showed 6·76 per cent. of nitrogen on the dry solids. The carbon dioxide liberated from a 37 per cent. sugar solution after $1\frac{1}{2}$ hours measured 450 c.c.

It appears from the above experiments, that the fermentative power of a pressed extract of yeast or the quantity of zymase present is, to some extent, dependent on the percentage of nitrogen, as Hayduck has shown to be the case with the fermentative power of yeast. The nature of the nitrogenous nutrition also will probably be found to influence the secretion of zymase.—J. F. B.

### *Yeast, The Formation of Glycogen in.* E. Kayser and E. Boullanger. Ann. Brass. et Distill., Feb. 25, 1898.

The method of determining the amount of glycogen formed under the varying conditions of the authors' experiments, consisted in counting the number of cells coloured dark brown on the addition of iodine. In order to ascertain the influence of aëration, cultivations of the same wine-yeast, were made in long tubes and in flat dishes. It was found that in the latter case less glycogen was formed, and also that that formed, disappeared more rapidly than when the yeast was cultivated in the tubes. The authors accounted for this on the ground that yeast increases more rapidly and to a greater extent at the surface than at the bottom of a cultivation, and that in consequence of the glycogen being distributed over a very large number of cells, the proportion of those containing glycogen was considerably less. Moreover since, according to Laurent, the presence of alcohol prevents the disappearance of glycogen, it was to be expected that at the surface of the cultivation where there is little alcohol, the glycogen should rapidly disappear. When the liquid contained from 10 to 15 per cent. of sugar, the quantity of alcohol produced was sufficient to prevent the glycogen from disappearing both at the surface and below. As a general rule, the more sugar there was, the more glycogen was produced, but when the solution of sugar was very concentrated, the production of glycogen was less rapid.

To determine the influence of different acids on the formation of glycogen, experiments were made with three varieties of wine-yeast, and three different acids—tartaric, malic, and citric—from which the following conclusions were arrived at:—(1.) The smaller the quantity of acid, the more glycogen is produced, and the more slowly it disappears. (2.) The production of glycogen is influenced by the nature of the acid present. Tartaric acid, for example, when present in large quantity, appears to have a strong restricting influence. (3.) The temperature of the fermentation and the nature of the yeast are also factors of importance. Hence there is a possibility of differentiating different species by their relation to the formation of glycogen. The fixed acids hinder the production markedly, but acetic acid has an opposite influence and prevents the disappearance of the glycogen produced.

Laurent showed that glycogen is formed at the expense of peptone, and this the authors confirmed by their experiments with various nitrogenous culture media, finding that a very large number of cells containing glycogen was produced. But whilst there was a very rapid production, there was an equally rapid disappearance. In the peptone cultivations, volatile acids were produced in much smaller quantity. When 15 per cent. of sugar was also present, the results in general were the same, but a solution containing one per cent. of peptone was then too rich, and so many yeast cells were reproduced that the quantity of glycogen was always very small.—C. A. M.

*Malt House, Ways and Means in the* [*Forced and Unforced Malts*]. E. R. Moritz, J. Fed. Inst. Brewing, 1898, **4**, 205—220.

From the point of view of output it is an advantage for the maltster to manufacture forced malts. The undesirable nature of these is, however, generally admitted amongst experienced brewers and scientific men, and inasmuch as they can be easily identified by analysis, there must be a set off to the apparent loss on reducing the output in accordance with the necessities of the non-forcing system. Thus, from a comparison of prices of the weights of forced and unforced malt, the fact is demonstrated that in the manufacture of forced malt, the losses due to rootlets and respiration are appreciably greater than when modification is attained more slowly, with less sprinklings, and at lower temperatures. These statements are supported by experiment.—J. L. B.

*Lager Beer, Higher Fermentation Temperatures in the Production of, Economic Advantage of.* F. Korte, Wochenschr. f. Brauerei, 1898, **15**, 366.

As a means for reducing the consumption of ice—no inconsiderable item in the production of (bottom-fermentation) lager beer—the author recommends, on the strength of his own practice and favourable experience, to brew one-half of the beer required on the "top-fermentation" system. This beer (fermented at a higher temperature, and, therefore, not necessitating an expenditure of ice) when about one month old, and having passed through a "secondary" fermentation, is blended with its own volume of new "bottom-fermentation" beer, and stored in the usual way at a low temperature. Some details, relating to the mashing and fermenting systems, are given.—H. T. P.

*Maltopeptone, Composition of.* O. Thomas and G. Warcollier. Ann. de la Brasserie et de la Dist. **1**, 172. Chem. Centr. 1898, **1** [23], 1202.

The composition of maltopeptone was found to be extremely variable. One sample gave on analysis :—48·16 per cent. of dry substance; 3·06 per cent. of acid, reckoned as tartaric acid; 14·42 per cent. of reducing sugar, reckoned as glucose; 21·62 per cent. of reducing sugar reckoned as maltose; 2·42 per cent. of total nitrogen, equivalent to 15·11 per cent. of albumin; and 7·80 per cent. of ash. The reducing sugars of maltopeptone are not completely fermentable—in one trial only one-half fermented. The sugars probably contain pentoses, formed by the hydrolysis of the cellulose-like constituents of the malt germs. The nitrogen of the maltopeptone occurs as follows:—0·138 per cent., as ammoniacal nitrogen; 1·331 per cent., as amido-nitrogen; and 0·949 per cent. as albuminoid nitrogen. The ash consisted of $K_2O$, 47·1 per cent; CaO, 5·13 per cent.; MgO, 1·52 per cent.; $H_2SO_4$, 18·75 per cent.; $P_2O_5$ 18·62 per cent; $SiO_2$, 0·45 per cent; Fe, trace.—A. S.

*Saccharine (Coal Tar) in the Brewery. Investigations as to the Preservative Action of.* Macheleidt. Wochenschr. f. Brauerei, 1898, **15**, 365—366.

The author's experiments show that comparatively large doses of saccharine are needed to inhibit the fermentative action of yeasts, bacteria, &c. The alcoholic fermentation of wort, *e.g.*, is not sensibly influenced by the addition to the liquid of $\frac{1}{5}$ to $\frac{1}{2}$ per cent. of saccharine; $\frac{3}{4}$ per cent. however decidedly checks, whilst 1 per cent. of saccharine completely arrests fermentation. The above remarks equally apply in the case of *Mycoderma* and *Penicillum glaucum.* The latter, indeed, grows, although very slowly, in wort containing 1 per cent. of saccharine. The acetic acid ferments (*B. oxydans* and *B. ascendens*) develop readily in wort plus $\frac{1}{5}$ per cent, not at all, in wort plus 1 per cent. of saccharine. Again, *sarcinae* grow rapidly in beer containing $\frac{1}{50}$ per cent. of saccharine, $\frac{1}{10}$ per cent. being required to check all growth.

It is clear, therefore, that saccharine when added to beers in the proportions (up to $\frac{1}{100}$ per cent.) obtaining in practice, can exert no preservative action whatever.
—H. T. P.

*Full Beers of Low Alcoholic Strength.* A. E. Berry, J. Fed. Inst. Brewing, 1898, **4**, 367–381.

In order to obtain a beer with a full taste and moderate degree of attenuation, it is necessary to increase the unfermentable carbohydrates to any desired extent, and also to maintain a proper balance between them and the fermentable carbohydrates. The difficulty of obtaining these results by brewing from malt alone is very great, and the author suggests the addition of an unfermentable substance of the desired properties to the wort in the copper. Various forms of unfermentable carbohydrates are described, as also are some attempts of the author to prepare these bodies.—J. L. B.

*Brewing, Botanical Problems, in.* H. Marshall Ward. J. Fed. Inst. Brewing, 1898, **4**, 334–355.

Among those fungi which attack wood, none is dreaded more than the *Merulius Lacrymans*, the organism of "dry rot." The term "dry rot" is anomalous, as experiments have proved that it can only grow in damp wood, and that if once the wood becomes really dry, the mycelium dies. The explanation of the term is found in the peculiar action of the hyphæ on the structure of the wood. By means of enzymes the hyphæ dissolve out the coniferin and cellulose constituents of the wood cell-walls, and leave behind a porous and extremely hygroscopic skeleton of so-called lignin, tannin, calcium oxalate, and other constituents, and, so long as the surrounding atmosphere is damp the skeleton retains its form. When, however, drier conditions prevail, the wood contracts and cracks, and owing to its deep brown colour—evidently due to an oxidation phenomenon—it looks as if it were scorched. This fungus can attack all common building woods, but cannot enter air dry timber.

Wortmann (Bott. Centralblatt, 1896, **78**) has shown that "corked wine" owes its peculiar flavour to *Penicillium*, and that *Mycoderma* oxidises the alcohol, and induces flatness, both these moulds finding the condition for attack and penetration of the corks in the damp air beneath capsules, in the fissures of the corks, borings of insects, &c. Miyoshis has proved that *Penicillium* can penetrate plant membranes, and in view of some of his own experiments on the behaviour of this mould to wood, the author suggests that it should be examined more particularly as regards its enzyme-secretion and penetrating power. The manner and conditions of excretion of enzymes by the living cells of fungi, including yeasts, bacteria, and higher fungi are discussed at some length, and a summary of the results of recent work on this subject is given.

Hugo Schulz has pointed out that every stimulus on a cell or a cell-tissue brings about some increase or diminution in physiological activity, according to the intensity of the stimulus. This was shown in the case of *Saccharomyces* by adding minute quantities of mercuric chloride, iodine, arsenious acid, &c., to yeast growing in a 10 per cent. glucose solution; for instance, an addition of 1 : 500,000 of mercuric chloride immediately raised the activity of fermentation, but in three hours the yeast had become used to the poison, and was working at the normal rate. Reference is made to Johannsen's work (*ibid*, 1896, **68**, 337) in which that investigator describes the effect of ether and chloroform narcosis on seeds, it being stated that the ripening of seeds is accelerated by weak doses of ether, the characteristic metabolism, condensation processes, &c. being hurried on. In this connection the author draws attention to Evans' work (this Journal, 1898, 480), in which an increase of the fermentative activity of yeast was found to be induced by pressure. It is suggested that this may be due to the increase of the narcotising effect of the compressed carbon dioxide.

The paper concludes with a discussion of the recent discoveries of Buchner, and questions of morphology connected with fungi, including the Saccharomycetes.—J. L. B.

### *Sarcina Infection in a Brewery, The Origin of.* F. Schönfeld. Woch. für Brau. 1898, **15**, [24], 321–325.

In a brewery coming under the author's notice the beer often became turbid, the cause of the trouble being usually the presence of sarcinæ. A systematic series of experiments showed that these were derived not from the use of impure yeast or from want of cleanliness in the fermenting tuns or pipes, but from the air, which in dry weather was heavily laden with sarcinæ derived from fields near the brewery, on which horse manure was spread. In the experiments the sarcinæ were readily isolated by making cultivations in sterilised and hermetically-closed flasks so as to avoid the development of acetic bacteria which took place when air was allowed to have access to the flask through a plug of cotton wool. By previously heating the beer to 40–42° R. in a flask, closed with cotton wool, the subsequent cultivations were quite free from yeast, and only in one experiment was a torula found. By placing a cylinder 5 cm. in diameter for 24 hours in the window ventilating the fermenting tun-room, 50 sarcinæ were collected, although the temperature of the air was below 0° C., and from this the author calculated that some four thousand sarcinæ must have passed through the window in that time. The earth all round the brewery, although far from the manure heaps, was also found to be swarming with them even after several days of frost. Since all cleanliness within the brewery was no protection against the infection thus derived from the outside, it was suggested that filtered air should be supplied to the tun-room, sarcinæ being most readily retained by an air-filter. But as this was not found practicable the author recommended, as the next best course, that the window of the tun-room should be kept altogether closed during the daytime, being only opened for a few hours in the early morning, while the ground was still moist, for the purpose of ventilation. This plan was carried out with good results, the infection of the beer gradually becoming less marked, and eventually disappearing altogether.—C. A. M.

### *Cider Making in France.* J. Soc. Arts, 1898, **46**, [2378] 674—676.

On account of the decline of the grape crop in France, increased attention has been paid to the cultivation of apples and the production of cider. The quality of the cider produced depends chiefly upon the proper fermentation of the must or crude juice of the apple; and the characteristics of the finished beverage depend upon the kind of ferment by which the transformation is effected. The apples should be ripe and fragrant, and should average from 2 to 4 per 1,000 of acidity. They should also contain some mucilage, a notable quantity of tannin (3 or 4 per cent.), and a large proportion of saccharine matter, say 15 per cent In gathering the fruit, the apples should be carefully protected from mud or other extraneous substances, and all rotten apples should be excluded. The fruit is crushed, and the resulting pulp pressed. The press consists of a circular cage with a bottom on which the pulp or mashed apples are placed, and then pressed by means of a screw—at first slowly, then progressively until the operation is completed. After draining, the pulp is taken out, macerated with water (2 or 3 gallons of pure, soft water to 100 lbs. of pulp), and again pressed. The crude juice or must is placed in barrels and allowed to ferment.

In the preparation of cider, it is essential that the fermentation be quickly established, and last only a short time, in order that the must *may* be clarified before the sugar has been entirely transformed into alcohol. The sugar which remains after the first active fermentation will still maintain a slow fermentation in the cider, which prevents the latter from becoming too acid, and being transformed into vinegar. The fermentation is chiefly influenced by the temperature, aëration, and the acidity of the crude juice. The best temperature to work at is 68—78° F., and aëration is effected by frequent stirring of the liquid and by drawing off a quantity at intervals and returning it to the barrel, the bunghole of which is preferably stopped with a little cotton-wool, so that air is admitted, but any injurious microbes excluded. If too many sour apples have been used, an acid must results, and fermentation is retarded. This is remedied, in some cases, by agitating the liquid with a quantity of wood-ashes, but since this treatment is said to result in a flat cider without colour, it is better to avoid an excess of acidity by a proper choice of fruit. French makers increase the fermentation of an indifferent must by adding a small quantity of must made from the best apples. Fermentation ceases after 10 or 15 days, when the cider, which forms a clear layer between the heavier sediment and the lighter less which rise to the surface, should have a specific gravity of 1·015 to 1·020.

The cider is drawn off into clean barrels, and after a certain time, undergoes a second fermentation, very much less energetic than the first. When carbonic acid is no longer disengaged, the barrels are completely closed.

It is stated that cider degenerates in quality, on keeping, when the barrel is not completely full; and in France, when the cider only partially fills the barrel, it is protected by what is known as "Noel's protector," which is practically the same as a simple covering or thin layer of oil.—A. S.

### *Wines, Micro-organisms of.* F. Bordas, Joulin and de Raczkowski. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1161—1171.

The empirical treatment to which sick wines are usually submitted is shown to be illusory. Thus, it is generally asserted that wines poor in alcohol and containing only a small proportion of tartar are liable to turn. The author has found that wines showing 12 degrees of alcohol and containing more than three grms. of tartar per litre have assumed, at the end of a few months, pronounced characters of a turned wine. From these wines two bacilli were isolated and studied in detail.

As the chemical treatment may render the growth of other ferments possible, and as it does not destroy the micro-organisms, they are always liable to resume development if any cause should modify the condition of the wine suitably. This shows the superiority of a bacteriological investigation, and although a summary examination may show whether a wine contains germs capable of altering it subsequently, a more complete study of the morphological and biological properties of the micro organisms will alone lead to the most economical method of preserving wines in good condition.—L. J. de W.

### *Wines, Boric Acid in.* P. Carles. Rép. de Pharm. 1898, **6**, 256, through Pharm. J.

It is stated that boric acid is present in notable proportions in many wines, more frequently however in white than in red wines. The addition of boric acid is partly to stop putrefaction of the substances—gelatin and isinglass—used for clarifying the wine. Sulphurous acid was used for many years for this purpose, but its disadvantages are that it deprives red wines of their colour, and may be readily detected by its odour. Boric acid is also used to check fermentation at a certain stage in the production of sweet white wines.—A. S.

### *Spirit, The Nature of the Action of Wood-Charcoal in the Purification of.* M. Glasenapp. Zeits. angew. Chem. 1898, [27], 617–621; [29], 665.

There are two views as to the nature of the action of the wood-charcoal used in the purification of spirit, one is that the purification is due to the direct absorption of the fusel oil by a kind of physical surface-attraction, the other attributes it to the chemical action, on the alcohol and its impurities, of the occluded gases, which are richer in oxygen than the surrounding atmosphere. The latter view is supported by the fact that, the lower the specific gravity of the charcoal, that is, the larger the size of the pores as compared with the mass, the better are the results obtained on filtration. Refiners always prefer to use charcoal, prepared from some light, soft wood, such as lime or willow, and having a specific gravity of about 0·15 when in pieces the size of a nut. Again, although spirit can be completely freed from fusel oil by rectification alone, the delicacy of the flavour and smell is vastly improved by filtration through charcoal, owing, apparently, to the formation of esters by oxidation.

In his experiments, the author has endeavoured to reproduce, as closely as possible, the conditions existing on the large scale. He used a commercial charcoal, in pieces the size of a nut, and passed the spirit at such a rate, that a volume of spirit, equal to the capacity of the filter, was filtered in four days.

The apparent fusel oil is estimated by Rose's method, by shaking with chloroform and measuring the extract, and the aldehyde is determined colorimetrically by Rosaniline bi-sulphite.

The free acid is found by direct titration, and the combined acid by the titration of the volatile acids, obtained after saponification of the esters by soda, evaporation of the alcohol and distillation with sulphuric acid, deducting the value found for the free acid.

The following table shows the effect of filtration on a pure spirit and on the same, with the addition of known quantities of impurities:—

| No. | Nature of the Spirit. | Alcohol per Cent. by Volume. | Apparent Fusel Oil contained in the Spirit by Volume. | Apparent Fusel Oil reckoned on 100 parts of Alcohol by Volume. | C.c. of $\frac{1}{10}$N Soda equal to the Free Acid per 100 Parts of Alcohol. | C.c. of $\frac{1}{10}$N Soda equal to the Acid combined in the Esters per 100 Parts Alcohol. | Aldehyde per 100 Parts Alcohol by Volume. |
|---|---|---|---|---|---|---|---|
| 1A | Purest brandy, free from fusel oil, unfiltered . | 40·01 | 0 | 0 | 0·33 | 0 | 0 |
| 1B | " " filtered through charcoal..... | 37·48 | 0·151 | 0·404 | 1·20 | 2·20 | 0·0025 |
| 2A | The same brandy, with 0·1 per cent. of isobutyl alcohol by volume, unfiltered. | 40·27 | 0·066 | 0·163 | 0·17 | 1·02 | Not estimated. |
| 2B | The same brandy, with 0·1 per cent. of isobutyl alcohol, by volume, filtered. | 37·76 | 0·279 | 0·740 | 1·31 | 2·50 | |
| 3A | Brandy, with 0·1 per cent. of amyl alcohol by volume, unfiltered. | 40·47 | 0·095 | 0·235 | 0·33 | 0·66 | |
| 3B | Brandy, with 0·1 per cent. of amyl alcohol by volume, filtered. | 38·22 | 0·225 | 0·588 | 0·08 | 3·67 | |
| 4A | Brandy, with 0·05 per cent. of normal propyl alcohol, unfiltered. | 40·12 | 0·009 | 0·022 | 0·33 | 0·30 | |
| 4B | Brandy, with 0·05 per cent. of normal propyl alcohol, filtered. | 37·90 | 0·169 | 0·446 | 0·63 | 3·70 | |
| 5A | Brandy, with 0·1 per cent. of acetaldehyde by volume, unfiltered. | 40·29 | 0 | 0 | Not determined. | | |
| 5B | Brandy, with 0·1 per cent. of acetaldehyde by volume, filtered. | 37·62 | 0·181 | 0·480 | 1·00 | 3·00 | |

It will be seen from the above figures, that considerable oxidation of the ethyl alcohol has taken place and that some of the products of oxidation are extracted by chloroform by Rose's method for the estimation of fusel oil. Brandy, perfectly free from the higher alcohols, yields, after filtration, an extract, which, if Rose's method were relied upon, would correspond to a fairly high proportion of fusel oil. Whether the higher alcohols, which were added in the other experiments, are also oxidised or absorbed by the charcoal, is not shown; but, as their taste and smell could still be recognised in the filtrate, in spite of the aroma due to the esters, the oxidation, if any, is probably only slight. In all cases, a large formation of esters is observed, owing to the combination of the alcohol with the acids produced. In experiment No. 3 the decrease of the free acid was probably caused by the alkali in the charcoal. The author has extended his researches to the refining of spirit on an industrial scale, with filters holding about 290 kilos. of charcoal, worked in batteries of four or six, the spirit being treated at a strength of 42 or 43 per cent. by volume. He has compared the composition of the crude spirit with that of the filtrate at various stages, when the charcoal is fresh and when its action is partially and totally exhausted, and has steamed out the exhausted charcoal and determined the composition of the fractionated distillates.

The presence of alkali or alkaline earth carbonates in the ash of the charcoal has a deterrent effect on the action, owing to the neutralisation of acids, which would otherwise form esters. In some cases it is found that a larger quantity of esters is introduced with the crude spirit than is afterwards recovered in the filtrate and steamed out from the char, and the cause of this apparent fixation of esters by the charcoal is under investigation. On steaming the charcoal, the first runnings are relatively rich in esters and aldehyde, the middle portions yield the purest alcohol, and the fusel oil is concentrated in the last runnings.

An increase of aldehyde, which has been put forward as an objection to the process, only takes place when the aldehyde in the crude spirit is very small and even then it is not considerable; when the latter contains a fair quantity of aldehyde, there is a decrease after filtration, probably due to further oxidation.

It is quite unnecessary to expel the air from the charcoal before use, with the idea of limiting the formation of aldehyde; on the contrary, the author recommends the previous aeration of the charcoal by means of a current of superheated steam and dry air, in order to increase its activity.

The charcoal has a very decided physical absorbing action on the esters and aldehyde, but practically none on the fusel oil, such as has been attributed to it by other authors. Since the result of filtration, is not the removal of fusel oil but the formation of pleasant smelling esters, filtration before rectification appears to be quite useless, because the esters are largely removed by the process of rectification, as well as the fusel oil, and a second filtration is necessary to restore the bouquet, at the expense of the alcohol.

If, therefore, absence of fusel oil is essential, it should first be removed by rectification and the purified spirit then filtered through charcoal.

It is found, however, that a finer delicacy of flavour can be obtained by filtering a spirit containing traces of fusel oil, than from one which is absolutely free from the higher alcohols.—J. F. B.

*Spirits, Detection of Repurified Denatured Spirit in.* Loock. Zeits. für öffentl. Chem. **4**, 316—319; Chem. Centr. 1898, **1**, [23], 1207—1208.

It is stated the denaturing of alcohol for trade purposes is effected by means of a mixture of two parts of crude wood spirit and one part of pyridine bases, which is added in the proportion of 3 litres to 100 litres of pure alcohol. The renaturing of such spirit is carried out by adding sulphuric acid to combine with the pyridine bases, then carbonate of lime, magnesia, &c., to neutralise the excess of acid, and afterwards distilling. It is impossible, however, even after repeated distillation, to obtain by this method a distillate quite free from pyridine, and since the peculiar odour of the latter is noticeable, even in quite small quantities, the employment of re-purified (re-natured) spirit is restricted to spirits, such as arrack or rum, which by their aroma, conceal the pyridine odour.

For the detection of re-purified spirit in spirituous liquors, the author makes use of the property which pyridine possesses of forming characteristic precipitates with certain metallic salts, as $HgCl_2$, $HgI_2.2KI$, $ZnCl_2$, $CdCl_2$, $PtCl_4$. The test is carried out as follows:—10—15 litres of the sample are treated with sulphuric acid and distilled till as much alcohol as possible has come over; then the residue, after neutralisation of the excess of acid with caustic soda, is again distilled. If pyridine be present, its unpleasant odour will be observed in the latter distillate. This distillate is then treated with a solution of $CdCl_2$ or $HgI_2, 2KI$. The formation of a precipitate is in itself no proof of the presence of pyridine, as the distillate from a spirit perfectly free from pyridine, would also give a precipitate under similar conditions. After the precipitate is completely deposited, which takes several days, it is filtered off, washed several times with cold water, and then heated, preferably in a closed test tube. If pyridine be present, its characteristic odour will be observed on removing the cork.—A. S.

*Rum and Arrack, Manufacture of. Do they contain Methyl Alcohol?* H. C. Prinsen-Geerligs. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1151—1161.

The arrack of Java, the only one found in European commerce, is distilled exclusively from the exhausted molasses of cane-sugar factories, which contains 25—35 per cent. of cane-sugar and 20—30 per cent. of reducing sugar. It is fermented with a special yeast obtained from rice straw (this Journal 1895, 47) containing two moulds, Chlamydomucor oryzae and Rhizopus oryzae. This yeast or "raggi" contains also two yeasts, Monilia javanica and Saccharomyces Vordermanni, which play an important part in the distillation of arrack, the quality depending on the relative development of these two organisms, the saccharomyces predominating in Batavian arrack, while that of the coast districts, of inferior quality, is fermented chiefly by Monilia.

In the manufacture of rum, the molasses is mixed with water and the vinasse from a previous distillation, so as to dilute it to 15° B. Defecation scums and damaged canes are also added, and before fermentation some dried sugar cane leaves are added to cause an etherification giving the rum its odour and characteristic taste.

The author could not find either in rum or arrack the least trace of methyl alcohol.—L. J. deW.

*Distillery Waste Waters, Utilisation [Agriculture] or Disposal of.* Saare. Zeits. Spiritusind. 1898, **21**, [28], 249.

The quantity of waste water to be removed, is greatest in the case of a potato-spirit distillery, and consists of potato-washings, the condensed water from the Henze potato-steamers, the water in which the barley has been steeped, and general washing-down waters. In corn and molasses spirit distilleries the two first mentioned do not appear, whilst in the case of a distillery manufacturing pressed yeast, the yeast-washing water must be added. Water is required for the various cooling operations but, as it is quite clean, it can generally be utilised for boiler-feed or potato-washing purposes. An approximate estimate of the quantity and composition of distillery waste waters per 1,000 litres of mash worked, is given as follows:—

| | | Grms. of dissolved solids. |
|---|---|---|
| Potato-washings...... | 2,000 to 2,500 litres containing | 1,000 to 1,250 |
| Steeping and Washing-down waters........ | 350 to 450 litres containing | 2,695 to 3,465 |
| Water from the Henze Steaming Apparatus | 100 to 125 litres containing | 750 to 937 |
| Total Waste Waters | 2,450 to 3,075 litres containing | 4,445 to 5,652 |

Or, in round numbers 2·5 to 3 cb.m., containing 1·8 kilo. of solid matter per cb.m. The waste liquor from the pressed yeast process contains about 7·7 kilos. of solids per cb.m., of which 6·2 kilos. are organic and 0·8 kilo. inorganic matter.

The waste waters from starch factories have been used with great advantage for the irrigation of meadow land, laid out in terraces on the Alsatian principle.

Since the waste waters from the distilleries very closely resemble those from the starch factories, it is suggested that they may be profitably employed for the same purpose.

One hectare of absorbent meadow-land, properly laid out, will dispose of 56 cb.m. of waste water per day; but this quantity must be regarded as a maximum, as the soil soon becomes over-manured and the grass withers in the summer. Where sufficient land is available, it is therefore well to distribute the waste waters over as large an area as possible and, if necessary, to dilute them with some of the clean water from the coolers.—J. F. B.

*Saccharine in Beer and other Liquids, Detection of.* A. Herzfeld and F. Wolff. Zeits. d. Vereins d. Deutsch Zuckerind. 1898, 558, through Woch. für Brau. **15**, [25], 335.

*See under* XXIII. *page* 802.

*White Wines prepared by the Decolorisation of Red Wines by Animal Charcoal, Reaction for Identifying.* A. Bimm. J. Pharm. Chim. 1898, **8**, [1], 9.

*See under* XXIII. *page* 802.

## PATENTS.

*Wines, Spirits, and other Alcoholic Liquors, Improved Process for the Rectification of.* P. E. Paulet, Juignac, Charente, France. Eng. Pat. 16,503, July 12, 1897.

Chlorine or oxygen-compounds of chlorine, either in the gaseous state or in solution, are agitated with the spirit or alcoholic liquor, whereby the unpleasant taste of the crude spirit is removed and the liquor rendered more suitable for consumption. A saturated solution of chlorine at 8° C. contains 3·167 grms. per litre, and 10 litres of this solution is about the average quantity required for the treatment of 100 hectolitres of spirit. The hydrochloric acid produced is said to be neutralised by bases occurring in the spirit and in the wood of the casks. J. F. B.

*Malt, Corn, or Grain for Brewing or like purposes, Improvements in the Treatment or Preparation of.* W. B. Turner, Leeds. Eng. Pat. 16,609, July 13, 1897.

Objectionable nitrogenous matter may be completely or partially removed from malt by immersing it in cold or warm water or saline solution for a length of time which is insufficient to wet the interior of the corns, and subsequently washing out the salt, if it has been used, and drying at a low temperature. The corns which sink and those which float may be treated separately; and the liquid in which the malt has been immersed may be worked up for the recovery of the by-products or be filtered and sterilised for further use. Raw corn, particularly British barley, may be treated in the same way, with favourable results. J. F. B.

*Straining Apparatus [Brewing], Improvements in Continuously Acting.* L. Prochazka, Turnau, Bohemia. Eng. Pat. 17,048, July 19, 1897.

To facilitate cleansing of the strainer, the usual revolving drum is replaced by a fixed gutter or trough with a sieve bottom of semi-cylindrical shape, preferably of slotted metal. Suspension brackets, removably connected to the ends of the trough, carry the screw conveyor fitting the bottom of the trough, so that the brackets with the screw conveyor may be lifted by counterpoised weights and pulleys for cleaning.

In cases where it is desired to allow solid bodies in the strained liquid to be deposited, an inclined gutter is arranged below the sieve trough, and from the lower end of the gutter a pipe leads down to the upper part of the inclined bottom of the collecting tank, whereby stirring up of the liquid is prevented. There is a lateral cock at a certain height above the bottom for drawing off the clear liquid, and another cock at the bottom for the discharge of the sediment. L. J. de W.

*Beer and other Alcoholic Liquors, The Manufacture of, Improvements in, and in Apparatus therefor.* F. S Masters, South Tottenham, and J. C. Shears, Surbiton. Eng. Pat. 18,645, August 11, 1197.

The process claimed, consists in fermenting wort or other saccharine liquids in closed vessels, under the pressure of the carbon dioxide evolved, and in utilising the gas given off at the surface, for the purpose of cooling and carbonating the fermenting liquid by pumping it off and injecting it back through the liquid from the bottom. A special form of yeast-skimming parachute is also described, which is raised and lowered by an externally threaded tube, through which passes the rod for working the yeast valve; both are thus on the same axis and can easily be adjusted from the outside. J. F. B.

*Brewers' and Distillers' Yeast, Improvements in the Treatment of the By-Products, or either of them, whereby a Substance is Obtained that can be Used as a Food by Man and for other Purposes.* C. O'Sullivan, Burton-on-Trent. Eng. Pat. 19,161, Aug. 19, 1897.

Yeast, the by-product of the brewer or distiller, is pressed in the usual way, packed into suitable vessels, and kept at a proper temperature, preferably 80 to 100 F., until it has acquired the greatest fluidity. The product is then filtered, with or without previous dilution, and the residue washed with water. The filtrate is heated to the coagulating point for some time and then heated to boiling and the coagulum filtered out. The filtrate is concentrated in a vacuum or under ordinary pressure to the consistency of a meat extract, or it can be reduced to a dry powder. It resembles ordinary meat-extract in odour, flavour, and composition. L. J. de W.

*Sterilising and Saturating with Carbonic Acid Gas, Liquids contained in Casks or like Vessels, A Process and Apparatus for.* C. Ameye, Iseghem, Belgium. Eng. Pat. 25,140, October 29, 1897. Compare Patents 1,926 and 16,091 of 1897. *See* this Journal 819, 1897.

This is a complete process for the treatment of beer, wine, or other liquid in casks, or other storage receptacles (other than bottles) by consecutively sterilising and saturating with carbonic acid gas.

The apparatus affords a means of imparting a rotatory motion to the liquid during the heating whilst keeping up the communication through the various channels to avoid pressure in the cask and prevent the liquid from being influenced by too long and too direct a contact with the source of heat. The arrangement, through the special channels in the shaft, combined with a system of gland or stuffing-box, forming a valve, allows the supply and draw-off cocks to remain fixed during the rotation of the shaft, and the pressure regulator lessens the shock of the entering gas and prevents pressure on the casks during rotation, and the discharge of the liquor or its scum or froth.—L. J. de W.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (A.)—FOODS.

*Proteids of the Horse-Bean* (*Vicia-faba*). T. B. Osborne, and G. F. Campbell. J. Amer. Chem. Soc. 1898, **20**, 393—405.

In his later researches Ritthausen (J. prakt. Chem. **26**, 504, and **29**, 418) states that the horse-bean contains the two proteids, conglutin and legumin.

The authors treated bean-flower with a 10 per cent. solution of sodium chloride, saturated the extract with ammonium sulphate, and subjected the precipitate and solution to a careful differentiation and purification. From their results it appears that in the horse-bean, as in the pea and lentil, the proteids soluble in a solution of sodium chloride are legumin, vicilin, legumelin, and proteose which have the respective average percentage compositions: carbon (for proteose see following abstract), 51·72, 52·29, 53·31; hydrogen, 6·95, 7·03, 6·97; nitrogen, 18·04, 17·43, 16·26; sulphur, 0·41, 0·17, 1·08; oxygen, 22·88, 23·08, 22·38.

By dialysing the aqueous extract of this bean, 16 per cent. of proteid was obtained; when, however, baryta was added to render the extract neutral, 18 per cent. of proteid was separated by dialysis. The proteid from the unneutralised extract was the purer. Both extracts, like those from the pea, lentil, and vetch when poured into water, produced a slight turbidity which vanished on shaking. Unlike the neutralised extracts as well as unlike both neutral and acid extracts of other legumes, the unneutralised extract of the horse-bean gave a heavy precipitate with neutral and pure sodium chloride. This precipitation of the globulin, according to the authors, indicates the presence of an acid compound. The addition of acetic acid gave a considerable precipitate, soluble in dilute brine, as did also calcium chloride and calcium sulphate.—J. L. B.

*Proteids of the Vetch.* T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1898, **20**, 406—410.

By extracting vetch meal with a solution of sodium chloride, the authors have obtained legumin, legumelin (see preceding abstract), and a very small proportion of a proteose having the percentage composition, carbon, 50·85; hydrogen, 6·75; nitrogen, 16·65; sulphur and oxygen, 25·75. Vicilin, which was found in the pea, horse bean, and lentil, is not present in detectable quantity in the vetch.—J. L. B.

*Proteids of the Soy Bean* (*Glycine hispida*). T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1898, **20**, 419—428.

The soy bean contains as its chief proteid constituent *glycinin*, a globulin similar in properties to legumin, but of a different composition, as the following numbers show:—Carbon, 52·12: hydrogen, 6·93: nitrogen, 17·53; sulphur, 0·79; oxygen, 22·63. (Compare preceding abstract on proteids of the vetch.)

This bean also contains a more soluble globulin, which appears to resemble phaseolin (this Journal, 1897, 820) in its composition, and reactions. Besides these globulins a small quantity of the albumin-like proteid—legumelin—was obtained, and also a proteose, regarding which, however, there is no evidence as to its purity or individuality.

—J. L. B.

*Roasting Coffee, The Volatile Products Obtained by.* H. Jaeckle. Zeits. f. Untersuch. d. Nahr. u. Genussm. 1898, **7**, 457—472.

The volatile substances produced by roasting coffee were condensed and collected, forming a light brown liquid. In this liquid the author found considerable quantities of caffeine, furfural, and acetic acid, together with small amounts of acetone, ammonia, trimethylamine, and formic acid. The presence of the body mentioned by Bernheimer (Monatsh. f. Chem. 1880, **1**, 156)—Caffeol—could not be detected. Another substance was also separated, but the quantity obtained was too small to allow of complete identification. It consisted of colourless crystals, which readily sublimed, melted at 115—117° C., and contained sulphur. The crystals were insoluble in water, almost insoluble in alcohol, but readily soluble in ether.—W. P. S.

*Cocoa Husks, The Valuation of.* G. Paris. Zeits. f. Untersuch. d. Nahr. u. Genussmittel 1898 [6], 389—390.

Cocoa husks are of value commercially for mixing with the fodder of cattle, and it is also possible to produce from them an infusion which can to a certain extent take the place of chocolate. The roasted husks examined by the author were supplied by a firm in Rome. They had the following percentage composition:—Water, 12·57; nitrogenous matter, 11·69; fat, 3·30; nitrogen-free extract, 45·76; fibre, 16·33; and ash, 7·35. These results are in accordance with those obtained by other chemists. A decoction of 50 grms. of the husks in 500 c.c. of water gave the following results on analysis:—Specific gravity, 1·1269; extract, 25·08; organic matter, 20·68; ash, 4·40; reducing sugar (dextrose), 0·21; theobromine, 0·79; acid (as tartaric), 0·12 per cent. The clear chocolate-coloured filter had a strong odour of cocoa, and an acid reaction. The colouring matter was found to be identical with that first examined by Mitscherlich and others, and termed "cocoa-red." Apart from the difference in chemical composition, an addition of powdered husks to cocoa powder would be readily detected under the microscope, the characteristic round, spiral, and hexagonal cells of the husk affording an easy means of differentiation.—C. A. M.

*Gelatine, Detection of, in Chocolate.* P. Onfroy. J. Pharm. Chim. 1898, **8**, [1], 7.

*See under* XXIII., *page* 802.

*Citric Acid in Vegetable Juices, Wine and Milk, Detection of.* G. Denigès. Rev. Chim. Analyt. Appl. **6**, [7], 110; Analyst, 1898, **23**, [267], 161.

*See under* XXIII., *page* 802.

## PATENTS.

*Sterilising Milk and other Nutritious Substances. Impts. in Apparatus for.* E. C. de Segundo, London. Eng. Pat. 3,926, February 16, 1897.

The apparatus claimed consists of a sterilysing chamber, with an air-tight removable cover. The bottles containing the liquid to be sterilised are fitted with wire fastened stoppers, and stand on shelves in the chamber. At first the stoppers only lie on the mouths of the bottles. Steam is admitted into the chamber until the temperature reaches 100° C.; the steam supply is then cut off and cold water cautiously run in, until it attains a level a little below the mouths of the bottles. When the contents of the bottles are cooled the water is drawn off and steam again admitted, raising the temperature to about 106° C., which is maintained for a certain time. The bottles are then closed by pulling down a rod passing into the chamber, and connected to cross arms, which force down the wires of the stoppers, and securely fasten them. The steam supply is now disconnected and the bottles are again cooled. The cold water must be allowed to enter cautiously, and at first it should be warmed a little by steam, in order not to crack the bottles.
—W. P. S.

*Food Products, Impts. in or relating to.* H. Higgins, Cambridge. Eng. Pat. 9,003, April 8, 1897.

The improvements claimed consist in mixing from 10 to 20 per cent. of gelatin, mucilage, gum, starch, dextrin, fish glue, &c. with the nitrogenous substance prepared according to Patent, No. 15,471, 1896, for the purpose of keeping this substance in suspension when used for soups. A little salt or sodium carbonate may also be added.
—W. P. S.

*Food Preparation, The Manufacture of an Improved.* A. M. Parker, London. Eng. Pat. 12,694, May 22, 1897.

The patentee adds to 100 lb. of liquid germ extract of wheat, prepared according to Patent No. 14,631, 1895 (see this Journal 1896, 608), about the same amount of flour, sago, rice flour or other farinaceous product. The mixture is then dried at a low temperature and formed into cakes or biscuits or may be used as a powder. The flour improves the keeping properties of the article.—W. P. S.

*Milk, Impts. in the Aëration and Preservation of, and Apparatus therefor.* R. G. Nash, Lucan, Ireland. Eng. Pat. 12,731, May 22, 1897.

The milk is heated to 160° F. for 15 to 20 minutes, and then run into a separator for the purpose of removing the cream. Coming from this vessel the milk enters a cooler where its temperature is reduced to 50° F. When cooled it is aërated or charged with sterilised atmospheric air and bottled. The sterilisation of the milk is completed by heating the full bottles as often as necessary to a temperature of from 160° to 186° F.—W. P. S.

*New Alimentary Extract, The Manufacture of a.* T. Hill-Jones, London. Eng. Pat. 15,145, June 24, 1897.

To effect the complete extraction of the contents of yeast cells, and to render soluble all proteid matter, the patentee heats the washed yeast with $2\frac{1}{2}$ per cent of sodium chloride to a temperature not exceeding 60° C., preferably *in vacuo*. The contents of the vacuum pan after the expiration of about three hours are treated with formic aldehyde or a digestive agent, and when peptonisation has taken place, the liquor is strained off and condensed. The digestive medium may consist of milk sugar, 20 oz., pepsin, 4 oz., pancreatin, 3 oz., ptyalin or diastase, 2 drachms, and lactic acid and hydrochloric acid, $2\frac{1}{2}$ drachms each. The hydration of the proteid matter may also be attained by the use of superheated steam, with or without pressure.
—W. P. S.

*Preserving Fresh Meat and other Analogous Comestibles by Fumigation, An Improved Mode of Apparatus for; and Materials to be used in.* T. Affleck, Sydney, Australia, and London. Eng. Pat. 15,406, June 28, 1897.

The meat to be preserved is suspended in a chamber into which are conducted the fumes obtained by burning a fumigating material in a closed retort. This material, the composition of which is claimed, together with the process, consists of oil of cinnamon or cassia, 1 part, extract of eucalyptus, 4 parts, saltpetre, 20 parts, sulphur, 55 parts, wattle bark, 60 parts, and charcoal, 160 parts.—W. P. S.

*Food Preparations containing Casein, The Production of.* S. Pitt, Sutton, Surrey, from The Chemische Fabrik auf Actien, vorm. E. Schering, Berlin. Eng. Pat. 17,915, July 30, 1897.

The claims are for mixtures of casein or casein salts with albumose or substances containing albumose and peptone. From these mixtures the casein does not precipitate on boiling aqueous solutions.—W. P. S.

*Alimentary Extract, The Manufacture of an Improved.* E. C. L. Kressel, London, and T. Hill-Jones, London. Eng. Pat. 18,714, August 12, 1897. (See also this Journal, 1896, 608.)

The proteid matter of yeast cells is rendered soluble by digesting the washed yeast with formic aldehyde, superheated steam, or a digestive agent (see Pat. 15,145, above). The claims are for the completed product, the process of producing the same, and for the apparatus for obtaining the extract by the use of superheated steam. This apparatus consists of a closed vessel or copper with an internal steam coil, the lower part of the coil being perforated. The copper has the usual fittings, such as safety valve, pressure-gauge, thermometer, blow-off cock, &c.—W. P. S.

*Food for Cattle, Process for the Production of.* A. Schmidt, Cassel, Germany. Eng. Pat. 26,628, November 15, 1897.

The claims are for foods prepared by mixing the glutinous liquid obtained by heating meat, bones, &c., under steam pressure, with suitable binding material, such as chaff, chopped straw, dry grain, bran, turnip shavings, fibrous peat, animal flour, or the like, and then adding molasses. By drying the product completely or partially, it is obtained in the form of a pulp or meal, and may be pressed into cakes or biscuits.—W. P. S.

*Antiseptic Bread or Rusks to be used in the Manufacture of Sausages and the like.* W. Preston, Leeds. Eng. Pat. 7336, March 26, 1898.

The antiseptic salt, consisting of boric acid and borax coloured with cochineal, is added to the flour to be used in making the sausages.—W. P. S.

*Canning Food, An Improved Process of.* E. Norton, Maywood, Cook County, Ill., U.S.A. Eng. Pat. 8514, April 12, 1898.

According to this patent the food is placed in cans fitted with specially constructed lids, which form an air-tight joint with the top of the can. The filled cans are introduced into the receiver of an air-pump, from which the air is then exhausted. The lids allow the air to escape from the cans under the reduced pressure, but prevent the return of air when the cans are removed from the receiver. When sufficiently exhausted the cans are withdrawn, and the lids now closed by the external pressure of the air, are securely fastened by means of an "expansion capper or seaming machine." The contents of the cans are then cooked or sterilized in the usual way.—W. P. S.

*Alimentary Product, made from Edible Nuts, and Process of Producing the same, New or Improved.* W. P. Thompson, Liverpool. From J. H. Hellogg, Battle Creek, Mich. U.S.A. Eng. Pat. 11,266, May 17, 1898.

The process claimed consists in thoroughly cooking the blanched kernels of the nuts (preferably peanuts). The cooked product is ground to a pulp, mixed with water, and sealed up in cans. The sealed cans are then heated to 215°—230° F. for from one to four hours.—W. P. S.

## (*B.*)—SANITATION; WATER PURIFICATION.

*The Treatment of Manchester Sewage.* City Surveyor's Annual Report. Chem. Tr. J., Aug. 6. 1898, 82.

The City Surveyor's annual report on the treatment of sewage at Davyhulme for the year ending 31st Dec. 1897, was submitted at the meeting of the Manchester City Council on Wednesday last. Details of expenditure for the year are given in tabulated statement under the following headings:—(1) Sewage Precipitation; (2) Sludge Pressing and Disposal; (3) Filtration; (4) Coal Supply; (5) Sundries and Incidental Expenses; (6) Summary of Annual Cost of Treatment. The net cost of the work for the year has been 19,089*l.* 9*s.* 7*d.*, as against 15,780*l.* 5*s.* 0¼*d.* for the year 1896. These sums are exclusive of interest and repayment of capital, and are equivalent to 2*l.* 11*s.* 9*d.* and 2*l.* 11*s.* 3*d.* respectively per million gallons of sewage treated. A summary of the cost of treating the sewage for the year 1897 shows a reduction of 2*s.* 0¾*d.* per million gallons on the cost of treatment for the year 1896. The volume of sewage delivered and treated during the year was 7,373,917,000 gallons, as against 5,818,200,000 gallons for 1896, being an increase of 1,555,717,000, or 26·7 per cent. on the preceding year. The estimated population contributing to the sewerage system has increased from 400,360 on Jan. 1st, 1897, to 512,500 on Dec. 31st, 1897, being an increase of 112,140, or 28 per cent. The increase of population contributing to the sewerage system during the year 1897 has exceeded the increase during the year 1896 by 49,780, or 79·8 per cent. The daily average flow of sewage was 20,426,363 gallons, that for 1896 being 15,896,721 gallons, showing an increase at the rate of 4,529,642 gallons per day, or 28·5 per cent. The average daily flow of sewage treated per head has ranged from a minimum of 39·3 gallons per head for the month ending May 19th to 50 gallons per head for the month ending Dec. 1st. The average amount of wet sludge precipitated has been 21·16 tons per million gallons, as against 21·84 tons for the previous year, yielding 7 tons 12 cwts of pressed cake per million gallons, as compared with 7 tons 18·4 cwt. for the year 1896. The proportion of chemicals used during the year was less than in the preceding year. When the sewage is diluted by large quantities of storm water it has been customary to suspend treatment during the flush, as the chemicals have but little effect in increasing the purity of effluent at such times, owing to the rapidity with which the water passes through the tanks, and to the extremely attenuated condition of flocculent organic matter which it is the function of the precipitant to carry down. When it is necessary to allow a portion of the storm water to pass directly by way of the storm overflow into the canal, the addition of chemicals would only increase the amount of suspended matter in the sewage. Treatment was stopped on account of storm water on 21 occasions during the year for a total period of 128 hours. The results of chemical treatment and precipitation are shown in a series of tables containing the results of the analyses made at various times. On no occasion did the oxygen absorption come within the requirements of the Mersey and Irwell Joint Committee, and the albuminoid ammonia was below the grain per gallon in only a few instances. Sand filters have been at work as far as practicable during the year. Experiments have been continued with the coke and cinder filters which were constructed at the end of 1895 under the advice of Sir Henry Roscoe. As in 1896 the cinder filter has throughout given better results than the coke, both as regards the percentage reduction of impurity effected, and in non-putrescibility. The results obtained from experimental coal filters are equal to or even better than those obtained from the cinder filter. During the year an experimental red sand and a burnt clay filter have also been tried, and likewise an automatic self-cleansing filter. Various experiments have been made during the year in connection with the settlement of solids in suspension in the sewage without the addition of chemicals, the exposure of the effluent to the air, and the passing of the air through the effluent.

*Effect of the Canal Water.*—Tests have also been made to ascertain the effect of the effluent on the water in the Ship Canal, and the effect of the admixture of acid and of water with the effluent. In all cases the effluent contains the larger amount of impurity than the Canal water, although in most cases, unless diluted by rain, the amount of impurity in the Ship Canal above Barton Locks is sufficient to cause putrefaction on incubating. The putrefaction which takes place in a mixture of Ship Canal water and tank effluent is generally greater in amount than in either taken separately. The experiments also showed that although it is possible to sterilise the effluent by the addition of a small quantity of acid, yet if a sterilised effluent be mixed with Ship Canal water the effect of the acid is largely destroyed and putrefaction again occurs. If an acidified sample of tank effluent be mixed with tap water the sterilising effect of acid appears to be maintained. When the Ship Canal water is largely diluted by rain, putrefaction does not, as a rule, take place in the mixture of effluent and Canal water. Sea salt has been added to samples of tank effluent with the result that a large quantity is found to be necessary to prevent putrefaction taking place.

*Sewage Filtration at Winsford, Cheshire.* Engineer, 1898, **85**, (2216), 567.

In the manufacture of salt at Winsford, large accumulations of cinders, basses, clinkers, and ashes have been formed, and some twenty years ago it was proposed to filter the sewage through these cinders, &c. Intermittent downward filtration beds of cinders, clinkers, basses, and ashes were laid down on either side of the river Weaver, and the sewage from the adjacent towns has been successfully filtered in this manner up to the present time. The sewage of Wharton (comprising about 5,000 inhabitants) is dealt with on the easterly side of the river, and that of Over and Winsford (containing about 7,000 people) on the opposite side. The filter beds are from a half to three-quarters of a mile from the towns. On the Wharton side the sewage flows at a fairly rapid rate down an open pipe track, whence it empties into a carrier, about three or four feet wide, made with cinders, basses, &c., with 15-in. pipe communications to the settling and filter beds beyond, of which there are seven, each measuring thirty yards by ten yards. The walls dividing the filter beds are nearly 3 feet in height and 4 feet wide, and are entirely composed of the rough clinkers, basses, and cinders from the adjacent salt works, filled in with soil. The sewage flows first from the carrier alluded to, through the 15-inch pipes placed rather more than a foot above the top line of filter bed, into the first or settling tank, where the sludge is chiefly deposited, and when the sewage here rises above the outlet pipe of this tank, it flows into the greater filtering tank beyond. The bed of the filters consists of waste cinders, &c., the top layer of ashes being 2 ft. 6 ins. deep, and the total depth 7 ft. or 8 ft., rough clinkers forming the bottom. While one pair of filters are nearly filled with sewage, which has been accumulating from a week to a fortnight, another pair may be partially cleared, on another set the sewage may only just have been turned on, and others may be empty and dry, covered with clean or newly raked-up cinders, all in readiness to take part in the purification process. It is stated that microbic action undoubtedly takes place, since in course of time, as the filters gradually dry, the sludge melts away, and what is at last wheeled from the dry filters and placed on the banks undergoes a further disintegrating process, until the residue cannot be distinguished from the neighbouring soil. The method of treatment is exactly similar on the Over side of the river, where there are five pairs of filter beds, the first or deposit areas being 18 ft. by 39 ft., and the larger filtering tanks beyond being 150 ft. long by 39 ft. wide. The sewage passes on to one pair of tanks for about a week or a fortnight, and is then shut off and turned on to another set, and thus the filter beds obtain rests of two months or more at a time, during which the top layers of the filters are stirred up, cleaned, and raked over, and made ready for again receiving sewage. There are no under drains to the filters, and the sewage passes through the whole of the cinders which stretch up to the brink of the river, and as the water of the river along the edge of the cinders shows not the slightest trace of discoloration or pollution, it must be concluded that the sewage is satisfactorily purified in its passage through the filters. The flow of the sewage through the filters is comparatively slow, and this is believed to be more beneficial with regard to effective purification than if the effluent were discharged all at once. The original cost of the whole of these filtration works was 300*l.* (exclusive of the cost of the land), and 50*l.* per annum suffices to keep them in working order.—A. S.

*Yorkshire, On the Water Supplies of.* T. Fairley. J. Fed. Inst. Brewing, 1898, **4**, 393–404.

The softest waters in which the solids do not exceed 7 grains per gallon are collected from the old igneous rocks and certain sandstones free from soluble substances found in the north, north-west, and west of Yorkshire. Waters of moderate softness containing dissolved solids from 7 to 15 grains per gallon are found in the north-west in the vicinity of the carboniferous limestone, as in the Upper Airedale, Wharfdale, Nidderdale, &c. The water from wells of a moderate depth over a large part of the county also come within these limits. Hard waters containing from 15 to 30 grains of solids per gallon are obtained from many moderately deep wells in or near the limestone. Such waters are also obtained from the coal measures of the south-west, in some parts of the magnesian limestone formation, and from the triassic deposits extending north-west and south-east in the vale of York. The waters from wells in the oolitic and lias formations in the north-east, and in the chalk of the East Riding, may be included in this class.

In the coal measures and trias of the central part of the county, waters are obtained from deep wells containing from 30 to 60 grains of solids per gallon. The author gives analyses of these different waters, and also discusses some of abnormal character.—J. L. B.

*Organic Matter in Water, A Method for the Differentiation of.* A. G. Woodman. J. Amer. Chem. Soc. 1898, **20**, [7], 497.

*See under* XXIII., *page* 806.

*Waters, Method of Determining the Nitrites in.* L. Robin. J. Pharm. Chim. 1898, **7**, [12], 575.

*See under* XXIII., *page* 797.

## PATENTS.

*Purification of Sewage, Impts. in or Relating to, and Apparatus therefor.* W. D. Scott-Moncrieff, 14, Victoria Street, Westminster. Eng. Pat. 18,555, August 10, 1897.

The bacterial purification of sewage is effected by the use of superimposed layers of filtering material, such as coke, with a clear air space between each. Full advantage of the porosity of the filtering material is obtained by intermittent filtration, by means of filters so arranged that the liquid from one discharge has time to drip away, leaving a highly aërated filter for the next discharge.

The most suitable dimensions of filters and air spaces, and the best number of filters to employ, should be experimentally determined according to the nature of the sewage. (*See also* Eng. Pat. 17,006 of 1892, 13,400 of 1897, and this Journal 1892, 705).—N. H. J. M.

*Precipitating Tanks and the like, Impts. in and Relating to* A. Fidler, Blackburn, Lancashire. Eng. Pat. 19,249, August 20, 1897.

The object of the invention is the removal of the collected sludge. The tank, which is circular, is provided with a central shaft to which a curved blade is fixed. This is of such a form that when the central shaft is rotated, the sludge at the bottom of the tank is carried into a cylindrical well. This receptacle is connected below with an outlet tube, which is kept closed while the sludge is collecting, whilst above it a loosely fitting piston is suspended. When the well is sufficiently full of sludge the piston is lowered until it reaches the mouth of the well. The valve of the outlet pipe is then opened when the pressure of liquid in the tank forces the piston down and thus expels the sludge. —N. H. J. M.

*Drying or Carbonisation of Sewage Matter, and Generally of Vegetable or other Substances; Impts. in Retorts for.* W. H. Bowers, West Gorton, Manchester. Eng. Pat. 19,284, August 20, 1897.

The retort is constructed with parallel flues extending longitudinally within it, side by side or in tiers, the intermediate space being occupied by the substance to be dried. The chamber is furnished with a ventilating shaft, a supply inlet, and a discharging outlet; it is divided by means of ribs into any number of equal spaces through which an endless chain, provided with bars or scrapers, is worked for the purpose of dragging the material to the outlet. —N. H. J. M.

*Purification Process for Treating Sewage and other Liquids, Impts. in.* J. T. Deery, Philadelphia, U.S.A. Eng. Pat. 5,603, March 8, 1898.

The sewer is connected by means of transverse pipes with two receiving chambers, each capable of holding the whole amount of sewage to be treated, so that while one is in use the filtering materials of the other can be renewed. The sewage is received by a distributor, an open sheet-iron vessel extending across the receiving chamber. This retains the heavier portion of the solid matter, whilst the liquid overflows in a thin even stream on to the screening floor below. This consists of a permanent lower iron floor and a movable upper one, the intermediate space being fill with coke or charcoal. The sludge is collected on the floor, from which it is afterwards removed in buckets. The upper iron floor is then taken up, the coke taken out and heated in an oven; the grease thus melted out is collected.

During the treatment the escape of foul air is prevented, but the different parts of the receiving chamber are thoroughly aërated, and day-light is admitted by means of a skylight.

The sewage, filtered and aërated as described, which collects at the bottom of the receiving chambers is next conveyed to elevated filter beds, through which it percolates, falling in drops to a lower filter. The then thoroughly purified liquid is finally discharged.

The thorough aëration, in conjunction with the action of light, promotes the growth of healthy micro-organisms, whilst disease-bearing germs are, at the same time, destroyed.—N. H. J. M.

*Sterilisation, or the Disinfection of Waters of all Kinds; Treatment of Sweetened Syrups and other Analogous Applications by Means of Ozone, Air or other Gases, Apparatus for.* H. H. Leigh, London, from La Soc. Anon. pour les Appl. Ind. de l'Ozone, Paris. Eng. Pat. 6,061. March 11, 1898.

*See under* XVI., *page* 779.

*Purifying and Sterilising Filtering Media, an Improved Process and Machine for.* J. Metzger, Worms-on-the-Rhine, Germany. Eng. Pat. 7,094. March 23, 1898.

The apparatus consists of a tank connected at both ends with a spiral conveyor underneath it. The filtering substance, such as fragments of cloth, is fed at one end of the tank containing the purifying liquid, passes into the conveyor, from which it is again forced into the other end of the tank. The passage through the conveyor is rapid, whilst the return through the purifying liquid is slow.

Any scum which rises to the surface of the liquid is removed by a rotating drum provided with paddles. The scum passes into the drum and can be drained off. Near the drum is a perforated pipe supplying jets of water for the purpose of washing off any pieces of filtering substance which happen to adhere to the drum.

Additional purifying material, together with steam, may be admitted directly to the conveyor.—N. H. J. M.

*Purification of Oils, Syrups, and other Liquids, and for Deodorising and Decolorising Purposes.* R. A. L. Hill, Wimbledon, Surrey. Eng. Pat. 7,808. April 1, 1898.

*See under* XII. *page* 774.

## XIX.—PAPER, PASTEBOARD, Etc.

*Cotton Rags, Bleaching of Black.* Papier Zeit. 1898, **23**, [55], 2,027.

Aniline black is looked upon as an indestructible dye, and cotton rags dyed with it have hitherto only been used for common wrapping papers. According to the Revue de la Papeterie, a method has been found of bleaching such rags without weakening the fibres too much. The method is as follows:—

By the influence of acid sulphite salts at a high temperature the black colour is turned to a light chamois. When such prepared pulp is treated alternately with weak acids, pure water, and a solution of bleaching powder, a pulp can be obtained sufficiently white to be used as an addition in manufacturing writing and book papers.—S. P. E.

*Paper Pulp, Estimate of Dry Fibres in.* Papier Zeit. 1898, **23**, [55], 2,027.

A letter has been received by the editor of the Papier-Zeitung respecting the rules adopted by the English and Scotch Papermakers' Union respecting the amount of moisture in wood pulp. The rule (No. 3) is as follows:—

3. Sampling and Testing.—In the event of a claim for deficiency of air-dry material, excess moisture or short weight, samples to be taken from near the top, centre and bottom of each, of not less than 2 per cent of the bales (at least five to be sampled) forming the delivery in dispute, said bales to be intact, and to be accurately weighed before sampling. The samples to be submitted to a public analyst of acknowledged good standing, and said samples to be drawn in the presence of the two parties, or their representatives, or by the analyst himself. The analyst to declare the actual amount of absolutely dry pulp found in the samples tested, and in estimating the total air-dry pulp, the weight of the bales before sampling to be taken into account. The costs of the reference to follow the result, and the award to be final.

If the seller takes orders subject to such rules, unjust objections will be impossible.—S. P. E.

*Waste Lyes from Cellulose Mills, Treatment of.* Papier Zeit. 1898, **23**, [45], 1,652.

At a lecture given by Mr. L. J. Darenfeld in June 1897, he quotes from Hofmann's Treatise, page 1510, referring to his own method of treatment of waste sulphite liquors. By this method sodium sulphate is added to the calcium bisulphite liquor, forming calcium sulphate and sodium bisulphite, about two-thirds of the lime being precipitated. It is claimed that the pulp obtained by boiling with this calcium-sodium-bisulphite liquor is of superior quality, and requires only 6 to 7 per cent of bleaching powder to bleach it. The spent liquor is neutralised with recovered soda, concentrated, and burnt and the alkali recovered in the usual manner. According to Darenfeld's experiments and calculations, spent sulphite liquor contains sufficient combustibles both to evaporate the liquor and leave a surplus of heat.

Two liquors from Flodquist digestors, one a sodium-bisulphite liquor and the other a calcium bisulphite liquor, both of 1·05 sp. gr., were evaporated to dryness, and the calorific power determined in a Berthelot's calorimeter as modified by Dr. Hempel. The average calorific power of the residue from one litre was 560 Thermal Units (metrical and centigrade), or one cubic meter = 560,000 T.U., which is equal to the calorific power of 80 kilos. of coal of 7,000 T.U. Somewhat similar results were obtained with other liquors, varying according to the chemical composition and concentration of the spent liquor. As a brown coal, developing 2,866 T.U. and containing 54·39 per cent. of water, gave a useful effect of 61·3 per cent., the useful effect of the concentrated waste liquor may safely be reckoned as 60 per cent.

Of the 560,000 T.U. obtained from one cubic metre, 60 per cent. = 336,000, may therefore be utilised in steam boilers fed with waste liquor. Assuming the liquor to reach the boiler at 50° C., and to leave it at 170° C., there are consumed (170 − 50) × 1,000 = 120,000 T.U. for heating the liquor, leaving 336,000 − 120,000 = 216,000 for raising steam. As the latent heat at 170° C. is 488 T.U., $\frac{216,000}{488} = 444$ kilos. of water are evaporated. One cubic meter of spent liquor contains 937 kilos. of water, and as 80 + 444 = 524 kilos. have already been evaporated in the incinerating furnace and the steam boiler, only 413 kilos. are left to be evaporated in the multiple vacuum apparatus.

As one kilo. of steam will evaporate at least 4½ kilos. of water from a liquor of 170° C., and reckoning 4 to be safe, 104 kilos. of steam are used for the vacuum evaporator. Accordingly, 340 kilos. of the 444 kilos. of steam raised in the boiler are applicable to other purposes, and this is equal to 43 kilos. of good coal, of steam raising power equalling 8·1. About 400 kilos. of drip water at 100° C. from the evaporator, and about 2,000 kilos. of water at 35° C. from the surface condensor must be added, which at a feed-water temperature of 10° C. means a further 15 kilos. of coal, making in round numbers the total surplus calorific power of one cubic meter of liquor after evaporation and incineration, equal to 58 kilos. of coal, of steam-raising capacity 8·1.

If the boiler is not fed with the neutralised liquor, the surplus is still equal to 52 kilos. of good coal.

As a proof of the correctness of these results dry wood develops a calorific power of about 4,400, and cellulose about 4,000 T.U. Therefore, 100 kilos. of air-dry cellulose give 90 × 4,000 = 360,000 u.h., and the corresponding 200 kilos. of bone-dry wood give 200 × 4,400 = 880,000 T.U. The difference of 520,000 belongs to the incrustations, and corresponds with the calorimeter tests, according to which 1 cubic meter of spent lye should deliver 530,000—560,000, 100 kilos. of cellulose giving at least 900—1,000 litres of spent lye.

Two faults are found in most mills, too much water being carried into the chamber of combustion and the burning being done with excess of air, the temperature for some periods of time being allowed to get so low as to effect dry distillation without combustion, causing a loss of 16 to 20 per cent. of heat.—S. P. E.

*Tannin-glue, Sizing with.* Papier Zeit. 1898, **23**, [55], 2025.

The principal aim of the formerly produced tannin-glue was to give a better "handle" to the paper, and make it bulky and strong. This it did, but was not suitable for rendering the paper impervious to writing-inks, and it has therefore been used in combination with resin-size, to obtain the proper results. On account of this, experiments were made, and a tannin glue is now produced which makes the paper tough, and also impervious to ink, thus doing away with the necessity of using resin-size. The bad smell of this product is now entirely destroyed, and the dull shade caused by using the glue is so far reduced as to enable the tannin-glue to be used for sizing white printing and light-coloured envelope papers. A large saving is also effected in the quantity of alumina used as against the quantity required to precipitate the resin, 25 to 30 kilos. of sulphate of alum being sufficient to precipitate 100 kilos. of the tannin-glue.—S. P. E.

*Formaldehyde, Action on Spent Sulphite Lyes, Asparagine, p-Phenolsulphonic Acid, &c.* C. Goldschmidt. Chem. Zeit. 1898, **22**, [38], 374.

*See under* XX., *page* 790.

## PATENTS.

*Paper, Cardboard, or Pasteboard ; An improved Process and Apparatus for Incorporating Dissolved and Suspended Substances into.* H. Schulte, Austria. Eng. Pat. 16,315, July 9, 1897.

To prevent the excessive loss of sizing material which takes place in manufacturing engine-sized paper, the inventor proposes to add the sizing material to the paper, after a large percentage of the water has been removed from the pulp. For this purpose the web of paper is passed over rollers or a spraying device, to which the solution of size is automatically supplied, after the paper has passed over the wire and gone through the first press rolls.—S. P. E.

*Waterproof and Washable Glazed Paper, Process for Producing.* T. Schiffer, Dulken, Prussia. Eng. Pat. 17,736, July 28, 1897.

Paper of ordinary quality is first coated with a solution of size, glue, or gelatine, in which the filling material, such as blanc-fixe, china clay, &c., with or without the addition of colours or dyes, is held in suspension. To this prepared paper a 1 or 2 per cent. solution of chrome alum or other chromic compound is applied, and the paper exposed to light. By this means the size used is rendered insoluble in water, without assuming a greenish tint, as heretofore.

To produce a highly glazed paper, a larger quantity of filling material is added to the size, and the treated paper is calendered. Paper so prepared may be cleaned with a sponge and soap without losing its gloss and colour, being impervious to moisture.—S. P. E.

*Paper and Pulp Manufacture, Impts. in Settling and Recovering Apparatus for.* W. Curtis, U.S.A. Eng. Pat. 4945, March 1, 1898.

The waste water from paper or pulp mills is run into a tank without agitation, and passes below baffle boards, being finally allowed to pass away near the surface of the tank. The baffle boards cause the suspended materials in the water to settle to the bottom of the tank, whence they are removed to a discharge outlet by scrapers. The tank may be either long and cylindrical or, if not much room is at liberty, it may be made circular. In the long cylindrical tank, hanging aprons are fastened to the bottom edges of the lower baffle boards, which aprons give to the scraper, and fall back into position, compelling the water to flow slowly over the boards.—S. P. E.

# XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Monazite Sands, The Yttria Earths contained in.* O. Boudouard. Comptes Rend. **126**, [23], 1648—1651.

After repeated fractionings of mixed oxides, the metals corresponding to the constituents of which mixtures gave atomic weights of 102·2 to 103 in one series, 95 to 99·1 in a second, and 96 to 112 in another, the author obtained an oxide of the yttrium group, with an atomic weight approximating to 96. This is far above that (89·5) of yttrium, and he is at present engaged on the question whether this oxide is homogeneous or is a mixture of yttria with earths of higher atomic weight.—J. T. D.

*Cerite Metals, Separation of the, and the Solubility of their Sulphates in Water.* W. Muthmann and H. Rölig. Ber. **31**, [11], 1718—1731.

The authors have shown that cerous sulphate forms three hydrates, each of which has a special solubility curve.

*Lanthanum Sulphate.*—The starting material consisted of crude didymium carbonate, containing much cerium carbonate, proportionately little La and Di salts, besides salts of the ytterite group and other impurities. It was strongly ignited till the colour was chocolate-brown and then digested with concentrated $HNO_3$ to dissolve the oxides of La, Nd, Prd and of the ytterite group, most of the Ce remaining as a slimy mass of basic ceric nitrate, which was removed by decantation. To the liquid was added ammonium nitrate equal to a quarter of the weight of oxides taken ; and on boiling down, cerium ammonium nitrate, which is little soluble in nitric acid, soon crystallised out, a further separation occurring when the liquid was cooled.

This crystallisation process is very suitable for preparing pure Ce salts, but one must be careful not to evaporate down too far, lest colourless crystals of the La double salt separate also.

To separate the last portion of the Ce, the mother-liquor was almost neutralised, a good excess of ZnO was stirred in and concentrated solution of $KMnO_4$ was gradually added until after prolonged stirring, the permanganate colour persisted. All the Ce was thus precipitated, only traces of La, Nd, and Prd going down—

$$3Ce(NO_3)_3 + KMnO_4 + 4ZnO + 6H_2O = \\ MnO_2 + KNO_3 + 4Zn(NO_3)_2 + 3Ce(OH)_4.$$

The filtrate was free from Ce, the test used being to add to the nitrates, after removing Zn and Mn, some sodium acetate and a few drops of $H_2O_2$, and boil. A precipitate forms in all cases, pure white in absence of Ce, but brownish when it is present. 1 mgrm. of $Ce_2O_3$ in 10 c.c. of solution may be thus detected. The filtrate was acidified with acetic acid, the zinc removed by $H_2S$, the earths precipitated by oxalic acid, and the oxalates reconverted into nitrates. The ytterite elements were now removed as usual by a double precipitation at the boiling temperature with $K_2SO_4$, the double sulphates of La, Nd, and Prd were digested for several hours with hot concentrated oxalic acid solution, superheated steam being passed through the liquor, and the oxalates were again converted into nitrates. To remove the Nd and Prd the somewhat concentrated, boiling, neutral nitrate solution was stirred violently and continuously with a Witt's stirrer, while very fine magnesia was gradually sifted in, until the solution no longer gave an absorption spectrum. It was then filtered and precipitated by ammonium oxalate after the addition of $NH_4Cl$ ; pure lanthanum oxalate being thus obtained.

*Solubility of Lanthanum Sulphate.*—By digesting the $La_2O_3$ with concentrated $H_2SO_4$, evaporating, and heating for a considerable time at about 500° C., a snow-white anhydrous sulphate was obtained. Its solubility in cold water seems almost unlimited. If solutions containing 15—20 per cent. of sulphate be allowed to stand at 0°, about six-sevenths of the substance crystallises out in needles containing $9H_2O$. Such a solution is supersaturated in respect of the hydrate, but is unsaturated in respect of the anhydrous salt. The separation is more rapid the higher the temperature, and it is always $La_2(SO_4)_3 + 9H_2O$ which is produced. The hexa-hydrate crystallises from solutions containing 50 per cent. of free $H_2SO_4$, but this hydrate changes at all temperatures into the enneahydrate when in contact with aqueous solutions. Solubility determinations were made by stirring the water and crystals together for 3—6 hours in a thermostat and then analysing the solutions, and the following results from the solubility curve may be quoted—at 0°, 3·01 ; at 14°, 2·55 ; at 50°, 1·55 ; at 100°, 0·70 parts $La_2(SO_4)_3$ to 100 parts of water. Thus the solubility decreases with the rise of temperature, as is the case with all the cerite earth sulphates.

*Didymium Sulphates.*—The precipitate obtained with magnesia in separating the lanthana as above was converted into nitrate and again precipitated by magnesia, but not this time till disappearance of the absorption spectrum. This precipitation was repeated, and after double precipitation with oxalic acid to remove the magnesia, the oxalate was converted into anhydrous sulphate. The proportion of Prd to Nd in the product was 30 : 70, as determined by a comparative spectroscopic test with solutions of known composition.

The mixture of the two sulphates was extremely soluble in ice-water. By agitating it for some time with water at 0°, a solution containing 37 per cent. of sulphate was obtained, but at this point crystals of the octohydrate separated out. The dissolving of the anhydrous sulphates ceases whenever from any cause, crystals of the hydrate begin to form. At all temperatures only the octohydrate was obtained, and the following are some results from the solubility curve:—at 0°, 14·2; at 14°, 12·52; at 54°, 6·35; at 100°, 2·30 parts $(NdPr)_2(SO_4)_3$ per 100 parts $H_2O$.

*Praseodymium Sulphate.*—The starting material was the ammonium double nitrate, of pure leek-green colour, containing no Nd but at least 20 per cent. of La double salt. To remove the latter, the Prd was precipitated by and then freed from magnesia as described above and converted into the sulphate. The anhydride was dissolved in ice-water, warmed to 20°, the first crystals (which would contain any La present) were removed, and this crystallisation was repeated. This praseodymium sulphate gave three hydrates, containing 12, 8, and 5 molecules of $H_2O$ respectively. The first appears to be stable only within a small range of temperature near 0° C. The octohydrate crystallises from solutions between 0° and 85°, above this temperature the pentahydrate is obtained. At about 75° the octo begins to change into the pentahydrate, and at lower temperatures the reverse change occurs. Some of the solubility results are—at 0°, 19·80; at 18°, 14·05; at 55°, 7·02; at 75°, 4·20; at 85° (pentahydrate), 1·60; at 95°, 1·00 parts $Prd_2(SO_4)_3$ per 100 parts $H_2O$.

*Neodymium Sulphate.*—The material used contained a small quantity of Prd and some element of the erbium group, but the amount of material was too small for further purification. Apparently there is an octohydrate of the sulphate, isomorphous with the octohydrate of the Prd salt. Some of the solubility results are—at 0°, 9·41; at 16°, 7·20, at 50°, 3·64; at 100°, 2·68 parts $Nd_2(SO_4)_3$ per 100 parts $H_2O$. There is thus an extraordinary difference in the solubility of the Nd and Prd sulphates; and by submitting 300 grms. of anhydrous pure didymium sulphate to 60 consecutive fractional crystallisations at 30°, 4 grms. of an octohydrate of almost pure $Nd_2(SO_4)_3$ were ultimately obtained, which a comparative spectroscopic test showed to contain at most 0·3 per cent. of the Prd salt.—H. B.

### *Colloidal Mercury.* A. Lottermoser. J. prakt. Chem. 1898, **57**, [10—11], 481.

Colloidal mercury is obtained in the following manner:—A very dilute solution of mercurous nitrate is stirred into a similarly dilute solution of stannous nitrate. Both solutions should contain only sufficient free acid to prevent the precipitation of basic salts. The ratio of the reagents is $2(HgNO_3 + 2H_2O) + Sn(NO_3)_2$, but a considerable excess of stannous nitrate must be employed to prevent the transformation of the colloidal mercury. The resulting mixture is a dark brown fluid. This is then mixed with a concentrated solution of ammonium citrate, which throws out the colloidal mercury as a black precipitate. The liquid is cautiously neutralised with ammonia, care being taken to avoid rise of temperature; the precipitate is then allowed to subside, and after decanting the bulk of the supernatant liquid, collected on a porous filter and dried *in vacuo* over $H_2SO_4$. The resulting colloidal mercury forms shining spangles, which are soluble in water, giving a dark brown solution.—J. O. B.

### *Silver Plumbite.* Fr. Bullnheimer. Ber. 1898, **31**, [9], 1287.

The silver plumbite obtained differs from previously described silver plumbites, in that it crystallises well and has constant composition $(PbO_2Ag_2)$.

*Preparation.* A silver nitrate solution is made alkaline with caustic potash, and so much ammonia is added to it that no turbidity may be produced when the solution is mixed in the cold with a solution of lead nitrate made alkaline with caustic potash. The silver solution thus prepared is poured into an excess of the lead solution (1 grm. $AgNO_3$ to about 2 grms. $Pb(NO_3)_2$). The mixture is slowly heated on the water bath, and a solid consisting of small brown needle crystals separates out. The mother liquor is poured off and the crystals washed with ammonia water, then pressed between filter paper and dried over caustic alkali.

The crystals under the microscope appear as brownish, flat, highly refractive monoclinic prisms. They show vivid polarisation colours, parallel extinction, colourless adamantine lustre, and are not pleochroic.

The constitution is Ag-O-Pb-O-Ag. Sp. gr. 8·60.

On heating it becomes greyish black without losing its lustre or form, and only on strong ignition does the decomposition according to the following equation take place:—

$$PbO_2Ag_2 = PbO + 2\ Ag + O.$$

It is insoluble in water, ammonia, or caustic potash, soluble in hot acetic acid, and fairly soluble in cold nitric acid. Dilute hydrochloric and dilute sulphuric acids only attack it on prolonged heating. Concentrated sulphuric acid, hydrofluoric acid, and hydriodic acid decompose it at once. The action of reagents is the same on silver plumbite, which has become black by heating.

It yields an olive-green powder and gives a black streak.
—J. McC.

### *Mercuric Sulphate, Organic Compounds of, with Ketones.* G. Denigès. Comptes Rend. 1898, **126**, [26], 1868—1871.

Having previously shown (Comptes Rend. **120**, 628—781; **126**, 1145 and 1277) that mercuric sulphate forms well defined compounds with thiophen and the hydrocarbons of the ethylene series, since these bodies contain non-saturated groups or double linkages, it was suggested that other bodies possessing analogous structure might also furnish definite compounds with that salt. This has been found to be the case with aldehydes and ketones. The mercuric reagent employed was obtained by dissolving 5 grms. of mercuric oxide in a mixture of 20 c.c. of sulphuric acid and 100 c.c. of water.

When a fatty ketone is boiled with a great excess of the reagent (1 c.c. of the ketone to 250 c.c. of mercuric sulphate solution, compounds having the general formula $(SO_4Hg)_2.3HgO.CO.R'_2$ are obtained.

With ordinary acetone the reaction is very complete and strictly quantitative. With the higher ketones, secondary reactions occur, the yield of the mercuric compound is less, and, through the oxidation of the ketone, mercurous sulphate is formed, which contaminates the product. With dimethylketone, however, a perfectly white crystalline compound is obtained, having the formula—

$$(SO_4Hg)_2.3HgO.C_3H_6O.$$

which dissolves entirely in HCl and only contains the theoretical quantity of mercury. Similar compounds have been obtained with other ketonic bodies. In a future communication the application of the results to the determination of ordinary acetone will be dealt with.—J. O. B.

### *Formaldehyde; Action on Asparagin, p-Phenolsulphonic Acid, Spent Sulphite Lyes, &c.* C. Goldschmidt. Chem. Zeit. 1898, **22**, [38], 374–375.

Asparagin and guanidine in weak alkaline solution give with an excess of formaldehyde, unstable addition products; oxanilic acid gives a reddish resin.

A 35 to 40 per cent. solution of formaldehyde dissolves quinoline, isoquinoline, and quinaldine; weaker solutions give a turbidity. This reaction may be used as a test for the concentration of solutions of formaldehyde, and affords a further proof that formaldehyde gas and water form a glycol-like compound.

Spent sulphite lyes heated in hydrochloric acid solution with excess of formaldehyde give a brown precipitate similar to the methylene compounds of wood sugars; it may be prepared pure through the benzoyl compound. Formaldehyde is worthy of attention as a means of purifying these waste lyes, it fixes the sulphurous acid and prevents excessive growth of moulds and algæ.

*p*-Phenolsulphonic acid similarly treated gives a white amorphous precipitate for which no solvent was found. Boiling soda lye decomposes it, evolving formaldehyde and leaving a pale yellow body. Hydrochloric acid, phenol, and formaldehyde give a product soluble in soda lye on heating, and which reddens on exposure to the air. Analysis shows 2 to 3 per cent. of sulphur, which the author regards as impurity, the formula deduced is $C_{16}O_5H_{18}$. It may be a high molecular anhydroproduct of oxydibenzyl alcohol.

—L. J. de W.

### *The Alkaloids of Macleya Cordata R. Br.* K. Hopfgartner. Monatsh. für Chem. 1898, **19**, [4], 179—210.

Eykmann (Rec. trav. chim. Pays-Bas, **3**, 182) extracted from the roots of this plant two alkaloids, sanguinarine and macleyine, the latter of which the author finds in the leaves and stems accompanied by a third base. The plants were cut up, extracted by very dilute hydrochloric acid, the liquid concentrated, alcohol added, the alcoholic solution poured off and the residue again extracted with alcohol. After distillation of the alcohol, caustic soda throws down bases. These are extracted from the liquid by allowing it to pass in a thin stream through a long column of chloroform. The alkaloids are extracted from the chloroform by dilute hydrochloric acid, precipitated by alkali, and converted into nitrates, of which macleyine nitrate is difficultly soluble in water.

*Macleyine.*—After repeated recrystallisation from alcohol, the base shows the melting point 207° C., it is identical with protopine obtained from *chelidonium majus* and *sanguinaria canadensis*, and with Hesse's protopine from opium. The formula appears to be $C_{20}H_{19}NO_5$; it contains no methoxyl group. The alkaloid unites with methyl iodide to form the compound $C_{20}H_{19}NO_5CH_3I$. Among the products of the oxidation by permanganate in acid solution were found formic acid and methylamine. On reduction with sodium amalgam a base is obtained, probably possessing the constitution $C_{18}H_{21}NO_4$.

*β-Homochelidonine.*—The second base, forming the more soluble nitrate, is identical with β-homochelidonine. It has the formula $C_{21}H_{23}NO_5$, and contains two methoxyl groups. In general the properties of this base and its salts agree with those described by Selle; the author, however, finds for the hydrochloride and platinum double salt the formulæ—

$$C_{21}H_{23}NO_5.HCl + 1.5H_2O \text{ and}$$
$$(C_{21}H_{23}NO_5.HCl)_2PtCl_4 + 2.5H_2O,$$

which differ in the amount of water of crystallisation from the salts prepared by Selle.—A. C. W.

### *Ouabaïn [Strophanthin], A Crystalline Heptacetin from.* Arnaud. Comptes Rend. **126**, [23], 1654—1656.

By heating Ouabaïn [Strophanthin], previously dried at 110° C., with acetic anhydride and zinc chloride at 70° C., and pouring the liquid after cooling into five or six times its bulk of water, a crystalline acetin is obtained, which is purified by recrystallisation from 85 per cent. alcohol. A considerable quantity of an amorphous acetin is formed at the same time The crystalline compound is insoluble in water, readily soluble in dilute acetic acid, in boiling alcohol, acetic ether, or acetone; very slightly soluble in cold alcohol or in ether. It is lævo-rotatory $[\alpha]_D = -68^\circ\cdot5$; melts at 310° C. with decomposition. Saponified by alkalis, it gives an acid, similar to, but not identical with ouabaic acid, and which is hydrolysed by boiling weak mineral acids, giving a resin insoluble in water, and a considerable amount of rhamnose. Analysis points to the formula $C_{44}H_{58}O_{18}$, and shows that the body with this formula contains seven acetyl groups. Ouabaïn $C_{30}H_{46}O_{12}$ appears thus to lose $H_2O$, and then form the derivative $C_{30}H_{37}(C_2H_3O_7)O_{11}$. The molecule of ouabaïn, then, contains nine hydroxyl groups, four belonging certainly to the rhamnose group $CH_3(CHOH_4).CHO$ contained in the glucoside, the other five belonging to the group of unknown constitution which forms the rest of the molecule. The amorphous acetin seems to be a derivative of a body formed from ouabaïn by yet further dehydration.

—J. T. D.

### *Ouabaïn [Strophanthin]. Nitro-Derivatives of; obtained by the Action of Nitric Acid.* Arnaud. Comptes. Rend. 1898, **126**, [26], 1873—1876.

By the action of concentrated nitric acid, ouabaïn is completely oxidised even in the cold, with the formation of oxalic acid, carbonic acid gas, and insoluble amorphous nitro-derivatives. With more dilute acid however (sp. gr. 1·2) no oxalic acid is formed, but several insoluble nitro-compounds are obtained, some of which are crystalline. One of these having the composition $C_{23}H_{24}(NO_2)_2O_6$, crystallises from acetone in yellowish, anhydrous, silky needles melting at about 300° C. It is obtained by digesting ouabaïn with the acid, first at 40° C., then at 70°—75° C., the latter temperature not being exceeded. It acts as an acid, and forms crystalline salts. Another crystalline body $C_{23}H_{25}(NO_2)O_6$ is obtained by the prolonged action of the same acid on ouabaïn in the cold, at a temperature not exceeding 15° C. In the course of several days, about 1 per cent. of transparent crystals of this body are formed, together with five or six times that amount of the dinitro-product before mentioned. The ammonium salt of this mono-nitro product is very insoluble, so that it may be precipitated from a solution in dilute sodium hydrate by the addition of ammonium chloride. The salt thus precipitated has the composition $C_{23}H_{24}NH_4(NO_2)O_6$, and occurs in small, yellow, anhydrous crystals. These two nitro-bodies are regarded as being primarily derived from the body $C_{21}H_{36}O_8$ which results from the hydrolysis of ouabaïn thus:—$C_{30}H_{46}O_{12} + H_2O = C_6H_{12}O_5 + C_{24}H_{36}O_8$. In the presence of nitric acid, this last product is both oxidised and nitrated, forming the mono- and di-nitro compounds above described, with the elimination of one atom of carbon. Other products of oxidation and soluble nitro compounds are formed at the same time, are now under investigation.

—J. O. B.

### *Halogen Albumin Derivatives.* F. Blum and W. Vaubel. J. prakt. Chem. 1898, **57**, [8 and 9], 365—396.

The results of the investigation are shortly expressed as follows:—The albumins possess a considerable affinity for the halogens, reacting with them as soon as they come in contact. In this reaction large quantities of the haloid acid are formed together with a substituted albumin. After removal of the free acid the albumin molecule is enabled to take up more halogen, thus by conducting the reaction in a solution kept neutral throughout, a derivative is obtained containing a constant quantity of combined halogen. The halogen atoms enter a benzene nucleus containing a hydroxyl group, the nucleus then ceases to give Millon's reaction. The portion of the albumin molecule which gives Millon's reaction may be separated from the portion containing sulphur; both portions retain the general characters of the albumins and both give the biuret reaction. Of the two groups in the molecule which can give the biuret reaction, one only is rendered incapable of doing so by the halogen substitution; if the molecule be split up by the action of alkalis, the group giving the biuret reaction is found in the portion containing sulphur.

—A. C. W.

### *Absinthin.* P. Bourcet. Bull. Soc. Chim. 1898, **19**, [12], 537—539.

Various formulæ have been ascribed to this glucoside which was first extracted by Braconnot from the leaves of the great absinthe (*Artemisia absinthium*). Duqusenel was the first to prepare it in a pure and crystalline state. (*Bull de therap.* **107**, 438), and the author has now obtained a pure preparation by a different process. The dried and powdered leaves were extracted with ether, and the viscous solid left on evaporation of the ether was mixed with twice its weight of sand and extracted with chloroform. On evaporating the latter a greenish residue was left which was dissolved in alcohol. An alcoholic solution of lead acetate was then added, the precipitate separated by filtration, and the excess of lead removed from the filtrate. The clear yellow solution thus obtained was digested for several days with gelatinous alumina, after which it was filtered and the alcohol evaporated. The residue consisted of absinthine with traces of resinous matter which prevented it from crystallising. It was freed from the latter by dissolving it in alcohol (90 per cent.), and employing fractional precipitation with brine, the latter precipitates containing the absinthine in the form of a pure white powder. This powder dried *in vacuo* was dissolved in absolute alcohol in order to free it from sodium chloride, sufficient water to just produce a turbidity was added to the solution and the liquid was left over sulphuric acid. After a very long time (often several months), white silken crystals were deposited, which were washed with dilute cold alcohol and dried. The yield was very small. As thus obtained, absinthin was a white inodorous body crystallising in fine prismatic needles, and characterised by a marked and persistent bitter taste. It melted at 68° C. It contained, according to the mean results of several analyses, carbon, 67·83; hydrogen, 7·82; and oxygen (by difference) 24·33 per cent. These figures correspond to the formula $C_{15}H_{20}O_4$, which is the same as that given by O. Senger. Cryoscopic determinations of absinthin in acetic acid gave the molecular weight as 258 and 261, figures which are very close to 264.—C. A. M.

### *Maracaïbo-Copaiva Balsam. Deutsch. Arz. Buch. III.*

K. Dieterich. Helfenberger Ann. 1897, 45—55; Chem. Centr. 1898, 1, [23], 1205.

Good Maracaibo copaiva balsam should, according to the author, conform to the following values:—Sp. gr., 0·9800—0·9960; acid number, 75·85; ester number, 3—6; saponification number, 80—90. In old goods the specific gravity, acid number, and saponification number are increased, whilst various adulterants, as Gurjun balsam, olive oil, castor oil, sassafras oil, turpentine oil, turpentine, colophony, and paraffin, influence the values in different directions.—A. S.

### *Parà-Copaiva Balsam.*

K. Dieterich. Helfenberger Ann. 1897, 56-62; Chem. Centr. 1898, 1, [23], 1205--1206.

The author fixes the following approximate limits:—Sp. gr., 0·95—0·97; acid number, 30—60; ester number, 2—8; saponification number, 30—65. The chief adulterant of the Parà balsam is Gurjun balsam, for which the following limiting numbers are given:—Sp. gr., 0·955—0·965; acid number, 5—10; ester number, 1—10; saponification number, 10—20. Gurjun balsam is thus essentially different from Maracaibo (*see* preceding abstract) and Parà copaiva balsams; Parà copaiva balsam stands between the Maracaibo and Gurjun varieties.—A. S.

### *Cascara Sagrada, Chemistry of.*

A. R. L. Bohme and H. Engelhardt. J. Amer. Chem. Soc. 1898, 20, [7], 534—546.

The *Cascara sagrada* bark used in the investigation was gathered in Oregon. Dried at 110° C. it lost 8·3 per cent. of moisture. On incineration it left 7 per cent. of ash. A chloroform extract was made in a specially constructed extraction apparatus. 7·5 per cent. of the air-dried drug was thus extracted, and the extract subjected to a steam distillation. An oil passed over with the chloroform, from which mixture it was separated, and then redistilled *in vacuo*. The oil possesses the characteristic smell of the drug. No alkaloids or glucosides were found in the residue from the steam distillation. This residual oil was saponified, and extracted with ether, and on evaporation of the ether, an oil was obtained which soon went solid, and on recrystallisation from alcohol gave white leafy crystals with m.p. 24°—26° C. Analysis showed this to be a dodecyl alcohol, and the m.p. points to the normal compound. The acids which are combined with this alcohol to form the oil are, according to the analyses, &c., palmitic and stearic. The residue from the chloroform extraction was extracted with 80 per cent. alcohol to the extent of 27·5 per cent. of the original air-dried bark. After distilling off the alcohol a hard residue, possessing the characteristic bitter taste of *cascara*, was left. This was treated with absolute alcohol, which dissolved the greater portion of it. This alcoholic solution was evaporated to dryness, and the residue dissolved in warm water. This solution, with basic lead acetate, gave a reddish-brown precipitate, which the authors believe consists of a mixture of the lead salt of the glucoside and the lead salts of the tannates present. It was filtered off, stirred up with water, and decomposed by sulphuretted hydrogen. The filtrate from the lead sulphide on evaporation left a brown amorphous residue which is soluble in alcohol, acetone, or ethyl acetate, and recrystallised from any of these, a small quantity of dark brown needles was obtained. This is the glucoside, and the name *purshianin* is proposed for it. The residue from the alcoholic extraction was macerated for 12 hours with water. 12·3 per cent. of the original bark was thus extracted. The undissolved portion was boiled with dilute sulphuric acid (to invert the starch), then filtered. The residue was extracted with dilute caustic potash solution. In this way 21·3 per cent. of the original bark was extracted, and on treating the residue with hypochlorite 16·1 per cent. of white cellulose was obtained. By difference, the air-dried bark contained 7 per cent. of starch, &c.

*The Glucoside.*—To obtain this the drug was extracted first with chloroform, then with 80 per cent. alcohol. The alcoholic extract was dried, then dissolved in hot water. On cooling, some resinous, waxy substance separated, and was filtered off. The filtrate gave, with lead acetate, a yellow precipitate, which was filtered off and stirred with hot water on a water bath whilst sulphuretted hydrogen was passed through. The lead sulphide was filtered off and the filtrate evaporated to dryness. The residue consisted mainly of tannins, and was not further investigated. The filtrate from the lead tannin with basic lead acetate gave a dark red-brown precipitate of lead glucoside. The precipitate was stirred with hot water and decomposed with sulphuretted hydrogen. The filtrate from the lead sulphide on evaporation left a hard brown substance, a small quantity only of which was obtained in the crystalline form by separation from ethyl acetate or acetone. These needle-shaped red-brown crystals had m.p. 237°. Sufficient was not obtained to make an analysis, but it certainly was not emodin. This glucoside, on heating with alcoholic hydrochloric acid, gives a sugar and emodin (the emodin was obtained in the form of reddish-yellow needles with m.p. 254°; but the sugar has not been identified). Purshianin is tasteless, odourless, soluble in alcohol, ethyl acetate, acetone, alkalis, and hot water. It resembles somewhat Thorpe and Robinson's frangulin, which is an orange-yellow powder with m.p. 225°, and which, on saponification, also yields emodin.—J. McC.

### *Turpentine, Oxidised.*

R. Robine. Rev. de Chim. Ind. 1898, 9, [102], 163—166.

The substance treated of in this paper is the product of the oxidation of the ordinary commercial turpentine, and so far back as the last century was believed to have special solvent power for copal resins. But the author's experiments have led him to conclude that any increase of solubility which takes place on leaving the resins in contact with the oil is due to oxidation of the former. Apart from its being used for painting on porcelain it has hardly any industrial applications. The only known process of manufacture in use consists in exposing the ordinary oil to the action of the air until a sufficient degree of oxidation has been reached. This is a tedious and costly process, for the oxidation requires months, and the loss by evaporation amounts to about two-thirds of the oil employed. Certain manufacturers shorten the process and obviate some of the loss by methods which they keep profoundly secret, but which, the author states, all consist in forcing air through the oil under predetermined conditions of temperature and pressure. In this way the oxidation can be carried out in days instead of months, but the danger of working at high temperature and pressure with such inflammable material is considerable, and the loss still amounts to about one-third of the original raw material.

Oxidised turpentine is a liquid with the appearance and colour of honey and an odour of turpentine. Its density is near that of water. It is soluble in turpentine, alcohol, ether, and chloroform, of which the last appears to be the best solvent. When heated it becomes more fluid, and at 130° C. froths abundantly from the presence of water; then the temperature suddenly rises and the product becomes brown. On cooling it is more viscous than before. On continuing the heating, the water distils over first, then yellow-coloured substances with an aromatic smell recalling that of lemon, and a resinous dark brown residue which solidifies on cooling is left behind. The liquid portion distils between 120° and 200° C., the bulk passing over at about 160° C.

The oxidised turpentine possesses certain properties which might be utilised in various manufactures. It dissolves nitro-cellulose readily at a temperature of 70°—80° C. The presence of alcohol favours the solution, but turpentine and toluene interfere. A mixture of alcohol and toluene, however, dissolves nitrocellulose. The oxidised body thus appears to play a part analogous to that of camphor in the manufacture of celluloid, favouring the solution of the cotton, and the author suggests that it might possibly be employed in place of camphor, with the advantage that celluloid or a celluloid substitute would be obtained at a much smaller price.

The resins required in the manufacture of varnishes are rendered soluble by being highly heated, the drawbacks of which process are that they lose about 25 per cent. in weight, and become more or less coloured, whilst the operation requires the most careful attention. Since the oxidised turpentine has the property of rendering soluble in turpentine certain resins which have only been heated so far as to lose from 8 to 10 per cent. in weight, there would be a great advantage in its employment. In experiments, the fusion has been carried out in glass retorts heated on a lead bath at 360°—380° C. The operation is stopped as soon as 8 per cent. of a yellow oily liquid has distilled over, and the residue is then heated at 100° C. with the copal, with constant agitation. The product is translucent and of a deep maroon colour, and dissolves readily in hot turpentine, giving a clear yellow solution. This property is possessed by the hard copal resins of Africa and Calcutta, but not by the soft copals of Sydney and Manilla, which, however, have a wider commercial application. The author mentions that resins prepared in this way and directly soluble in turpentine are to be bought in Germany, but that the processes by which they are manufactured are kept secret.

—C. A. M.

*Violet Oil from Lemon Grass Oil.* J. Ziegler. J. prakt. Chem. 1898, **57**, [10 and 11], 493.

In the preparation of "violet oil," instead of starting with citral as recommended by Tiemann, the author employs ordinary lemon grass oil, or preferably the oil freed from citral by fractionation, and having a boiling point of 115°—130° C. at 15 mm. pressure. This oil is then heated with acetone, chlorinated lime solution and alcohol, with the addition of a little cobalt nitrate. The product obtained differs from the pseudo-ionone of Tiemann, having a higher boiling point, 155°—170°, under reduced pressure (12 mm.), and a lower gravity, 0·898. This product, termed by the author "pseudo-violet oil" has the composition $C_{13}H_{20}O$. When subjected to prolonged boiling with hydrogen sodium sulphate solution, it is converted into an isomeric body, exactly as pseudo-ionone is converted into ionone. This isomer appears to be identical with "technical violet oil," which has been introduced into commerce. When purified by fractionation, it forms a yellowish green oil, having an odour somewhat recalling sandal wood oil. It boils at 140°—150° C. under 12 mm. pressure, and at 271°—273° at 760 mm. and has a specific gravity of 0·952 at 15° C. These figures differ from those of ionone obtained by Tiemann; it also forms a semi-carbazone like ionone, which however, melts at 142° C.; whereas the ionone semicarbazone melts at 109°—110° C. This "violet oil," therefore, is not identical with ionone, and it is stated to surpass that body as to purity and strength of its violet-like odour.

—J. O. B.

*α-Ionone, The Semicarbazone of.* F. Tiemann. Ber. 1898. **31**, [11], 1736—1739.

The semicarbazones of certain aldehydes and ketones, *e.g.*, citral and ψ-ionone, cannot well be used for the identification of these compounds, because they exist in modifications of different crystalline form and melting point. This is now found to be the case also with α-ionone. Pure α-ionone (this Journal 1898, 599) yields a semicarbazone melting at 107°—108° after recrystallisation from petroleum ether. The melting point, however, rises on repeated recrystallisation, from 60 per cent. alcohol, and becomes constant at 137°—138° C. The α-ionone separated from this high melting semicarbazone is pure, and identical with the compound obtained from the low melting semicarbazone.—A. C. W.

*Peru and Copaiva Balsams, Examination of; Especially for the D. A. B III.* K. Dieterich. Helfenberger Ann. 1897, 69; Chem. Centr. 1898, [23], 1206.

*See under* XXIII., *page* 806.

*Sumatra Benzoin.* K. Dieterich, Helfenberger Ann. 1897 76; Chem. Centr. 1898, **1**, [23], 1,207.

*See under* XXIII., *page* 806.

*Siam Benzoin.* K. Dieterich. Helfenberger Ann. 1897, 87; Chem. Centr. 1898, **1**, [23], 1,207.

*See under* XXIII., *page* 807.

*Balsams, Resins, and Gum Resins, Examination of.* K. Dieterich. Helfenberger Ann. 1897, 105; Chem. Centr. 1898, **1**, [23], 1,205.

*See under* XXIII., *page* 807.

*Lichens; Contribution to the Knowledge of the Constituents of.* O. Hesse. J. prakt. Chem. **57**, [10 and 11], 409.

*See under* XXIV., *page* 807.

*Tropine Pinacone.* B. Willstätter. Ber. 1898, **31**, [11], 1672.

*See under* XXIV., *page* 808.

*Mixed Carbonic Esters of the Fatty and Aromatic Series, General Method for the Preparation of.* P. Cazeneuve and A. Morel. Comptes Rend. 1898, **126**, [26], 1871.

*See under* XXIV., *page* 808.

## PATENTS.

*Scent, Artificial or Musk-like Substance, A new Method for Producing.* J. W. Mackenzie, London. From C. Schmid, Brussels, Belgium. Eng. Pat. 10,535, April 27, 1897.

The process is claimed for the production of a substance of musk-like odour, which consists in the oxidation of the mixture of oily and waxy products obtained by the distillation of Zanzibar copal, amber, or retin-resin at 240°—350° C. by steam at one atmosphere pressure. The oxidation is performed by slowly (for 5 hours) stirring 1 part of powdered potassium bichromate into a mixture of 1 part of the product with 10 parts of strong sulphuric acid, and filtering through asbestos when cold. The filtrate is neutralised by a mixture of equal parts of alcohol and ammonia, the alcohol driven off and the odoriferous bodies extracted by ether, from which they separate as an oil on evaporation. The product is also claimed.

—A. C. W.

*Purine, Amino-Derivatives of; Impts. in the Manufacture of.* J. Y. Johnson London. From C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 18,494, August 9, 1897.

The manufacture of amino-purines, by causing ammonia to act on a chloro-purine containing the chlorine in the alloxan nucleus, is claimed, as also the products, as new compounds.

—A. C. W.

*Artificial Camphor, a Process for the Treatment of; either in the Crude State or after Re-sublimation.* T. Bird, London. Eng. Pat. 18,698, Aug. 12, 1897.

The manufacture of an altered artificial camphor from crude terpene hydrochloride, is claimed, by a process which consists in placing it in a suitable electrolyte (preferably water) and subjecting it to the action of an electric current. The mixture of camphor and water so obtained is then heated; the purified camphor sublimes.

—A. C. W.

*Ionone or Pseudoionone, Manufacture of a new Product from.* E. de Laire, Paris, France. Eng. Pat. 23,719, October 14, 1897. (*See* this Journal, 1893, 542, 1898, 599.)

The manufacture of iso- or *β*-ionone by treating ionone or *α*-ionone or *ψ*-ionone with more or less concentrated acid, and the product as a new article of manufacture, are claimed. The process is as follows:—Ionone (Eng. Pat. 8736, 1893, this Journal, 1897, 1038) is slowly dropped, with constant stirring, into 3—4 parts of cooled sulphuric acid (66° B.), the temperature being allowed to rise to 30° C., the mixture is poured into water, the solution extracted by ether, the ethereal solution washed, neutralised, and the residue, after removal of the solvent, steam distilled. The mixture of ionone and isoionone so obtained is fractionated, and the portion boiling between 135°—145° C., under 18 mm. pressure is collected as crude isoionone. This may be further purified by recrystallisation of the semicarbazone. Pure iso-ionone boils at 127°—128·5° C., under 10 mm. pressure, has the specific gravity 0·946 at 17° C., and the index of refraction is $n_D = 1 \cdot 521$. The *p*-bromophenylhydrazone melts at 114°—115° C., and the semicarbazone at 118° C.—A. C. W.

*Seaweed, Impts. in the Process of Manufacturing Valuable Organic Products from ["Tang-Acid"].* A. Krefting, Christiania, Norway. Eng. Pat. 12,275, May 31, 1898.

An improvement on the process described in Eng. Pat. 1896, 11,538, which consists in the application of a solution of hypochlorite of soda, potash, or magnesia, to dissolve the acid-treated seaweed, or in the use of an electrolysed solution of sodium, potassium, or magnesium chlorides or of electrolysed sea-water.—A. C. W.

*Sea-weed, Impts. in the Manufacture of Valuable Organic Products from ["Tang-Acid"].* A. Krefting, Christiania, Norway. Eng. Pat. 12,277, May 31, 1398.

The claim is for the improvement in the manufacture of products from sea-weed (Eng. Pat. 1896, 11,538), which consists in the application of solutions of acid and alkali of not more than 2—3 per cent. strength.—A. C. W.

*Cellulose, Hydrocellulose, and Oxycellulose, Nitration of.* L. Vignon. Comptes Rend. **126**, [23], 1658.

*See under* XXII., *page* 794.

## XXI.—PHOTOGRAPHY.

### PATENTS.

*Photographic Paper, Plates, and Diapositives, Impts. in and connected with.* M. Jolles and L. Lilienfeld, Vienna. Eng. Pat. 11,053, May 4th, 1897.

For the film to be sensitised, the authors claim the use of albuminous substances of vegetable origin (fibrine, mucedine, gliadine) soluble in alcohol, which are either deposited on the paper suitably salted and afterwards sensitised, or the sensitising may be done by pouring into the salted alcoholic solution a hot alcoholic solution of silver nitrate, followed by a proportion of glycerine. This emulsion is then applied to the paper and dried. Paper so prepared keeps well, is very sensitive, is not easily scratched, can be treated at a high temperature if desired without blistering, and needs no special bath for toning.—J. T. D.

*Sensitised Photographic Paper, Treatment of; A New and Improved Process for.* A. Schwarz, Berlin, Schöneberg. Eng. Pat. 25,933, Nov. 8th, 1897.

An arrangement of four baths, vertically above one another, for toning, fixing, and washing rolls of printed paper. The roll is fed from a bracket into the first trough by feeding and guide rollers, travels then in the opposite direction through the second, and similarly through the third and fourth baths. The paper is visible during the whole of the treatment, and the rate of travel of all the feed and guide rollers is under the control of the operator. In the third and fourth baths a stream of water passes through, in each in a direction opposite to that of the travel of the paper. As the paper emerges from the fourth bath it passes through a heating chamber so as to be thoroughly dried, and is finally wound upon a roll.—J. T. D.

*Embossing and Colour Decoration; Impts. in the Preparation of Paper for, chiefly in Connexion with Photography.* M. W. McDonald, Cleveland, Ohio. Eng. Pat. 27,033, Nov. 18th, 1897.

Paper with a glazed or burnished surface, or ordinary glazed sensitised photographic paper, has the surface ground off by the careful application, by hand, of finely ground emery or pumice-stone, till the glaze just disappears, and a perfectly matt surface remains. The paper (if sensitised) can then be printed and toned in the usual way, or can be embossed or coloured, and gives very soft gradations of tone.—J. T. D.

*Photographic and other Films, Improvements in Apparatus for Manufacturing.* H. M. Reichenbach, Rochester, New York. Eng. Pat. 4445, Feb. 22, 1898.

An apparatus consisting of two chambers, with a passage between them, wherein large sheets of aluminium are placed side by side or one above another, and are so arranged that they can be supplied by means of a system of belts or rollers passing from one chamber to the other, in either direction, across the passage already referred to. In the passage is a carriage, capable of motion both vertically and also horizontally in a direction at right angles to the motion which is communicated to the aluminium sheets. As the sheets pass to and fro they are cleaned, covered evenly with nitrocellulose in a semi-fluid condition, coated (after the nitrocellulose has solidified through the evaporation of its solvent) with emulsion, and finally (the emulsion having stiffened) they are stripped of the coated film, which is wound upon rollers ready to be cut up as desired. It is proposed to give a thin coating of petroleum spirit or benzene to each cleansed aluminium plate, to prevent the nitrocellulose from sticking thereto; and the devices for spreading the nitrocellulose and coating the plate with emulsion, consist of hoppers with valves for delivering the liquid and a special form of scrapers for regulating the thickness of the film. In each chamber are coils of pipe, through which steam or a refrigerating liquid may be circulated, to hasten the evaporation of the solvent of the nitrocellulose or the solidification of the emulsion, as the case may be.—J. T. D.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Cellulose, Hydrocellulose, and Oxycellulose, Nitration of.* L. Vignon. Comptes Rend. **126**, [23], 1658—1661.

Cellulose and its hydro- and oxy- derivatives are treated with a mixture of three parts of sulphuric acid and one part of nitric acid at 10° C., and the nitration carried to its maximum. About equal weights of product were obtained from equal weights of the original substances. The products contained approximately equal quantities of nitrogen. The products "were all inflammable"; they all gave Schiff's reaction, but did not respond to this test more definitely than the original substances; the derivatives of cellulose and hydrocellulose reduced Fehling's solution on boiling, that of oxycellulose more energetically and below the boiling point. Caustic potash of density 1·3 slowly attacks the nitrated cellulose, which dissolves to a clear yellow solution. The nitrated hydrocellulose is attacked more rapidly, and the nitrated oxycellulose more rapidly still, giving a brown solution with evolution of gas, and elevation of temperature to about 60° C. Distilled with hydrochloric acid, all three nitrates give off great quantities of nitrous fumes, and the distillate in each case shows small amounts of furfural, more in the case of the oxycellulose derivative than with the other two. Three successive nitrations of the product (well dried before each immersion in the acids) gave practically no increase in the proportion of nitrogen. 1 grm. of the dried substance yielded 208—213 c.c. of nitric oxide; the fixation of 11 $NO_2$ for a molecule of cellulose of $C_{24}$ would correspond to 214 c.c. of nitric oxide from 1 grm. of the product. It seems, then, that cellulose, in its conversion into hydrocellulose or oxycellulose, does not undergo any profound modification of its molecular structure.—J. T. D.

### PATENTS.

*Danger from Explosion in Vessels charged with Explosives, Means for Preventing.* The Compagnie Française de l'Acetylene dissous, Paris. Eng. Pat. 20,668, April 5, 1897.

"This invention consists in filling a container before the introduction of the explosive, with a porous, filamentous, pulverulent, or equivalent inert material of such a kind as to leave between its compouent parts or within its substance only very small interstices." The explosive, gas, for example, is then forced in and is absorbed by the filling substance.
—W. M.

*Blasting and Blasting Cartridges, Improvements in.* J. Macnab, London. Eng. Pat. 16,777, July 15, 1897.

A cartridge for blasting is described, preferably charged with meal guncotton, in which is embedded one or more glass tubes filled with a strong solution of ammonia. A safety tamping pellet of the same diameter as the cartridge, composed of common salt or ammonium oxalate, and provided with a hole through which the fuse can pass, is either laid on the top of the explosive cartridge or wrapped up in the case along with the explosive. For use in coal pits the cartridge is enclosed in a strong metal case, and detonation is said to be brought about by an ordinary fuse without the aid of a detonator.—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

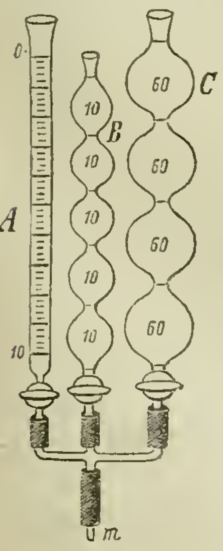
### APPARATUS, ETC.

*Glass Test Tubes, &c., Alkaline; Warning Against.* C. Liebermann. Ber. **31**, [11], 1818.

One of the largest glass merchants in Germany is sending out test tubes made of glass so alkaline that the ordinary indicators when placed therein immediately reveal the presence of alkali. Even after cleaning, the same reaction occurs directly the liquid contents are boiled. The tubes, which are of very good appearance, clear, easy to clean, and never crack, are suitable for lecture experiments to show the reaction of dyestuffs (*e.g.*, Magenta), with alkali. Washing with 5 per cent. nitric acid for 12 hours is only of temporary utility, alkali being again extracted when the contents of the tubes are boiled; even five hours' boiling with the same acid affords no permanent remedy, though slight devitrification is produced.

Preparation tubes, apparently of the same glass, employed for storing acid dyestuffs, become coated internally with a deposit of the corresponding sodium compound.—C. S.

*Measuring Liquids.* O. Bleier. Chem. Zeit. 1898, **22**, [38], 376. (See also this Journal, 1898, 272 and 376.)



The apparatus consists of a burette with three arms, as shown in the figure; by its use large quantities of liquids may be measured with precision. Each bulb of the limb B holds as much as (or less than) the graduated scale of A, and each bulb of C as much as A and B together.

In measuring definite quantities of liquid, the latter is drawn from the largest limb first, from mark to mark; where indefinite quantities are withdrawn and the level remains between two marks in C or B, it is necessary to bring up the level to the mark (next above or below, in C), by blowing in air and thus displacing liquid in A and B to C, &c., before taking the reading. The amount of liquid used for the titration is thus equal to the sum of the readings in the three limbs.—L. J. de W.

### INORGANIC CHEMISTRY.—QUANTITATIVE.

*Concentrated Sulphuric Acid by Mercury at ordinary Temperatures, Decomposition of.* [Gas Analysis.] C. Baskerville and F. W. Miller. J. Amer. Chem. Soc. 1898, **20**, [7], 515.

Reply to J. R. Pitman's objection (J. Amer. Chem. Soc. 20, 100) to the authors' former work.

The acid used by the authors in their work, contained 99·65 per cent. $H_2SO_4$. Placed in an air-tight stoppered bottle with pure mercury, in 24 hours sufficient sulphur dioxide was evolved to bleach a very dilute solution of potassium permanganate.

The action of sulphuric acid on mercury depends entirely on the strength of the acid. 95 per cent. acid seems to be without action on mercury within a reasonable time. In ordinary gas-analysis 94 per cent. acid serves very well for desiccation, as it appears to be unacted upon by mercury.
—J. McC.

*Perchlorate in Saltpetre, Estimation of.* F. Freytag. Zeits. für öffentl. Chem. **4**, 321–323; Chem. Centr. 1898, **1**, [23], 1,203 (*See also* this Journal 1898, 275 and 694).

The saltpetre to be examined is finely pulverised and passed through a silken sieve. The chlorine is determined in 5 or 10 grms. by Volhard's method. A further portion (5 or 10 grms.) is heated over a small flame in a porcelain crucible that is supported in a piece of asbestos card, so that about two-thirds of the crucible projects above the asbestos. After placing the lid on the crucible, the flame is increased till a moderate evolution of oxygen begins; the half flame of an ordinary Bunsen burner is usually sufficient. In using the method for the first time it is preferable to cover the crucible with a small watch glass, in order that the course of the reaction may be observed. After about 20 minutes, or when the evolution of oxygen is extremely slow, the flame is extinguished, and the crust adhering to the lid and the upper part of the crucible scraped into the latter by means of a platinum spatula. The crucible is then heated for 10 minutes, with three-quarters of the full flame, and the melt made up to 300 c.c. with water. No appreciable formation of nitrite is said to occur, but it is recommended in all cases to acidify the solution with 2—3 c.c. of nitric acid (sp. gr. 1·24), which has been previously boiled, and then to boil for 10—15 minutes. After cooling, the chlorine is determined volumetrically by Volhard's method, and the increase of silver solution as compared with the first titration indicates the amount of perchlorate. The iodate content of the refined saltpetre is usually less than 0·01 per cent.; if it be appreciably higher, the iodine is determined in the original sample by means of potassium iodide and thiosulphate, and the equivalent quantity of chlorine deducted from that used for calculating the amount of perchlorate.

It is stated that if the above method be carefully carried out, an addition of alkali in presence of magnesium chloride is unnecessary. The method gives satisfactory results, the limit of error being from −0·09 to +0·08 per cent. of $KClO_4$: average −0·01 per cent.—A. S.

*Perchlorate, Determination of; in Sodium or Potassium Nitrate.* N. Blattner and J. Brasseur. Bull. Soc. Chim. 1898, **19**, [12], 539.

The chlorine present as sodium chloride is first determined in the usual way. From 5 to 10 grms. of the sample dried and finely pulverised, are then heated in a covered platinum crucible over a Bunsen burner for about 15 minutes with from 8 to 15 grms. of pure calcium oxide. When cold the mass is dissolved in dilute nitric acid and the total chlorine determined volumetrically or gravimetrically. The difference between the two chlorine determinations gives the amount present in the form of perchlorate (or chlorate, if any be present). The tabulated results of test mixtures prepared by the authors are very satisfactory.—C. A. M.

### *Manganese precipitated by the Chlorate Process, Oxidation of the.* F. A. Gooch and M. Austin. Amer. J. Science, 5, [4], 260—268; Chem. Centr. 1898, 1, [23]. 1203—1204.

The method of estimating manganese by precipitating it from its nitrate solution by potassium chlorate has up to the present been considered unsatisfactory, and the authors state that this is due to the uncertain degree of oxidation of the manganese precipitate and to mechanical retention by the latter of the slightly soluble chlorate. The latter difficulty may be overcome by the use of sodium instead of potassium chlorate. A sample of manganese nitrate, free from sulphate and chloride, was boiled for five minutes with 85 c.c. of concentrated nitric acid and 5 grms. of sodium chlorate, a further 15 c.c. of nitric acid and some more crystals of chlorate were then added, and the heating discontinued. On examination of the filtrate with KI and $As_2O_3$, only 0·001 grm. of Mn. was found, although if the solution were boiled for a longer period, the loss increased from 0·001 to 0·003 grm. The authors recommend the use of an excess of chlorate, and an asbestos filter for the filtration of the concentrated nitric acid solution.

The authors found that the limits of error in estimating manganese by means of, (1) acidified iodide, and, (2) iodide and $As_2O_3$, were (when using pure salts of manganese:—(1) from − 0·0004 to + 0·0007 grm. and (2) − 0·0004 to − 0·0008 grm. If the manganese compounds be contaminated with iron, it is preferable to use the chlorate method. Assuming that the manganese is precipitated in the chlorate process as $MnO_2$, it was found by experiment that 0·0017—0·0056 grm. of Mn. was lost, whilst the filtrate contained at the most only 0·00008 grm., so that it is clear that the precipitate does not consist of pure dioxide. In order to obtain oxygen in the same proportion as in the dioxide, the authors made use of the fact that potassium permanganate at 80° C. in presence of zinc sulphate carefully mixed with manganese sulphate yields an oxide, which, although combined to the alkali, contains the oxygen in a proportion exactly corresponding to that of the dioxide (Wright and Menke, J. Chem. Soc. **37**, 36). Three-fifths of the manganese fixed in the precipitate so obtained, represents the amount of manganese originally present in the manganese salt. By careful work, the authors obtained in this way very good results (limits of error − 0·0012 to + 0·0009).—A. S.

### *Iron from Manganese, Aluminium, &c., Electrolytic Separation of.* M. Engels. L'Éclairage Électr., 1898, **14**, [106]. (From Dissertation, Berne.)

*Separation of Iron from Manganese.*—Iron cannot be separated from manganese by the addition to the electrolyte of oxalic acid (Classen), acetic acid (Becquerel), alkali and tartaric acid (Vortmann), citric acid or pyrophosphate solution (Brand). The only method which effects a complete separation is as follows:—The solution to be electrolysed is placed in a platinum basin, which serves as cathode, and connected by means of a siphon with very dilute sulphuric acid contained in an adjacent glass vessel, into which dips a platinum anode. A current of 0·4—0·8 amp. at 40—60 volts is passed through the apparatus for 13 hours, and at the same time a current of hydrogen is led through the anode compartment in order to prevent the formation of permanganic acid. The anode is removed about once an hour, and after washing with water, the manganese peroxide deposited on it is removed by means of a warm solution of oxalic acid. In this way two solutions are finally obtained, one containing all the manganese, and the other all the iron, in which the metals are determined as usual.

*Separation of Iron from Aluminium.*—This separation is best effected in a solution to which just sufficient, and no more, Rochelle salt has been added, to form the double salt. After making slightly alkaline, a current of 1·6 amp. at 2·2 volts is used during the electrolysis. The method succeeds even when phosphoric acid is present.

*Separation of Iron (and Manganese) from Nickel.*—Ammonia is added to the solution of the peroxidised sulphates until the precipitated nickel hydroxide is redissolved. The unfiltered liquid is then electrolysed with a current of not more than one ampère. After the nickel has been completely deposited, the ferric (manganese) hydroxide can be filtered off and weighed.

Cobalt cannot be separated in this way, and chromium cannot be separated from iron by the methods of Classen and Smith. The separation of zinc from iron can, however, be effected by Vortmann's method.—J. S.

### *Manganese in Iron and Ores, Use of Hydrofluoric Acid in the Determination of.* A. P. Ford and I. M. Bregowsky. J. Amer. Chem. Soc. 1898, **20**, [7] 504.

The authors recommend the following method for the determination of manganese in iron or steel:—The manganese is precipitated in the usual way with potassium chlorate, then a few drops of hydrofluoric acid are added. The solution is then boiled until the hydrofluoric acid is evaporated. About 1 grm. more of potassium chlorate is added, and the liquid evaporated a little further. The liquid thus obtained filters well, and the manganese dioxide precipitated, is readily soluble in ferrous sulphate, which the authors recommend in preference to oxalic acid. The graphitic carbon is entirely oxidised, because the enveloping silica is removed. In the ordinary method the latter forms a protective coating.

In the case of ores it has been found that the first addition of potassium chlorate does not completely precipitate the manganese, but if, a minute or two after the addition of the chlorate, a few drops of hydrofluoric acid be added, then complete precipitation is ensured.—J. McC.

### *Steel, Note on Drown's Method of Determining Silicon in.* G. Auchy. J. Amer. Chem. Soc., 1898, **20**, [7], 547.

In using Drown's method, sometimes the silicon is not all obtained. If, however, instead of evaporation with nitric and sulphuric acids, the evaporation be performed with *aqua regia* and sulphuric acid, then the iron salt is kept in solution till the very last moment, when it suddenly crystallises out. It is reasonable to suppose that this method is more favourable to a complete dehydration of the silica than the nitro-sulphuric acid process in which the iron sulphate separates out before the complete expulsion of the nitric acid. If a second evaporation be made before filtering, it may confidently be expected that no notable amount of silica will escape dehydration. To carry out the method the steel is dissolved in nitro-sulphuric acid, then hydrochloric acid is added and the mixture evaporated down rapidly on a very hot iron plate. The evaporation goes on without bumping or sputtering.—J. McC.

### *Lead in Alloys. Determination of.* W. E. Garrigues. Eng. and Mining J. 1898, **65**, [26], 761.

The following method is described, by which it is stated the lead in alloys can be determined without first removing the tin:—0·5 grm. of soft solder (containing lead 58·83 and 58·80 per cent. by the usual sulphate and chromate methods respectively) was oxidised with nitric acid, 20 c.c. of strong sulphuric acid added, and the solution evaporated till dense fumes were evolved. The metastannic acid was completely dissolved. 80 c.c. of cold water were added, the solution stirred for a few minutes and the lead sulphate filtered off on a Gooch crucible. The result indicated 59·00 per cent. of lead. The sulphuric acid filtrate was made alkaline with ammonia, then again acid with sulphuric acid, but only sufficiently so to turn methyl orange a decided red. The voluminous white precipitate of metastannic acid thus obtained, was filtered off, after first boiling the liquid, and on ignition over the blast, gave stannic oxide equivalent to 41·10 per cent. of tin. If much more than 80 c.c. of water was used for diluting the fuming acid, or if the diluted liquid was allowed to stand on the water bath for an hour or so, the tin began to precipitate slowly. Antimony, when present in considerable quantity, comes down with both tin and lead, and also partly remains in the last filtrate.

The author states that in determining zinc as phosphate, lead, tin, and copper, if present, may be separated by oxidising with nitric acid, expelling the latter with as little sulphuric acid as possible, precipitating the copper as thiocyanate, and removing them all in one filtration. Washing with 3 per cent. sulphuric acid to prevent re-solution of lead sulphate has no effect on either the tin or copper precipitates. This method is stated to be unequalled for accuracy and dispatch in determining small amounts of zinc in bronzes.—A. S.

### *Copper, Analysis of Slimes Deposited during the Electrolytic Refining of.* A. Hollard. Bull. Soc. Chim. [3], **19**, 470—472.

The slimes deposited from the dissolving anode in the process of electrolytic copper refining are rich in silver, and often contain gold. Four typical analyses show in percentages :—

| | | | | |
|---|---|---|---|---|
| Silver ................ | 25·8160 | 36·5210 | 38·4800 | 46·5800 |
| Gold ................... | ·0337 | ·0768 | ·1020 | ·1504 |
| Copper ................. | 18·4750 | 24·0420 | 18·5160 | 18·4750 |

In the usual mode of dry assay, with litharge, for silver and gold, no nitre is employed, lest it should oxidise some of the silver and carry it into the slag; but without nitre there is always danger that sulphide and arsenide of gold may be formed, which then do not find their way into the lead button which is subsequently cupelled. The authors find it preferable to make a dry assay for gold only, and to determine silver and copper in the wet way, as follows :—

*Assay for Gold.*—A mixture of dried and powdered slime (12·5 grms.) with litharge (50 grms.), nitre (10 grms.), sodium carbonate (25 grms.), and powdered fused borax (15 grms.), is placed in a capacious crucible, covered with a layer of sodium carbonate, and slowly heated to fusion. A mixture of 20 grms. of litharge and 0·4 grm. of charcoal is then added, and the heat maintained for a few minutes longer. On cooling, the button of lead (15—20 grms.) is cupelled, and the silver in the remaining button parted from the gold in the usual way. The fusion must not be stirred with an iron rod, as that may reduce antimony, which prevents the effective cupellation of the lead.

*Assay for Silver and Copper.*—5 grms. of the dried powdered slime in a porcelain boat are heated gradually to redness in a glass tube through which a current of chlorine is passed, and the operation is stopped when no more volatile chlorides sublime. The residue in the boat consists of chlorides of gold, silver, copper, and lead. It is treated with water acidulated with nitric acid, which dissolves the cupric chloride; this solution, with 5 c.c. of sulphuric acid added, is evaporated almost to dryness, the residue taken up with 20 c.c. of nitric acid, and diluted with water to 300—350 c.c., and the copper determined electrolytically. The silver chloride is dissolved on the filter by washing with 120—140 c.c. of a 20 per cent. solution of potassium cyanide, and the solution made up to 200 c.c. Of this, 50 c.c. are diluted to 300 or 350 c.c., and the silver deposited by electrolysis. Several assays for silver and copper can be conducted at once.—J. T. D.

### *Bismuth, A New Method of Determining.* L. Vanino and F. Treubert. Ber. 1898, **31**, [9], 1303.

On warming and at the same time stirring a mixture of alkaline formaldehyde and a bismuth salt solution, the bismuth is completely precipitated as metal.

A slightly acid solution of the bismuth salt is mixed with formaldehyde and a large excess of caustic soda solution (10 per cent.) is added. The mixture is heated on a water-bath with continual stirring, until the precipitated bismuth has settled and the liquid has become quite clear. More formaldehyde and caustic soda are added and the mixture is heated over the open flame for a few minutes. The precipitate is repeatedly washed with water by decantation and is pressed to a metallic mass with a glass rod. The bismuth is collected on a tared filter or in a Gooch crucible, washed with absolute alcohol and dried at 105° C. If the heating over the open flame be continued too long the liquid is apt to become yellow, but this is without influence on the result of the analysis.

The reaction is applicable for the separation of bismuth from other metals, such as aluminium, zinc, &c.—J. McC.

### *Nitrites in Water, Method of Determining.* L. Robin. J. Pharm. Chim. 1898, **7**, [12], 575–577.

The method is based on the well-known reaction between nitrites and alkaline iodides, in solutions acidulated with acetic acid, the equivalent of iodine liberated being titrated with thiosulphate solution in the usual way. 50 c.c. of water are taken, 2 c.c. of a 20 per cent. solution of chemically pure potassium iodide solution added, and then 2 c.c. of glacial acetic acid; after agitation, the mixture is allowed to repose for exactly half an hour before titration. By this method 0·000005 grm. of nitrous acid in 50 c.c. or 0·0001 grm. per litre, may be determined. Waters containing hydrogen sulphide should be first precipitated with silver sulphate and then distilled, one volume of distillate being collected from every two volumes of water originally taken.—J. O. B.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

### *Organic compounds, Use of Phloroglucol-vanillin Solution for Detecting Sulphur and Nitrogen in.* P. N. Raikow. Chem. Zeit. 1898, **22**, [38], 377–379.

The author has previously shown (Chem. Zeit. **22**, 20) that an alcoholic solution of phloroglucol-vanillin serves as a reagent for detecting the presence of halogens in organic compounds, and he now shows that the same solution will serve to indicate the presence of sulphur, by a red coloration, and of nitrogen in amines, hydrazines, and hydrazo compounds, by a yellow colour.—L. J. de W.

### *Guaiacum as a Re-agent for Oxidising Agents, On the Value of Tincture of.* P. Breteau. J. Pharm. Chim. 1898, **7**, [12], 569—575.

Although tincture of guaiacum has long enjoyed a reputation as a reagent for oxidising agents, for blood, cupric sulphate, hydrocyanic acid, ozone, and other bodies, it is found that the characteristic blue reaction is given by so many other substances, or may be so modified by details of manipulation, as to be apt to lead to fallacious deductions. Even the reaction originally described by Schoenbein, the production of an intense blue colour when equal volumes of the tincture of guaiacum and aerated oil of turpentine are mixed and then shaken with water, is not found to give invariable results. Thus, if the tincture be first diluted with water before adding the turpentine, no reaction is obtained. The guaiacum and turpentine blood reaction of Van Deen, which has been much employed for the detection of minute quantities of blood, is also found to be unreliable, since the colour is not due, as hitherto supposed, to the action of oxyhæmoglobin on the guaiacum, but to the presence of a very stable body, which is also present in milk. The reaction may be obtained perfectly from blood serum deprived of oxyhæmoglobin, and from the whey of milk. In the same manner traces of many other bodies, such as minute quantities of copper sulphate, ether, alcohol, benzene, and animal charcoal, give an immediate blue colour with the guaiacum turpentine test. It is also observed that, although the well-known "guaiacum paper" gives the blue reaction for ozonised air, the colour is not produced if the same tincture of guaiacum be exposed on porcelain, or in narrow capillary tubes to the same air. Thus it is evident that the paper itself plays an important part in the reaction. It is shown that this guaiacum test paper, in its various modifications, develops a blue colour under so many diverse conditions, that it is utterly worthless for special tests such as those for blood, hydrocyanic acid, or phosphorus.

From these observations it is evident that the guaiacum reaction cannot be relied upon as giving results from which definite deductions may be obtained.—J. O. B.

*Petroleum Spirit from Baku, Detection of Di-isopropyl in.* O. Aschan. Berichte, **31**, [11], 1801—1803.

The author has already shown (Chem. Zeit., 1897, 288) the abnormal character of the hydrocarbon fractions, boiling below 70° C., of Russian petroleum. An examination of the fraction 57°—59° C. now reveals the chief constituent as di-isopropyl or tetramethylethane, the sp. gr. at 17·5° being 0·66485, and thus agreeing closely with that of synthetic di-isopropyl (0·668) of b.p. 58° C. The chlorination products of this hydrocarbon, on separation by fractional distillation, gave two main fractions, boiling at 117°—119° C. and 123°—125° C. respectively, the chlorine content of which averaged 29·5 per cent.—the theoretical percentage in $C_6H_{13}Cl$. In sp. gr., the lower fraction — 0·8769 at 22° C.—nearly approaches the value—0·8784 at 19° C.—determined by Pawlow for tertiary di-isopropyl chloride. Only negative results were yielded by this fraction when examined for the nitrolic acid reaction; but the higher fraction gave a brown-red coloration under the same treatment.

The di-isopropyl prepared by the author, reacts with chlorosulphonic acid at the ordinary temperature, becoming completely dissolved with brisk evolution of hydrogen chloride. The reaction is of a more complex nature than the formation of a sulphonic acid, the syrupy product depositing, in ice water, an oil soluble in ether and smelling both of fusel oil and sulphur. It turns green in the air, red in presence of dilute acid, and by its rapid decolorising action on permanganate, appears to be an unsaturated compound. The isopentane (b. p. 30°) from Baku oil, gives similar reactions, and the author remarks that, should other paraffin hydrocarbons be found thus susceptible to attack, the theory of their resisting power will need modification.—C. S.

*Methylpentamethylene in Caucasian Petroleum Spirit. Occurrence of.* O. Aschan. Berichte, **31**, [11], 1803—1805.

In attempting to form nitro-products without the application of pressure from a fraction of Caucasian petroleum spirit—sp. gr. at 20° C. = 0·7145; b.p. 71° C.—with six volumes of fuming nitric acid, the acid liquid was found to deposit acicular crystals, melting at 185° C., and consisting of succinic acid. The filtrate, on dilution, yielded a precipitate soluble in alcohol and ether, having an odour of camphor, and melting at 185°—187° C., but too small in quantity for analysis. A quantity of acetic acid was also detected in this solution.

On heating the same fraction (60 grms.) with ordinary concentrated nitric acid a considerable quantity (4 grms.) of nitrobenzene was produced.

The formation of the above oxidation products is only compatible with the presence of methylpentamethylene in the substance examined.—C. S.

*Dyestuffs, Natural and Artificial; Scheme for the Analysis of.* A. G. Rota. Chem. Zeit. **22**, [44], 1898, 437—442.

The first part of this paper refers to the analysis of separate dyestuffs, the second to that of mixtures. The principal method consists in the study of the behaviour of the dyestuff on reduction, and more particularly its action in very dilute solution (1 : 10,000) towards a solution of stannous chloride and hydrochloric acid. The aqueous or alcoholic solution is diluted to about 1 : 10,000, and 5 c.c. of this are treated with 4—5 drops of concentrated hydrochloric acid and the same quantity of a 10 per cent. stannous chloride solution obtained by dissolving tin in hydrochloric acid. The mixture is shaken and if necessary heated to boiling. If the solution does not decolorise entirely, the dyestuff is to be still further diluted or a further quantity of stannous chloride added. Accepting Armstrong and Nietzki's quinone theory it would appear that under certain conditions, stannous chloride reduces those dyestuffs which can be referred to the mono- and di-imido types, whilst those in which one oxygen atom is replaced by a divalent carbon group are not reduced. Thus considering O : R : O as an *o*- or *p*-quinone, dyestuffs belonging to the nitro, nitroso, azo, and quinone imide groups, *i.e.*, having the constitution O : R : N. (oxyquinone-imide) or . N : R : N. (quinone-di-imide) are reducible, whilst oxyquinone and triphenyl-methane dyestuffs O : R : C : (oxycarboquinone), and . N : R : C : (imidocarboquinone), are not reduced. The reduced solution after neutralisation with potash lye is treated with a few drops of ferric chloride, or is shaken with air. Some solutions remain unchanged, whilst in others the dyestuff is regenerated. Belonging to the former are the nitro, nitroso, and azo dyestuffs which give stable amines on reductions, whereas the latter, the quinone-imido derivatives, forming leuco derivatives, are easily oxidised. The non-reducible dyestuffs can be further divided into two classes, the oxycarboquinone and the imidocarboquinone derivatives. The latter include the magentas, acridines, &c., which on treatment in hot aqueous solution with caustic potash lye are decolorised or precipitated, whilst the former, by virtue of their acid character give coloured salts with alkalis usually easily soluble in water. Dyestuffs can thus be divided into four groups, to which two or three large sub-divisions belong having similar chromophores, the differences depending upon the different salt forming groups amido, imido, carboxyl, or sulphonic acid. The detection of these is effected by ether or by means of suitable fibres, as will be shown later. The dyestuff is examined by the help of the following tables.

Table A.—*Classification of Organic Dyestuffs.*

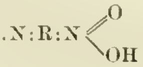
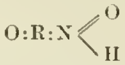
A portion of the aqueous or aqueous alcoholic solution of the dyestuff is treated with hydrochloric acid and then with stannous chloride solution.

| | | | |
|---|---|---|---|
| The solution is decolorised.*<br>Reducible Dyestuffs.<br>The decolorised solution after neutralisation with potash lye or sodium acetate is oxidised with ferric chloride or atmospheric oxygen. | | No further change than with hydrochloric acid alone.<br>Non-reducible dyestuffs.<br>A portion of the original solution is treated with 20 per cent. potash lye, and warmed if necessary. | |
| No change. | Original colour returns. | Solution decolorised or a precipitate is formed. | No precipitate, the colour becomes more intense. |
| Non-oxidisable dyestuffs. | Re-oxidisable dyestuffs. | Imidocarboquinone dyestuffs. | Oxycarboquinone dyestuffs. |
| Group I. | Group II. | Group III. | Group IV. |
| Nitroso, nitro, and azo dyestuffs, including azoxy- and hydrazo colours. | Indogenide and quinone imide dyestuffs. | Amido derivatives of di- and triphenyl methane, auramines, acridines, quinolines, and thiobenzenyl dyestuffs. | Non amidated diphenyl-methane dyestuffs, oxyketone dyestuffs, (including most of the natural organic dyestuffs). |

* Some Indulines are only decolorised with difficulty, the solution never becoming quite colourless.

### Table B.

*Group I.—Dyestuffs Reducible with Stannous Chloride and Hydrochloric Acid, the Colour not Returning on Oxidation.*

| | | | | | | |
|---|---|---|---|---|---|---|
| *Nitro-dyestuffs* $R.NO_2$. Yellow or orange dyestuffs soluble in water, dyeing wool and silk directly, but not cotton. The aqueous solution tends to decolorise with hydrochloric acid. On partial reduction with hydrochloric acid and stannous chloride, they give red nitro-amido derivatives (nitro-amines) or nitrophenols turning red in potash lye .......... | *Nitro-amines* soluble in ether in presence of KOH .................................. | | | |  .N:R:N | *e.g.* Aurantia |
| | *Nitrophenols* insoluble in ether in presence of KOH .............. | *Unsulphonated*, soluble in ether, in presence of acetic acid. | | |  O:R:N | *e.g.* Victoria Yellow. |
| | | *Sulphonated*, in every case insoluble in ether .................................. | | | | Naphthol Yellow S. |
| *Nitroso-dyestuffs* O:R:N.OH. Brown or green, usually insoluble in water. Dye indirectly. Give a blue coloration with sulphuric acid and phenol (Liebermann's reaction) ............................ | *Unsulphonated*, insoluble in alcohol, soluble in ether in presence of acetic acid .................................... | | | | | *e.g.* Dioxine |
| | *Sulphonated*, soluble in water, insoluble in ether .................. | | | | | *e.g.* Naphthol Green B. |
| *Azo dyestuffs* R.N:N.R. Recognisable as not belonging to either of the two foregoing subdivisions. Their aqueous solution treated with potash lye and extracted with ether behaves as follows ............ | The solution is coloured, the colour being extracted by dilute acetic acid. *Basic dyestuffs* .................. | Unsulphonated amido-azo dyestuffs .. ..... | | | .N:R:N.NHR′ | *e.g.* Bismarck Brown. |
| | Coloured solution. The colour is not extracted by dilute acetic acid. *Neutral dyestuffs** ........................ | Oxyazo dyestuffs not containing a carboxyl group .................. | | | O:R:N.NHR′ | *e.g.* Sudan I. |
| | Colourless solution, acetic acid does not extract anything. *Acid dyestuffs* ............ | *Unsulphonated*, soluble in ether from dilute acetic acid ............ | Oxyazo dyestuffs with a carboxyl group ............ | Dye cotton indirectly | | Diamond Yellow. |
| | | | | Dye cotton indirectly | | Chrysamine. |
| | | *Sulphonated*, insoluble in ether from dilute acetic acid ............ | Non-amidated, unchanged by $HNO_2$† | Dye cotton directly | | Bordeaux B. |
| | | | | Dye cotton directly | | Azo Blue. |
| | | | Amidated, react with $HNO_2$ ...... | Dye cotton indirectly | | Fast Yellow N. |
| | | | | Dye cotton directly | | Congo Red. |

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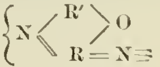
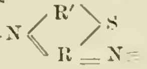
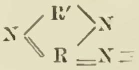
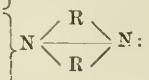
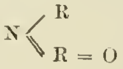
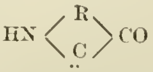
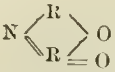
* Some amido-azo dyestuffs (Aniline Yellow) behave like neutral dyestuffs, but differ from these in being decolorised by nitrous acid.

† The presence of an amido group is detected by treating 5 c.c. of the warm solution with 2—3 drops of a 1 per cent. dilute acetic acid and the same quantity of a 1 per cent. potassium nitrite solution. Amido derivatives are decolorised or the colour is modified, whilst non-amidated compounds remain unchanged or are not further affected than by acetic acid alone.

### Table C.

*Group II.—Dyestuffs Reducible with Stannous Chloride and Hydrochloric Acid, the Colour Returning on Oxidation.*

| | | | | | | |
|---|---|---|---|---|---|---|
| The aqueous or alcoholic solution (5 c.c. of a strength 1:10,000), is treated with 4—5 drops of a 20 per cent. potash lye and extracted with 10—15 c.c. of ether. This solution washed with water (once with an equal volume if the original solution was aqueous, but 2—3 times if alcoholic), shows the following reactions: | The solution is *coloured* or *colourless*, but imparts the original colour to 5 per cent. acetic acid when shaken with it.<br>*Basic Dyestuffs.*—Dye wool from an alkaline bath. | The solution is easily reduced by hydrochloric acid and stannous chloride in the cold ............ | *Oxazines* (free from sulphur) ........... | |  | *e.g.*, Meldola's Blue. |
| | | | *Thiazines* (contain sulphur) ........... | |  | *e.g.*, Methylene Blue. |
| | | The solution is only reduced with difficulty and frequently imperfectly. It requires to be warmed, and an excess of hydrochloric acid and stannous chloride ........... | *Indulines.* — Blue colour with concentrated sulphuric acid giving a blue solution on dilution with water* ........ | |  | *e.g.*, Spirit-soluble Induline. |
| | | | *Safranines.* — Green colour with sulphuric acid turning blue and then violet on dilution ......... | |  | *e.g.*, Safranine T. extra. |
| | *Coloured.*—The colour is not extracted by acetic acid.<br>*Neutral Dyestuffs.*—Insoluble in water, soluble in alcohol; dye the fibre from an indigo vat. | Bluedyestuffs affected by warm hydrochloric acid ........ | | *Indophenols* |  | *e.g.*, Indophenol. |
| | | Red or blue dyestuffs unchanged by hydrochloric acid; give isatine with nitric acid ........ | | *Indogenides* |  | *e.g.*, Indigotine. |
| | *Colourless*, acetic acid does not extract anything.<br>*Acid Dyestuffs.*—Soluble in water, dyeing wool from an acid bath. | *Unsulphonated.* — Soluble in ether in presence of acetic acid ............... | | *Oxazones* |  | *e.g.*, Fluorescent Blue, Orceïn. |
| | | *Sulphonated.* — Not soluble in ether .... | Easily reduced by stannous chloride and hydrochloric acid ............... | *Sulphonated Indogenides.* | | *e.g.*, Indigocarmine. |
| | | | | *Sulphonoted Thiazines.* | | *e.g.*, Thiocarmine R. |
| | | | Reduced with difficulty by stannous chloride and hydrochloric acid ........ | *Sulphonated Indulines.* | | *e.g.*, Water-soluble Nigrosines. |

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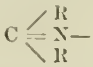
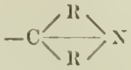
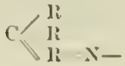
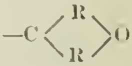
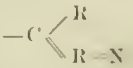
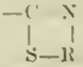
* The Indulines and Safranines differ in their basicity; the former can be liberated by ammonia and extracted by ether, whilst potash is necessary to obtain Safranine base.

### TABLE D.

### *Group III.—Dyestuffs not Reduced by Stannous Chloride and Hydrochloric Acid having the Imidoquinonecarbonchromophore .N : R : C :*

| | | | | | |
|---|---|---|---|---|---|
| The aqueous or alcoholic solution of the dyestuff is treated with potash lye, and then extracted with ether. | Coloured or colourless solution. The colour is extracted by 5 per cent. acetic acid. *Basic Dyestuffs.* Dye wool from an alkaline ($NH_3$) bath.......... | Colourless, non-fluorescent, ethereal solution. The solution in acetic acid is non-fluorescent. The aqueous solution is decolorised by potash, and decomposed by hydrochloric acid........... | *Auramines* .......... |  | *e.g.* Auramine O. |
| | | Colourless, greenish, fluorescent, ethereal solution. The aqueous solution is precipitated by potash, and only slightly changed by hydrochloric acid. Nitric acid colours it red..... | *Acridines* ........... |  | *e.g.*, Phosphine. |
| | | Colourless or coloured non fluorescent ethereal solution. The acetic acid extract is reddish-violet, blue, and green, without fluorescence. The aqueous solution is generally decolorised by potash on warming, and is turned yellow by hydrochloric acid (with the exception of the phenylated rosanilines)...... | *Rosanilines* (unsulphonated). |  | *e.g.*, Magenta. |
| | | The ethereal solution is colourless, and does not fluoresce, The solution in acetic acid is pink and fluorescent. The aqueous solution is decolorised by potash,.................. | *Pyronines* (are turned yellow by hydrochloric acid).<br>*Rhodamines* (unsulphonated, are unaffected by hydrochloric acid). |  | *e.g.* Pyronine G.<br>Rhodamine S. |
| | The colour is not extracted by acetic acid from the coloured ethereal solution. *Neutral Dyestuffs.* Insoluble in water: soluble in alcohol..... | The ethereal solution is yellow, and does not fluoresce. The alcoholic solution is yellow and non-fluorescent, and is unaltered by aqueous acids or alkalis.................... | *Quinophthalones* * (unsulphonated). |  | *e.g.*, Spirit soluble Quinoline Yellow. |
| | The ethereal solution is colourless; acetic acid does not extract anything. *Acid Dyestuffs.* All soluble in water, and dye wool from an acid (HCl) bath................ | The aqueous dyestuff solution is boiled with bleached cotton.<br>The cotton is not dyed.<br>Yellow dyestuffs soluble in water, without fluorescence, unchanged by aqueous acids or alkalis.............................. | *Sulphonated Quinophthalones.* | | *e.g.*, Water soluble Quinoline Yellow. |
| | | Water soluble dyestuffs, reddish violet, blue, or green, generally decolorised by potash lye, little affected by hydrochloric acid.... | *Sulphonated Rosanilines.* | | *e.g.*, Magenta S. |
| | | Red or violet dyestuffs giving fluorescent aqueous solutions not affected by potash, or only slightly........................ | *Sulphonated Rhodamines.* | | *e.g.*, Violamine R. |
| | | The cotton is dyed.<br>Yellow or orange dyestuffs giving more or less fluorescent aqueous solutions, dyeing silk, wool, and cotton directly............ | *Thiazols.* † |  | *e.g.*, Primuline. |

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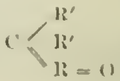
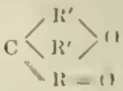
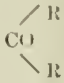
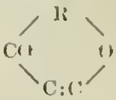
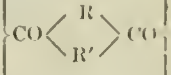
* The chromophores of the quinoline dyestuffs Berberine and Flavaniline, are not as yet understood, they are probably compounds intermediate between the auramines and acridines.

† The thiazol dyestuffs are generally sulphonated, with the exception of Thioflavine T, which, although soluble in ether, dyes wool from an alkaline bath.

### TABLE E.

### *Group IV.—Dyestuffs not reduced by Stannous Chloride and Hydrochloric Acid having the Oxyquinonecarbonchromophore O : R : C :*

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| The alcoholic solution of the dyestuff is treated with a few drops of a dilute (1 : 1000) solution of ferric chloride | Remains unchanged. *Nonamidated triphenylmethane dyestuffs,* generally soluble in water, and usually dyeing wool directly. | The dyestuff is dissolved or suspended in boiling water. | It does not dye wool directly. Generally insoluble in water, soluble in alcohol without fluorescence | | | *Aurines* |  | *e.g.*, Aurin. |
| | | | Dyes wool directly. Generally soluble in water and alcohol to fluorescent solutions ............ | | | *Phthaleïnes* |  | *e.g.*, Eosin. |
| | Turns green or olive green. *Oxyketone dyestuffs,* generally insoluble in water, dye fibres indirectly. | The original dyestuff is treated with 1 per cent. potash lye. | Dissolves with a yellow or reddish-yellow colour. *Monoketones.* | The alkaline solution is treated with an excess of hydrochloric acid. | Usually decolorised (with decomposition) especially on warming. | *Benzophenones.* |  | *e.g.*, Alizarin Yellow A. |
| | | | | | Gives an intense yellow without decomposition. | *Flavones* |  | *e.g.*, Quercetin. |
| | | | Dissolves with a red, reddish-violet, blue, or green colour. *Diketones* (Quinones). | The alkaline solution is acidified with acetic acid. | The free acid is precipitated. Generally soluble in ether, and dyeing the fibre indirectly. | *Anthraquinones* (unsulphonated). |  | Alizarin. |
| | | | | | The dyestuff acid remains in solution, is insoluble in ether and dyes wool directly. | *Anthraquinones* (sulphonated). | | Alizarin Red S. |

With the help of the foregoing tables, the dyestuff in question is assigned to the particular group or sub-division to which it belongs, and is then further identified by one of the published tables of dyestuffs. It is, however, to be borne in mind that these tables only contain some of the many dyestuffs now on the market. It may thus be necessary to examine the solution spectroscopically or to decompose the dyestuff into simpler or more easily recognisable components. For example, the various phthaleïns may be recognised by boiling the solution with potash and zinc dust, and examining the filtrate after neutralisation with acetic acid, for bromine or iodine. An alternative method is to heat the dyestuff with lime to redness, dissolve in nitric acid, and examine the solution for the halogen. The detection of sulphur to distinguish for example thiazines from oxazines is effected by fusing the dyestuff with potassium nitrate, dissolving, and examining the filtrate for sulphates. If the dyestuff be reducible by stannous chloride, the solution is freed from tin by sulphuretted hydrogen, and the filtrate is examined for the reduction product. Thus picric acid yields triamidophenol, which on treatment with ferric chloride gives blue amido-di-imidophenol. Azo dyestuffs on reduction give at least two primary amines which frequently can be separated by ether. The reduced solution after removing the tin, is treated with an excess of potash and extracted with ether. The unsulphonated amines are removed whilst the sulphonated ones remain in the aqueous solution. The latter can be recognised by giving characteristic azo compounds with certain diazo derivatives. For example sulphanilic acid, obtained by reducing Naphthol Orange, gives, when combined with tetrazodiphenyl chloride, a yellow tetrazo dyestuff, naphthionic acid gives Congo Red and certain hydroxysulphonated amines such as 2:1′:3′, $C_{10}H_5.NH_2.OH.SO_3H$ give a violet colour (Diamine Black R, &c.). The presence of a *p*-diamine is recognised by the thiazine reaction (ferric chloride and hydrochloric acid in presence of sulphuretted hydrogen), and in non-amidated dyestuffs this reaction affords a means of distinguishing between mono- and disazo dyestuffs. It is also possible to decide in this case whether a sulphonic acid group is in the side or middle ring, since in the former case a thiazine is formed which in presence of potash can be extracted by ether, whereas a sulphonated thiazine remains in the aqueous solution.

The analysis of mixtures is a more complicated problem, and the only methods already known are microscopical examination, projection on to sulphuric acid or moistened paper, and absorption by filter-paper, &c. The two chief reagents suggested by the author are ether and wool. With regard to separations by the former, 100 c.c. of the dyestuff solution are treated with 1 c.c. of 20 per cent. potash lye and then extracted with three times the volume of ether, which is repeated until the ether is colourless, even after acidifying with acetic acid. The aqueous alkaline solution containing the acid dyestuffs is neutralised with acetic acid and subsequently examined. The ethereal solution is washed with very slightly alkaline water, and extracted with one-third of its volume of 5 per cent. acetic acid, which is then evaporated on the water bath; the residue contains the basic dyestuffs. Some dyestuffs, even after extraction with acetic acid, remain in the ether, which in this case is itself evaporated. In presence of caustic potash it frequently happens that some (very few) acid or rather neutral dyestuffs pass into the ether, *e.g.*, spirit soluble Quinoline Yellow, Indophenol Blue, the different Sudans, &c. In extracting with ether, the choice of an alkali is not immaterial. The safranines, for example, require potash, whilst the magentas are basified by ammonia; the indulines, oxazines, and acridines are basified by very dilute ammonia, whilst dyestuffs like Chrysoidine, Bismarck Brown, Rhodamine S., Victoria Blue, &c., are dissociated in their dilute aqueous solutions. Hence the dilute aqueous solution is first extracted with ether, then treated with 1 per cent. ammonia and extracted, subsequently with concentrated ammonia, and finally with 20 per cent potash lye. A further separation depends upon a difference of solubility in water and ether. When the ethereal solution is shaken with an equal volume of water, some dyestuffs are extracted, whilst others less soluble, or quite insoluble, remain in the ether. Acridine Yellow can in this way be separated from Phosphine. By means of successive extraction with ether, the acid dyestuffs contained in the original aqueous solution fall into three groups: 1. Those extracted by ether in presence of 1 per cent. acetic acid. 2. Those extracted in presence of sulphuric or hydrochloric acid. 3. Those insoluble in ether. In this way it is possible to separate Erythrosine from Roccelline and from Bordeaux B. and Direct Yellow from Congo Brown and Congo Red. If the mixture consists exclusively of acid dyestuffs, a fourth group has to be taken into consideration, *viz.*, dyestuffs having so feebly an acid character that they can be extracted from the neutral solution by ether, like Sudan I, Orchil, and Sudan G. In this manner Sudan G can be separated from Victoria Yellow, and Orchil from Eosin. Moreover, by extracting the ethereal solution with water, a further separation can be effected; for example, picric acid from Martius Yellow (dinitronaphthol is only very slightly soluble in water) and Diamond Black from Naphthol Orange. The fractional addition of ammonia may also be resorted to. When the separation cannot be effected by ether recourse is had to wool. All basic dyestuffs are taken up by woollen fibres from a faintly alkaline or neutral bath, whilst acid dyestuffs are left in the bath. The solution of the dyestuff (1:1000) is made alkaline with ammonia (4—5 drops per 100 c.c. of solution), and a skein of wool is immersed in it and boiled for 3—5 minutes. A second skein is then immersed, and, if necessary, a third, so long as the wool is dyed. The solution contains the dyestuff acids. The wool is boiled first in very weak ammonia, subsequently in pure water, and is then extracted with hot 5 per cent. acetic acid. This solution is evaporated on the water-bath, when the basic dyestuffs remain as a residue. Either the dyeing or extraction can be carried out fractionally, since some dyestuffs attach themselves or are removed more easily than others. Wool acts more effectively in the separation of acid dyestuffs. A 0·1 per cent. solution of the dyestuff is acidified with hydrochloric acid (4—5 drops of concentrated hydrochloric acid per 100 c.c.), and successive skeins of wool are immersed for 3—5 minutes in the boiling solution, as above. The solution contains the indirect mordant dyestuffs. The skeins are washed first with dilute hydrochloric acid, subsequently with water, and are finally extracted at the boil with 5 per cent. ammonia, which then contains the direct dyestuffs. A complete separation is only effected by repeating this treatment, since some of the indirect dyestuffs are absorbed by the fibre. It is possible thus to separate—

| | | | |
|---|---|---|---|
| Direct .... | Bordeaux B. | Biebrich Scarlet. | Acid Yellow. |
| Indirect .. | Euocyanine. | Cochenille. | Saffron. |

By dyeing in a neutral or more or less strongly acid bath further separations can be carried out. For example, the following dyestuffs can be separated:—

| | | | |
|---|---|---|---|
| Dye from neutral bath. | Alkali Violet. | Acid Violet 4 B N. | Orchill. |
| Dye from acid bath | Ponceau 6 R B. | New Coccine. | Bordeaux B. |

With a strongly acid solution (1 c.c. hydrochloric acid per 200 c.c. of solution), the following separations may be made:—

| | | |
|---|---|---|
| Dye from strongly acid bath ..... | Bordeaux S. | Bordeaux B. |
| Dye from weak-acid bath ........ | Orange G. | Methyl Orange. |

In all these cases the wool is washed with pure water, faintly acid, or strongly acid water, according to the nature of the dye-bath, the fibre subsequently being extracted with hot dilute ammonia, which may also be carried out fractionally. In certain cases, when neither ether nor wool give decisive results, it is necessary to employ cotton. Amongst direct basic colours for cotton are Pyronine, Rhodamine (partially), and Thioflavine, whilst acid colours are Curcumine, Bixine, Carthamine, the tetrazo dyestuffs, &c. For the separation of direct from indirect cotton dyestuffs, the cotton is boiled for 10 minutes in the neutral or faintly alkaline (soap) bath, and then repeatedly washed with boiling water. The following dyestuffs can be separated in this manner:—

| | | |
|---|---|---|
| Dye cotton directly... | Carbazol Yellow. | Cotton Yellow R. |
| Dye cotton indirectly. | Diamond Yellow R. | Phloxine B. |

By varying the conditions of acidity or the strength of the bath it is also possible to effect separations: *e.g.*, Brilliant Congo is easily dyed from an acid bath with hydrochloric acid, whilst Brilliant Yellow only does so with difficulty. If neither ether, wool, nor cotton lead to separations, other solvents must be made use of, such as petroleum ether, amyl alcohol, chloroform, and the method employed as given in the case of ether. Thus, by means of petroleum ether it is possible to separate Eosin from Martius Yellow. See "*The Qualitative Analysis of Coal Tar Colouring Matters*," by A. G. Green, this Journal, 1893, 3.

—T. A. L.

### *Citric Acid in Vegetable Juices, Wine, and Milk, Detection of.* G. Denigès. Rev. Chim. Analyt. appl. **6**, [7], 110—112; Analyst, 1898, **23**, [267], 161.

Under the influence of permanganates and in an acid medium, citric acid forms with mercury a compound insoluble in presence of mercuric sulphate. A method of detecting citric acid, based on this reaction, is applied by the author as follows:—

*In Vegetable Juices.*—A few drops of the juice are diluted with 4—5 c.c. of water, and heated to boiling with 1 c.c. of mercuric sulphate solution (mercuric oxide, 5 grms.; conc. sulphuric acid, 20 c.c.; water, 100 c.c.), 5 to 6 drops of a 2 per cent. solution of potassium permanganate are then added, or 1 drop if the solution be very dilute, when if citric acid be present, the liquid is decolorised, and a white precipitate formed. 0·5 mgrm. of citric acid may be detected in this way.

*Small Quantities of Citric Acid in presence of a large amount of Tartaric Acid.*—5 c.c. of a 2 per cent. solution of the average sample are heated with 1 c.c. of 2 per cent. permanganate, until the mixture acquires a brown tinge, and a few bubbles of gas are evolved. On decolorisation, 1 c.c. of the mercuric sulphate solution is added, and the liquid again heated to boiling. A distinct white turbidity is obtained in presence of less than 0·5 per cent. of citric acid.

*In Wine.*—10 c.c. of the wine are well shaken with 1—1·5 grm. of lead peroxide, and 2 c.c. of the mercuric sulphate solution. The liquid is filtered, and 5—6 c.c. of the filtrate heated to boiling and treated with 1 drop of permanganate, 9 additional drops being added in succession as decolorisation ensues. With normal wines a very slight haze is formed, due to the traces (5—6 cgrms. per litre) of citric acid, which are stated to be invariably present. In presence of 0·1 grm. per litre, of citric acid, a distinct turbidity is produced, and above 0·4 grm. per litre, a flocculent precipitate.

*In Milk.*—10 c.c. of milk, 2 c.c. of a 5 per cent. solution of sodium metaphosphate, and 3 c.c. of the mercuric sulphate solution are well mixed and filtered, the first runnings being rejected. 5—6 c.c. of the filtrate are heated to boiling and shaken up with successive drops of permanganate In presence of citric acid, a distinct white turbidity ensues after the addition of 4 or 5 drops in the case of cows milk, whilst with 8—10 drops, a flocculent white precipitate is produced, accompanied by a yellow coloration, which may be removed by hydrogen peroxide. Acetic, tartaric, &c. acids, glycerin, gum, and other substances likely to occur with citric acid do not interfere with the reaction, but chlorides, bromides, or iodides, must be first removed by silver sulphate, and if oxalic acid or other substance capable of reacting on mercuric sulphate be present, the mixture must be first oxidised in an acetic acid medium by a slight excess of permanganate, which is then reduced by hydrogen peroxide, before the test is applied.

—A. S.

### *Saccharine in Beer and other Liquids, Detection of.* A. Herzfeld and F. Wolff. Zeits. d. Vereins. d. deutsch. Zuckerind. 1898, 558, through Woch. f. Brau., **15**, [25], 335—336.

The authors have examined certain methods or tests for the detection of saccharine, more especially with the view of ascertaining the presence (or absence) of this substance in beer. The most simple test is the intense sweetness of the residue obtained by extracting the beer with ether and evaporating the latter. It was found, however, that by extracting beers, known to be free from saccharine, some lævulose passed into the ether solution and yielded a sweet residue. Börnstein's reaction, which is based upon the fact that a green fluorescence is obtained by heating the ether residue with one-and-a-half times its weight of resorcinol, plus a few drops of concentrated sulphuric acid, and then making the solution alkaline with soda, is useless, because all beers contain resins, which also give the green fluorescence. The methods of Wauters, and of Herzfeld and Reischauer, are also unreliable from a similar cause; the former depends upon the formation of salicylic acid by fusing saccharine with an alkali, and in the latter the sulphur in the saccharine is oxidised into sulphuric acid, and tested for in the same way. The writers, however, find that the following method is satisfactory. Half a litre of the beer is acidulated with a few drops of phosphoric acid solution, and it is then well shaken for fifteen minutes with twice its volume of ether. The ether is separated, evaporated to a small bulk (a few c.c.), and is then brought on to a plug of asbestos. This is dried at 100° C., and placed in a suitable sublimation apparatus. The saccharine sublimes and the above tests may be applied to the sublimed product.

Out of 180 beers analysed, no less than 60 contained saccharine. Quantitative estimations were found to be impossible.

It is interesting to note that, after the 1st of October, the use of saccharine in food materials will be forbidden in Germany.—W. P. S.

### *White Wines, prepared by the Decolorisation of Red Wines by Animal Charcoal, Reaction for Identifying.* A. Bimm. Jour. Pharm. et Chim. 1898, **8**, [1], 9—11.

The author has observed the presence in animal charcoal of a soluble, oxidising substance, which is extracted by the wine during its treatment for decolorisation. This substance gives most of the reactions for nitrates, and it is proposed to utilise the blue coloration, given with diphenylamine, for the identification of wines so treated.

The reagent is prepared by dissolving 0·10 grm. of diphenylamine in 100 c.c. of sulphuric acid 1:4, and making up the solution to 500 c.c., with acid of sp. gr. 1·842. Two c.c. of the reagent are placed in a porcelain capsule, of such a size that the bottom is covered to a depth of 4 or 5 mm. Six drops of the wine are then allowed to flow down the sides of the capsule, and the formation of a blue ring at the circumference of the liquid indicates the presence of matter extracted from the char.

All the samples of suspected wines, examined by the author, showed the blue coloration with diphenylamine, whilst genuine white wines gave no reaction.—J. F. B.

### *Gelatin in Chocolate, Detection of.* P. Onfroy. J. Pharm. Chim. 1898, **8**, [1], 7—9.

When chocolate is adulterated with 5 per cent. of gelatin, it is possible for the manufacturer to add 10 per cent. of water, without in any way altering the appearance of the article. Consequently, if the chocolate shows on analysis a very high percentage of water, adulteration with gelatin may be suspected. If a quantity of 25 grms. of the chocolate, mixed with 100 c.c. of hot water, gives a solid mass on cooling, gelatin is probably present in considerable quantity. This can easily be identified by treating 5 grms. of chocolate with 50 c.c. of water, and adding 5 c.c. of a 10 per cent. solution of lead acetate. If gelatin be present, the filtrate will give a yellow precipitate on treatment with a few drops of a saturated aqueous solution of picric acid. Occasionally, however, the amount of gelatin is so small that the whole of it remains behind in combination with the tannin of the cocoa. In this case, the fatty matter must be extracted from 10 grms. of chocolate by means of ether, and the residue treated with 100 c.c. of water; 5 to 10 c.c. of a 10 per cent. solution of caustic potash and 10 c.c. of lead acetate solution are then added. The compound of gelatin and tannin is soluble in potash, and is not completely precipitated by the lead acetate. The gelatin may therefore be detected in the neutralised filtrate by means of picric acid.

The reaction of gelatin with picric acid is said to be extremely delicate; the presence of theobromine does not affect the result, as this alkaloid gives no precipitate with picric acid. Since pure cocoa contains about 50 per cent. of fat, a rough idea of the quantity of gelatin which has been added to the chocolate can be obtained by deducting the sum of the sugar, water, and double the fat, found by analysis, from 100.—J. F. B.

*Naphtha (Petroleum) Acids, Some Properties of the Salts of the; and Qualitative. Distinction of the latter from Salts of the Fatty Acids.* K. Charitschkow. J. russ. phys.-chem. Ges. **29**, 691; Chem. Centr. 1898, **1**, [23], 1191.

*See under* III, *page* 751.

## ORGANIC CHEMISTRY—QUANTITATIVE.

*Paraffin, Commercial; Determining the Solidifying point of.* R. Kissling. Chem. Rev. Fett- u. Harz-Ind. **5**, [5], 87—88.

Replying to Singer's criticism (this Journal, 1898, 609) the author points out that the only important novelty claimed by him is the agitation of the paraffin by stirring with the thermometer, a modification which increases the immunity from external influences and consequently enhances the reliability of the method.

In connection with the influence on results of the depth of substance in the testing glass, his experiments show that this is negligeable, the solidifying point of one and the same sample of paraffin differing by only 0·1° C. (53·1°—53·2 C.) when the quantity was varied between 25 and 80 grms. and the depth in the glass between 20 and 60 mm. —C. S.

*Paraffin, Quantitative Estimation of, in Distillates from Crude Petroleum and Brown Coal-Tar.* D. Holde and A. Allen. Mitth. Königl. tech. Versuchsanst. **16**, [2], 97—113.

The experiments detailed in the paper were instituted in order to ascertain whether the method previously adopted (this Journal, 1897, 362) is generally applicable to products of the brown coal tar industry as well as those from petroleum; and also to test the method proposed by Aisinmann (Chem. Rev. Fett- u. Harz-Ind. **4**, 106), and the experience of Eisenlohr in connection with the volatility of paraffin from brown coal-tar (this Journal, 1897, 701).

It was found advisable, in the case of oils containing a moderately high proportion of paraffin, to ascertain the approximate quantity of alcohol-ether mixture required for the solution of the oil and precipitation of the paraffin at −20° to −21° C., by a preliminary experiment, and then dissolve the oil at room temperature, this procedure resulting in a more complete separation of the paraffin when immersed in the refrigerating mixture.

Also, in the case of semi-fluid to solid paraffin masses (mixtures) it was found preferable to reduce the quantity tested from 3—10 grms. to 0·5—2 grms., the samples being dissolved at room temperature in ether (avoiding an excess), an equal volume of absolute alcohol being then added before cooling. If too consistent to filter, the substance was diluted with a little alcohol-ether (1 : 1).

The precipitated paraffin was, in every instance, redissolved in petroleum spirit (b.p. 50° C.) and carefully evaporated on the water-bath, traces of water being removed by the aid of alcohol and evaporation.

The investigation of the relative solubility of hard paraffins in alcohol and alcohol-ether showed that the former has a greater solvent power than was assumed by Aisinmann, 100 parts dissolving 0·1—0·19 part at 20° C., and 0·015—0·017 part at −15° C. Alcohol-ether is a more powerful solvent, 100 parts taking up 0·03—0·06 part at −19° to −20° C. In either case the solubility of the paraffin increases as the melting point falls.

The conclusion arrived at by the authors respecting the applicability of the alcohol-ether method for the examination of petroleum distillates is affirmative, so far as the paraffins melting above 48° C. (candle paraffin) are concerned. For the softer kinds the quantitative results obtainable by the method are relative merely and not absolute.

No appreciable volatility at 105° C. was detected in any of the samples of paraffin examined, with the exception of some specimens of brown-coal tar paraffin supplied by Eisenlohr, which suffered a loss of from 3·1 to 7·43 per cent. This loss can, however, be reduced by ceasing to apply heat, when the odour of the solvent is no longer manifest, and by completing the drying in the desiccator.

When applied to several samples of crude paraffin and distillates from brown-coal tar, supplied by Krey, the alcohol-ether method gave results from 1 to 3 per cent. higher than those yielded by the same products in actual work in the refinery, a concordance regarded as sufficient to render the method suitable for technical use.

A re-examination of the Aisinmann alcohol method confirmed the authors' opinion of its inferiority to that in which the alcohol-ether mixture is employed.—C. S.

*Aniline, Quantitative Estimation of; Note on.* M. N. Schapoehnikoff. Rev. Gen. des Mat. Col. 1898, **2**, [18], 213—214.

Reinhardt (Chem. Zeit. **17**, 1893, and Zeits. Anal. Chem. **33**, 1894) has described a method for estimating aniline quantitatively, by brominating with a solution of potassium bromate, the amines being dissolved in excess of hydrobromic acid solution.

The author has improved this method by employing commercial potassium bromate, which can be obtained very pure, instead of making it in each experiment as in Reinhardt's method. A solution of 8 grms. of $KBrO_3$ per litre is used, and to dissolve the amine 1 grm. of base is taken for 60 grms. of hydrobromic acid of 25 per cent. HBr. This solution is diluted so that 1 litre contains 10 grms. of base.

Control experiments are made with pure aniline distilling at 182° C. and with pure *p*-toluidine of m. pt. 45° C. The bromate solution is run into the amine solution until, after allowing the precipitate to settle, the colour is just yellow. If much toluidine is present, the end point is rather difficult to see, owing to the precipitate and the solution being slightly red. The table on next page shows some of the results obtained by this method, using mixtures of aniline and toluidine.

The results are calculated from the formula—

$$\frac{x}{t} + \frac{p-x}{t^1} = n.$$

Where $p$ = Total weight of bases employed.

$x$ = Weight of aniline.

$t$ = No. of c.c. of $KBrO_3$ Solution when pure aniline is used.

$t^1$ = No. of c.c. of $KBrO_3$ solution when pure toluidine is used.

$n$ = No. of c.c. of $KBrO_3$ solution used in the experiment.

$t$ and $t^1$ are determined in a separate experiment. The potassium bromate solution does not change during a month.

G

| Mixture Prepared. | | | | Total Weight of Amines. | c.c. of $KBrO_3$ Solution. | Per Cent. of Aniline. | Difference. |
|---|---|---|---|---|---|---|---|
| Aniline. | Toluidine. | Aniline. | Toluidine. | | | | |
| c.c. | c.c. | Per Cent. | Per Cent. | | | | |
| 50·0 | — | 100 | — | 0·5 | 97·0 | — | — |
| 49·5 | 0·5 | 99 | 1 | 0·5 | 96·7 | 99·3 | + 0·3 |
| 49·0 | 1·0 | 98 | 2 | 0·5 | 96·1 | 98·1 | + 0·1 |
| 48·5 | 1·5 | 97 | 3 | 0·5 | 95·6 | 97·0 | 0·0 |
| 48·0 | 2·0 | 96 | 4 | 0·5 | 95·0 | 95·8 | − 0·2 |
| 19·0 | 1 0 | 95 | 5 | 0·2 | 37·8 | 94·7 | − 0·3 |
| 18·8 | 1·2 | 94 | 6 | 0·2 | 37·6 | 93·5 | − 0·5 |
| 18·6 | 1·4 | 93 | 7 | 0·2 | 37·4 | 92·6 | − 0·4 |
| 18·4 | 1·6 | 92 | 8 | 0·2 | 37·3 | 91·7 | − 0·3 |
| 18·2 | 1·8 | 91 | 9 | 0·2 | 37·1 | 90·8 | − 0·2 |
| 18·0 | 2·0 | 90 | 10 | 0 2 | 36·9 | 89·8 | − 0·2 |
| 16·0 | 4·0 | 80 | 20 | 0·2 | 35·0 | 80·1 | + 0·1 |
| 14·0 | 6·0 | 70 | 30 | 0·2 | 33·1 | 70·1 | + 0·1 |
| 12·0 | 8·0 | 60 | 40 | 0·2 | 31·1 | 59·8 | − 0·2 |
| 10·0 | 10·0 | 50 | 50 | 0·2 | 29·2 | 49·9 | − 0·1 |
| 8·0 | 12·0 | 40 | 60 | 0·2 | 27·4 | 40·2 | + 0·2 |
| 6·0 | 14·0 | 30 | 70 | 0·2 | 25·4 | 30·0 | 0·0 |
| 4·0 | 16·0 | 20 | 80 | 0·2 | 23·5 | 20·1 | + 0·1 |
| 2·0 | 18·0 | 10 | 90 | 0·2 | 21·5 | 9·7 | − 0·3 |
| — | 50·0 | — | 100 | 0·5 | 49·1 | — | — |

—C. M.

*Formic Acid, Determination of, in presence of easily Oxidisable Organic Substances.* A. Leys. Bull. Soc. Chim. [3], **19**, 472—478.

The method depends on the precipitation of mercurous acetate from a hot solution of mercuric acetate, the formation of one molecule of mercurous acetate $Hg_2(C_2H_3O)_2$ corresponding with the destruction of one molecule of formic acid H.COOH. The reaction occurs with very weak solutions of formic acid, preferably not over 1 per cent.; the authors have assured themselves of the accuracy of the method in the cases of mixtures of formic acid with acetic acid, ethyl or methyl alcohol, formaldehyde or acetaldehyde. Details are as follows:

*Formic and Acetic Acids.*—The total acidity of the solution is first determined in terms of acetic acid. If the solution contain much acetic acid and but little formic, it should be diluted so as to correspond with a total acidity of 20 to 30 per cent; but if the formic acid be as high as one-twentieth the amount of the acetic acid, then the total acidity of the diluted solution should not be more than 2 per cent. Of this diluted solution 10 c.c. are measured into a beaker of about 300 c.c. capacity, furnished with a 100 c.c. mark; 20 or 30 c.c. of a 20 per cent. solution of mercuric acetate are added, and distilled water added up to the 100 c.c. mark. The whole is then heated just to ebullition (which should take seven or eight minutes) and then allowed to cool and to stand for 24 hours. The precipitate is collected in a funnel with a plug of glass wool in the neck (using the filtrate to rinse the beaker out completely), and washed, four or five times with 95 per cent. alcohol containing 2 per cent. of glacial acetic acid, three times with alcohol alone, and three or four times with anhydrous ether. It is then dried in a vacuum, rapidly dissolved by plunging the funnel into nitric acid diluted with an equal volume of water, and the mercury in an aliquot portion of this solution is determined by precipitating with sodium chloride, collecting the calomel on a filter, and drying as rapidly as possible at 100° C.

*Formic Acid and Alcohol.*—The acidity is determined and brought by dilution to 1 or 2 per cent. The process is then conducted as above, save that 2 c.c. of glacial acetic acid are added before the mercuric acetate.—J. T. D.

*Earthnut (Arachis) Oil, New Method of Determining in other Oils.* F. Jean. Rev. de Chim. Ind. 1898, **9**, [102], 162—163.

In place of Renard's method, which the author finds is incapable of detecting less than about 30 per cent. of earthnut oil in a mixture, the following process based on the qualitative tests of Girard and Blarez is proposed. 10 grms. of the oil are saponified by being heated in an enamelled iron dish at 110° C., with a mixture consisting of 3 grms. of potash dissolved in 3 or 4 c.c. of water and 5 c.c. of alcohol (36°). The mass is well stirred with a spatula and the heating continued until the soap becomes dry. When cold, the soap is transferred to a flask and mixed with 100 c.c. of alcohol (36°) previously saturated with potassium arachidate at 11° to 12° C. The flask is warmed under a reflux condenser until the soap just dissolves, and is then left for 12 hours at a temperature of 15° C. The precipitate is filtered off and recrystallised in the same way from the saturated alcohol. It is then collected, transferred to a flask, and boiled with 50 c.c. of water containing some hydrochloric acid, in order to liberate the arachidic acid. The latter is subsequently extracted with petroleum spirit in a separating funnel, and after evaporation of the solvent, dried at 100° C. and weighed. Its melting point should not be lower than 72° C. Since edible earthnut oil contains in the mean 4·55 per cent. of arachidic acid the proportion of that oil in the mixture under examination can be readily calculated from the result. (See Analyst, **21**, 330.)

—C. A. M.

*Fatty Acids, Separation of Saturated from Unsaturated.* K. Farnsteiner, Zeits. Untersuch. Nahr. u. Genussmittel, 1898, **1**, [6], 390—399.

In place of the various modifications of the method depending on the solubility of the lead salts of the unsaturated fatty acids in ether, the author has devised a means of separation in which benzene takes the place of ether, preliminary experiments having shown that the lead salts of the saturated fatty acids are invariably less soluble in the former than in the latter. The following method of making the determination was adopted:—From 0·6 to 1 grm. of the fat was saponified in an Erlenmeyer flask with alcoholic potash, the solution neutralised with acetic acid, and after evaporation of the alcohol, the soap was dissolved in about 100 c.c. of boiling water and precipitated with 30 c.c. of a boiling solution of lead acetate (containing about 1 grm.). When cold the liquid is filtered and the residue in the flask and on the filter washed with cold water, freed from the latter as completely as possible and dissolved in 50 c.c. of hot benzene. The solution is left at the ordinary temperature for about 15 minutes, and is then cooled for about 2 hours at 8° to 12° C. In order to separate the liquid from the crystalline deposit, the flask is closed with a cork having two holes, through one of which passes a short straight tube, whilst the other holds a tube reaching to the bottom of the flask, and having its exterior end bent downwards outside the flask. The interior opening of this tube is covered with a plug of cotton-wool which serves as a filter, and the liquid is driven upwards through this by forcing air into the flask through the short straight tube. When the liquid has been removed as completely as possible in this way, the flask is washed with 10 c.c. of benzene at about 10° C., which is also expelled. The precipitate is then dissolved in 25 c.c. of hot benzene, again cooled for an hour at 8° to 12° C., and the liquid again filtered. In the same way a third precipitation and filtration are carried out, so that altogether from 120 to 130 c.c. of the benzene filtrate are obtained.

The liquid fatty acids are recovered from the united filtrates by shaking the latter with 10 per cent. hydrochloric acid, washing twice with water, filtering the solution of the fatty acids through cotton-wool into a flask and distilling off the benzene in a current of hydrogen. The solid fatty acids are determined by heating the lead salts in the flask with 25 to 30 c.c. of benzene for a short time, then adding 10 per cent. hydrochloric acid, continuing the heating for about 15 minutes under a reflux condenser, washing the solution, and evaporating the benzene.

When free fatty acids are to be examined, the best method is to dissolve them in benzene and to heat the solution under a reflux condenser with lead hydroxide obtained by precipitating a solution of lead acetate with sodium hydroxide, washing the precipitate with water, alcohol, and ether, drying it at a gentle heat and finely powdering it. The proportions required for one part by weight of solid and of liquid fatty acids are 0·4 and 0·2 parts respectively.

The author gives in a table the results of the analysis of experimental mixtures of liquid and solid fatty acids from various sources, from which it appears that the amount of the former, as determined by this method, is from 1 to 3 per cent. too low, whilst that of the solid acid is, in the maximum, 1·65 per cent. too high. The results obtained with ordinary fats and oils are reserved for a future communication.—C. A. M.

*Fats, Contributions to the Technical Analysis of.* A. Schukow and P. Schestakow. Zap. imp. rus. techn. obschtsch, 1898, **32**, [3], 100. (Through Chem. Zeit. Rep. 1898 [17], 145.)

The authors consider that the most accurate method of determining the water is to dry the fat to constant weight at 100° C. Solid foreign substitutes (organic or inorganic) can only be determined accurately by an extraction process when they form no definite compound with the fat. Thus, in the case of bone fats the method is inapplicable since calcium salts of the fatty acids are always present, and these behave very differently with different solvents and under the varying conditions of temperature, time of extraction, amount of water and fatty acids present, &c. For example, low-boiling petroleum spirit can dissolve less of the calcium salts than can acetic ether; and in one experiment the same fat extracted with boiling and with cold carbon bisulphide left 1·1 and 6·5 per cent. of insoluble residue respectively. If the fat before treatment with the solvent be first dried for the determination of the water, a certain amount of decomposition of the fatty acid salts present takes place, and in the subsequent extraction more of the soap goes into solution and a smaller residue is obtained. A further objection to this method of valuation is that the amount of fatty acids left behind in combination with the calcium are not taken into account, although they are available material for the manufacture of "stearine." The determination of the percentage of ash also gives no certain criterion for the amount of foreign matter, since on the one hand organic substances are completely burnt off, and on the other, the ash of the calcium soap is left as calcium oxide or as calcium carbonate. But since in bone fat the calcium is chiefly present as a calcium soap, and is but seldom found as calcium phosphate, and then only in small quantity, the authors consider that by determining the amount of calcium in the ash and taking the molecular weight of the fatty acids combined with it at about 260, the percentage of calcium soap present in the fat may be calculated with a nearer approach to accuracy, than it can be determined by extracting the fat with solvents. They recommend that only in ash-free fats should the amount of foreign substances be estimated by extracting the fat with petroleum spirit.—C. A. M.

*Rosin and Rosin Oil in Linseed Oil, Note on the Estimation of.* L. de Koningh. Chem. News, June 1898.

The writer's process is as follows:—The bromine-acidity figure of the sample is taken. If the amount of unsaponifiable matter comes to about 5 per cent., and betrays the presence of rosin oil by its odour, its bromide-acidity figure is also taken. It does not do to liberate the acids from the extracted soap-ley, and then to take their bromide-acidity number; then, even when free from rosin, they will often give an acidity figure high enough to account for 5 per cent. of this substance. The writer believes that this may be accounted for by the water of hydration.

If the original sample gives only a slight acidity, this may be caused by the presence of free fatty acids, but a strong bromine-acidity may be put down to joint rosin-oil and rosin, part of which, however, may be derived from the rosin oil, supposing any of the latter is found in the unsaponifiable matter. Actually added rosin is generally added as a metallic resinate, so if there be no ash, the presence of the true rosin becomes very doubtful.

*Fats and Oils, Cold Saponification of.* K. Dieterich. Helfenberger Ann. 1897, 124—133; Chem. Centr. 1898, **1**, [23], 1209.

The author found almost complete agreement between the values obtained by the hot and cold methods of saponification with hog's lard, pressed tallow, mutton tallow, sweet oil, cod-liver oil, crude yellow and white fatty acids, and stearic acid, but noticeable differences with cocoa butter, mace butter, olive oil and castor oil; rosin oil was found to be quite unsaponifiable by the cold method.—A. S.

*Wax, Examination of, by the Hot and Cold Methods.* K. Dieterich. Helfenberger Ann. 1897, 218–229; Chem. Centr. 1898, **1**, [23], 1209. (See this Journal, 1898, 383, and 1897, 1047.)

On comparing the saponification methods of Hübl and Henriques, it was found that, almost without exception, the values obtained by the cold method are somewhat lower than those by the hot, and that, except in the case of ceresin, where the numbers obtained by the hot method are much too low, ordinary adulteration of wax may be detected by the cold, equally as well as by the hot, method. In doubtful cases, where the hot saponification gives uncertain results, the author considers that the cold method may be used as confirmation. Complete agreement, however, must not be expected, as even with filtered wax a perfect sample cannot be taken, owing to the non-homogeneity of the wax itself. The author agrees with Soltsien, in opposition to Buchner, that the use of either high or low-boiling petroleum spirit for cold saponification, causes no difference to the results.
—A. S.

*Massecuites, Determination of Crystallised Sugar in.* H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1137—1143.]

The massecuite is mixed with an equal weight of a cold saturated solution of sugar and poured into a small centrifugal machine, already in motion. As soon as the sugar clears it is washed with a series of clairces of gradually increasing alcoholic strength, and finally with 96 per cent. alcohol.—L. J. de W.

*Sugar Analysis. The Correction for Unfermentable Reducing Substances in.* T. A. Glendinning. J. Fed. Inst. Brewing, 1898, **4**, 363—365.

Attention has been drawn by Morris (this Journal, 1898, 496) to the necessity of making corrections for certain unfermentable opticalty active and cupric reducing substances which are always present in commercial invert sugars and glucoses. The author shows that the raw cane sugars and syrups from which the invert sugars are made contain unfermentable reducing matter, and he considers that this may be the partial cause of the cupric reduction shown by the residue of fermented invert sugar. The following table illustrates the percentage of unfermentable reducing matter in some typical sugars:—

| | Per cent. |
|---|---|
| Demerara (partly refined) | 0·47 |
| Egyptian | 0·58 |
| Low Egyptian syrups | 1·54 |
| 75 per cent. raw beet | 0·82 |
| Jaggery | 1·67 |
| Low native Penang | 2·24 |
| Native Penang | 2·15 |
| „ Madras | 2·20 |
| Treacle, good quality | 2·57 |
| „ inferior | 2·64 |

There appears to be no relation between the amount of unfermentable reducing matter present in invert sugar and the quality of the sugar—a No. 1 quality frequently requires as much correction for reducing power after fermentation as a No. 3 quality. The author was unable to obtain the unfermentable matter free from ash: the aqueous solutions exhibited a slight dextro-rotation, a cupric reducing power of about 30, and a solution density of D = 4.—J. L. B.

### *Invert Sugar, Estimation of, Colorimetrically.* D. Sidersky. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [11], 1134—1136.

A QUANTITY of the clarified solution prepared for the polarimeter representing 4 grms. of sugar is taken out by a pipette and placed in each of two 100 c.c. flasks, and 5 c.c. of Fehling solution added. One flask is heated to boiling and maintained at the boil for three minutes, 50 c.c. of cold water are added, and the flask is placed in a bath of cold water, in which the second flask, which is not heated, is also placed. When cold, both are filtered. By comparing the colours, the unreduced copper is determined, and from this the invert sugar may be calculated. With dark sugars it is advisable to add 10 per cent. of ammonia, or to acidify with acetic acid and add a few drops of potassium ferrocyanide.—L. J. de W.

### *Organic Matter in Water, A Method for the Differentiation of.* A. G. Woodman. J. Amer. Chem. Soc. 1898, **20**, [7], 497.

FORCHAMMER's potassium permanganate method for the "oxygen consumed" in a water analysis, gives only comparative results, and in no way indicates the absolute amount of carbonaceous matter present; and, further, the results compared are only of value when the organic matter in the various samples has the same composition. The readiness with which organic matter is oxidised by permanganate, varies with its composition and its manner of being present, as also according to how the determination is made.

Barnes (this Journal, 1896, 83) proposed a method for distinguishing different kinds of organic matter by determining the ratio of the oxygen consumed from permanganate to that consumed from chromic acid.

This method has been applied in the case of water, and the following is the procedure:—In the first place, Kubel's hot acid permanganate determination is carried out; 8 c.c. of sulphuric acid (1 : 3) are added to 100 c.c. of the water, then 10 c.c. of $\frac{1}{100}$ normal permanganate. The liquid is boiled for exactly five minutes, and after cooling for one minute, 10 c.c. of $\frac{1}{100}$ normal oxalic acid are added. The excess of oxalic acid is then determined with $\frac{1}{100}$ normal permanganate.

For the oxidation with chromic acid the following solutions are required: (*a*) 6·2 grms. of potassium bichromate dissolved in distilled water, 50 c.c. of concentrated sulphuric acid added, and the solution made up to 1 litre; (*b*) 18 grms. of crystallised ferrous sulphate dissolved in a mixture of 100 c.c. of concentrated sulphuric acid and 200 c.c. of water, and made up to half a litre; solution (*b*) does not keep. To carry out the determination, 50 c.c. of the water are placed in a quarter-litre flask, and 20 c.c. of sulphuric acid (sp. gr. 1·84) are added, then 10 c.c. of the chromic acid (*a*) solution. The flask is shaken and heated for an hour on the water-bath, being covered with a watch-glass. It is allowed to cool for a few minutes, then 10 c.c. of the ferrous sulphate (*b*) solution are added, the whole poured into 500 c.c. of distilled water, and titrated with $\frac{1}{100}$ normal permanganate. A blank determination must be made with water free from organic matter.

The "oxygen ratio" is obtained by dividing the number of c.c. used in the first determination by the number required in the second for an equal amount of water. This "ratio" is generally a fraction less than unity.

Determinations have been carried out on several waters, and from the results obtained, the author concludes that (1) peaty waters give a "ratio" higher than 0·6, and (2) sewage and sewage-contaminated waters give a ratio which is lower than 0·6. The ratio is constant if the character of the organic matter does not change, and is independent of the amount present.—J. McC.

### *Peru and Copaiva Balsams, Examination of, especially for the D.A.B. III. [Deutsch. Arz. Buch. III.]* K. Dieterich. Helfenberger Ann. 1897, 69—74; Chem. Centr. 1898, **1**, [23], 1206—1207.

*Peru Balsam.*—The author has previously tried to show that all commercial varieties of Peru balsam are adulterated, and that they correspond to the natural product very nearly in the acid, ester, and saponification numbers, but not in their content of resin ester and cinnamein. Balsams with the highest possible content of cinnamein and lowest of resin ester are stated to be the most valuable. A sample should contain not less than 65 per cent. of cinnamein and aromatic substances, and not more than 28 per cent. of resin ester. Pure Peru balsam behaves quite different to the D.A.B. III., and a balsam should not be designated as pure just because it corresponds to the D.A.B. III.

For the D.A.B. III., the author recommends the following method of examination:—Sp. gr. 1·135—1·145. Exactly 1 grm. of the sample is dissolved in 200 c.c. of strong alcohol with addition of a few drops of phenolphthalein, and $\frac{1}{10}$ N. alcoholic potash is added until the separated flocks quickly settle and the supernatant liquid appears distinctly dark red. For this purpose, from 10—15 c.c. of the potash should be used.

Exactly 1 grm. of the balsam is weighed out into a litre flask, 50 c.c. of petroleum spirit (sp. gr. 0·700 at 15° C.) and 50 c.c. of $\frac{1}{2}$ N. alcoholic potash added, the flask well stoppered, and allowed to stand 24 hours at the ordinary room temperature. The liquid is diluted with 300 c.c. of water to dissolve the separated resin soap, and then, on titrating back with $\frac{1}{2}$ N. sulphuric acid and phenolphthalein, 40·3—41·5 c.c., corresponding to 8·5—9·7 c.c. of combined potash, should be required. 1 grm. of the balsam is first warmed with ether, and then extracted on a small weighed filter till completely exhausted. The ethereal solution is shaken with 20 c.c. of 2 per cent. caustic soda solution, the ethereal layer run off, and the resin ester precipitated from the alkaline solution by hydrochloric acid. On washing free from chlorine and drying at 80° C., not more than 28 per cent. of resin ester should be obtained. The ethereal solution is allowed to evaporate, then, when the odour of ether is no longer perceptible, placed for 12 hours in the exsiccator, weighed, placed for a further 12 hours in the exsiccator, and weighed a second time. The mean of the two weighings should show not less than 65 per cent. of cinnamein and aromatic compounds.

*Copaiva Balsam.*—The balsam from South American varieties of *copaïfera* is preferable to that from *copaïfera officinalis* and *copaïfera guianensis*. Copaiva balsam, D.A.B. III. is a clear brownish-yellow liquid, not at all or only slightly fluorescent, of peculiar aromatic odour, and of a persistent, sharp, bitter taste. Sp. gr. 0·98—0·99. 1 grm. of the sample is dissolved in 50 c.c. of strong alcohol, and titrated with $\frac{1}{2}$ N. alcoholic potash and phenolphthalein, till a red coloration is produced. Between 2·7 and 3 c.c. should be used for this purpose.

1 grm. of the balsam is treated with 20 c.c. of $\frac{1}{2}$ N. alcoholic potash and 50 c.c. of petroleum spirit (sp. gr. 0·700) in a litre flask, the latter well stoppered and allowed to stand for 24 hours, then, after diluting with alcohol and titrating back with $\frac{1}{2}$ N. sulphuric acid and phenolphthaleïn, 16·75—17 c.c., corresponding to 3—3·25 c.c. of combined potash, should be used.—A. S.

### *Sumatra Benzoin.* K. Dieterich. Helfenberger Ann. 1897, 76—86; Chem. Centr. 1898, **1**, [23], 1207.

THE acid number is estimated in the following manner:—1 grm. of the benzoin taken from a larger average sample of the very finely divided drug, is treated in a small flask with 10 c.c. of $\frac{1}{2}$ N. alcoholic potash and 50 c.c. of strong alcohol, the mixture allowed to stand for exactly 5 minutes—not longer—and then titrated back with $\frac{1}{2}$ N. sulphuric acid and phenolphthaleïn until a yellow coloration appears, that is, until a drop of the acid on falling is no longer coloured red, and the salt separated out, rapidly and completely settles. The supernatant liquid must be coloured pure yellow. The acid number is obtained by multiplying the number of c.c. of combined potash by 28. The saponification number is determined as in Copaiva balsam (see preceding abstract.)

The following limiting values have been deduced from the examination of a large number of commercial samples:—

| Benzoin. | Per Cent. of Ash. | Acid No. | Ester No. | Saponification No. |
|---|---|---|---|---|
| Sumatra .... | 0·368—1·829 | 103·6—132·3 | 65·8—123·2 | 184·8—231·7 |
| Penang ..... | 0·380—0·773 | 121·8—137·2 | 87·5—91·7 | 210·0—226·8 |
| Padang ..... | 1·070 | 121·8—124·6 | 79·8—81·2 | 201·6—205·8 |
| Palembang.. | 1·101—4·023 | 113·4—130·9 | 84—91 | 198·0—219·8 |

It may thus be seen that a distinction between the benzoins from individual provinces is not possible by means of these values, and further that the latter vary within wide limits when the samples are contaminated by vegetable matter. Acid and ester numbers are always influenced by addition of large quantities of foreign matter. A good Sumatra benzoin should show the least possible contamination; should contain at least 70 per cent. of matter soluble in alcohol; should have ash not greater than 1·5 per cent.; acid number, 100—130; ester number, 65—120; and saponification number, 180—230.—A. S.

*Siam Benzoin.* K. Dieterich. Helfenberger Ann. 1897, 87–93; Chem. Centr. 1898, **1**, [23], 1207.

The author does not support the old view that Siam benzoin is free from cinnamic acid. The examination of Siam benzoin is carried out exactly as with Sumatra benzoin (see preceding abstract). The following normal values are given:—Ash, 0·03—1·5 per cent.; matter insoluble in strong alcohol, not more than 5 per cent.; acid number, 140—170; ester number, 50—75; and saponification number, 220—240.—A. S.

*Balsams, Resins and Gum Resins. Examination of.* K. Dieterich. Helfenberger Ann. 1897, 105–111; Chem. Centr. 1898, **1**, [23], 1205.

The author sets out the advantages of quantitative methods as opposed to the old qualitative tests, and he gives the precautions necessary in estimating the acid and saponification numbers. Henriques' method of cold saponification was found to give good results with tests lasting 24 to 48 hours. Many resins, &c. (Peru balsam, Copaiva balsam, benzoin, myrrh, and styrax) are saponified cold by alcoholic alkali and petroleum spirit; with others (ammoniacum, galbanum, euphorbium, gamboge, dragons' blood, and lactucarium), a fractionating saponification is carried out. The number obtained by the action of alcoholic alkali and petroleum spirit is known as the resin number, whilst that from the action of alcoholic and aqueous alkali together is called the saponification number. The author also estimates the acetyl number as a measure of the oxy-acids.
—A. S.

*Spirit, the Nature of the Action of Wood Charcoal in the Purification of.* M. Glasenapp. Zeits. Angew. Chem. 1898, [27], 617.

*See under* XVII., *page* 781.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Nitric Oxide, Absorption of, by Ferrous Salts.* Thomas. Bull. Soc. Chim. 1898, **19**, [10], 419–422.

The researches of Gay (Dissertation, Paris, 1885) have shown that the amount of nitric oxide absorbed by ferrous salts is independent of the nature of the salt and of the degree of dilution of the solution; is proportional to the minimum weight of iron it contains, and is a function of the temperature.

The author confirms Gay in that all solutions of ferrous salts absorb nitric oxide: he finds that these salts preserve this property in other than aqueous solutions, and also that the absorption of nitric oxide varies according to the solvent employed, being, for instance, greater in the case of alcohol than in water. (Compare Graham. Phil. Mag. **4**, 231 and 266.)—J. L. B.

*Copper and Potassium, Double Thiosulphates of.* W. Muthmann and L. Stützel. Ber. **31**, [11], 1732—1735.

*Potassium Cuprithiosulphate*, $Cu(S.SO_3K)_2$, was obtained by mixing solutions of 80 grms. of potassium thiosulphate (prepared from barium thiosulphate and potassium sulphate) and 20 grms. of copper sulphate, diluting to 1 litre and letting the pale yellow liquid stand at the room temperature for 10 hours. Colourless crystals formed, readily soluble even in cold water, and fairly stable in the air. Kept for a few days, the solution becomes slightly dim, owing to separated sulphur. If the crystals be allowed to remain in the mother-liquor for two or three days, a brown colour, due to copper sulphide, is developed. The salt is a cupric compound. Its aqueous solution when treated with NaOH remains colourless and becomes at most, faintly turbid, whereas cuprous double thiosulphates give with NaOH a precipitate of cuprous sulphide. With $K_4FeCy_6$ it gives the reddish-brown precipitate of cupric ferrocyanide, whereas the cuprous compounds give a bright rose-coloured precipitate of cuprous ferrocyanide, which becomes white with HCl. $H_2S$ liberates colloidal cupric sulphide, the precipitation of which is promoted by the addition of acids and indifferent salts. Ammonia does not produce the blue colour which cupric salts usually give; it only appears faintly after some minutes shaking. The substance is evidently not a double salt, but the potassium salt of a complex acid, cuprithiosulphuric acid, $Cu(S.SO_3H)_2$.

*Potassium Cuprous Thiosulphate.*—If solutions of 10 grms. of copper sulphate and 15—20 grms. of potassium thiosulphate are mixed and diluted to 200 c.c., the mixture has a strong yellow colour and soon deposits fine yellow prismatic crystals having the composition—

$$Cu_2S_2O_3.2K_2S_2O_3 + 2H_2O.$$

The salt dissolves readily in warm water, and crystallises as the tetrahydrate from pure aqueous solutions. When crystallized from somewhat dilute thiosulphate solutions it yields a mixture of the di- and tetra-hydrates. It gives all the characteristic reactions of cuprous salts, and is a true double salt.—H. B.

*Manganese in Minerals, Vegetables, and Animals.* P. Pichard. Comptes. Rend. 1898, **126**, [26], 1882—1885.

The author has applied his method (this Journal, 1898, 273), to the examination of the ash of a wide range of animal, vegetable, and mineral substances for the presence of manganese. This metal is found to be almost universally distributed in organised bodies, whether animal or vegetable. In phanerogamous plants, the seeds are extremely rich in manganese, and in cryptogams, the rapidly-growing Hymenomycetes are also well supplied; in the animal kingdom it is detected in many marine animals, also in flesh, eggs, fish, and hair, as well as in cow and horse excrement. The fact that it is found reserved in larger quantities in the seed and rapidly-growing structures, and also its presence in the two last-named manures, is not without significance, as bearing on the important part played by the metal in the organised economy.—J. O. B.

*Lichens; Contribution to the Knowledge of the Constituents of.* O. Hesse. Journ. für prakt. Chem. **57**, [10 and 11], 409—447.

In continuation of his researches on the chemistry of the lichens the author has examined many species. Among others *parmelia perlata*, from various sources, was found to contain *atranorin* and *lecanoric acid*. The grey variety of the lichen was found to give about 0·5 per cent. of this latter acid, and the greenish variety 18·9 per cent. It is to the presence of this acid that the characteristic red coloration of these lichens with chloride of lime solution is due. In one case, from a specimen growing on cinchona bark, another crystalline principle named *perlatin* was isolated. This had the formula $C_{19}H_{14}O_5$ $(OCH_3)_2$. It occurred in long, pale yellow, fragile crystals, which did not lose weight when heated to 120° C. It coloured when heated to 250° C. and partly sublimed; it melted at 274° C. It was separated from atranorin by digestion in potassium monocarbonate solution in which perlatin was insoluble, but atranorin readily dissolved.

*Parmelia physodes* was found to give *atranorin*, *capraric*, and *physodic acids*, and *physol*, an amorphous yellow substance, having the composition $C_{20}H_{24}O_5$. It was neutral, gave a dark green colour with ferric chloride, and, at first, an intense red with chloride of lime, changing to red brown, and finally to yellow. Physodic acid occurs in small white needles melting at 190°—192° C., and has the composition $C_{30}H_{22}O_6$. On prolonged boiling with alcohol, acetone, or other solvents, the crystalline acid was converted into an amorphous modification. The so called *ceratophyllin* of Gerding was found to be identical with physodin.

*Parmelia caperata* was found to contain *capraric acid*, which occurs in white needles, which decompose without melting between 240°—260° C. The formula $C_{21}H_{20}O_{12}$ is assigned to it; also *caperatic acid* in shining plates or needles, having the composition $C_{22}H_{35}O_7$ $(OCH_3)$, and melting at about 132° C. *Caperin* in white prisms, to which the formula $C_{30}H_{60}O_3$, is attributed. It melts at about 243° C. *Caperidin* $C_{24}H_{40}O_2$ forming white scales or short prisms, melting at 262° C., and *usninic acid* $C_{18}H_{16}O_7$, melting at 196° C.

*Candelaria concolor* was found to contain *dipulvinic acid* $C_{36}H_{22}O_9$, crystallising in minute brick red needles melting at 211° C.

*Sticta pulmonaria* gave *proto-cetraric acid* $C_{30}H_{22}O_{15}$. *Nephromium arcticum* yielded a new crystalline body termed *nephrin*, forming white needles melting at 168° C. Its composition proved to be $C_{20}H_{32}+H_2O$. It is therefore a diterpine hydrate. The same body was found in *Nephromium lusitanicum* as well as *nephromin* $C_{16}H_{12}O_6$, forming small ochre-coloured needles, melting at 196° C.

—J. O. B.

*Tropine Pinacone.* R. Willstatter. Ber. 1898, **31**, [11], 1672—1675.

In the reduction of the ketone tropinone (obtained from tropine) by means of sodium, ψ-tropine is obtained as the chief product, accompanied by a small quantity of a base, $C_{16}H_{28}N_2O_2$. This is the pinacone of tropine. No benzoyl esters could be obtained.—A. C. W.

*Anthranilic Acid, The Fluorescence of.* B. Pawlewski. Ber. 1898, **31**, [11], 1693.

In aqueous solution anthranilic acid shows a feeble blue fluorescence, solutions in alcohols, fatty acids, ketones, and esters exhibit a more decided fluorescence, whilst in ether, benzene, chloroform, &c., a fine violet fluorescence appears. The most beautiful appearance is given by solutions in oenanthol, oleic acid, and glycerin. The phenomenon is strongly marked in aqueous formaldehyde solutions. In carbon bisulphide, carbon tetrachloride, formic acid, and aniline, no fluorescence appears. In all cases the fluorescence is destroyed by the addition of caustic alkalis.

—A. C. W.

*Mixed Carbonic Esters of the Fatty and Aromatic Series, General Method for the Preparation of.* P. Cazeneuve and A. Morel. Comptes Rend. 1898, **126**, [26], 1871—1873.

The authors effect the preparation of the mixed carbonic esters, by heating neutral phenolic carbonates, in the presence of certain organic bases, in various alcoholic media. Thus phenyl carbonate, when heated with urea in many homologous alcohols of the fatty series, gives combined carbonates of phenyl with these other alcohols. In this way the compound carbonates of phenyl-methyl, phenyl-ethyl, phenyl-propyl, and of secondary propyl, &c., and even phenyl-allyl carbonate, have been obtained. The lower members of the series are obtained in a very pure condition, but the higher compounds are more difficult to purify, in consequence of the formation of allophanates and urethanes, which distil over, even in vacuo. Other bases, such as aniline, pyridine, and dimethylpiperazine, act in a similar way, without the formation of these impurities. By substituting other carbonates, such as those of guaiacol, of thymol, or of cresol, analogous double compounds are obtained. The intervention of the bases is essential, since unless they are present, either no reaction takes place, or, at the best, it is extremely slow. The bases doubtless first form alcoholates, which afterwards undergo double decomposition. It is known that sodium ethylate acts in a similar manner with phenolic carbonates, giving by double decomposition the double carbonate, and the sodium salt of the base.—J. O. B.

## New Books.

COMMITTEE OF REVISION AND PUBLICATION OF THE PHARMACOPŒIA OF THE UNITED STATES OF AMERICA. 1890—1900. ALKALOIDAL ESTIMATION. A Bibliographical Index of Chemical Research prepared from Original Literature for the Committee of Revision. By PAUL I. MURRILL, under the Direction of ALBERT B. PRESCOTT. Published by the Commission of Revision. Ann Arbor. 1898. [Not for sale.]

A SHORT COURSE IN INORGANIC QUALITATIVE ANALYSIS FOR ENGINEERING STUDENTS. By J. S. C. WELLS, Ph.D., Instructor in Analytical Chemistry, Columbia University. First Edition. John Wiley and Sons, New York. Chapman and Hall, Limited, London. 1898. Price 6*s*. 6*d*.

8vo volume containing preface, table of contents, 291 pages of subject-matter, and alphabetical index. The subject-matter is subdivided into three sections. SECTION I. INTRODUCTION.—Grouping of the Metals. Acids. Bases and Salts. Chemical Equations. REACTIONS OF THE METALS. Groups I., II., III., IV., V., and VI. SECTION II. Groups I., II., and III., SECTION III. COMPLETE ANALYSIS. Preliminary Examination. DISSOLVING SOLID SUBSTANCES. INSOLUBLE SUBSTANCES. METALS OR ALLOYS. SILICATES. INSOLUBLE CYANIDES. REAGENTS.

A LABORATORY GUIDE IN QUALITATIVE CHEMICAL ANALYSIS. By H. L. WELLS, M.A., Professor of Analytical Chemistry and Metallurgy in the Sheffield School of Yale University. First Edition. John Wiley and Sons, New York; Chapman and Hall, Limited, London. 1898. Price 1·50 dols.

THIS is a book of 200 pages, 8vo size, containing preface and table of contents. The last 33 pages are composed of labels for bottles, perforated, for tearing off. The subject-matter is subdivided generally as follows:—PART I. ANALYTICAL COURSE. PART II. Theory. PART III.—Descriptive.

JAHRBUCH DER ELEKTROCHEMIE. Berichte über die Fortschritte des Jahres 1897. Unter Mitwirkung der Herren Prof. Dr. ELBS, Giessen, Prof. Dr. F. W. KUSTER, Breslau, and Dr. H. DANNEEL, Gottingen. Herausgegeben von Dr. W. NERNST and Dr. W. BORCHERS. IV. Jahrgang. Wilhelm Knapp, Halle a/S. 1898. Price M. 15. H. Grevel and Co., 33, King Street, Covent Garden, W.C.

THIS Year Book of Electrochemistry forms an 8vo volume, containing 382 pages of subject-matter, illustrated with 206 engravings, exclusive of the preface and table of contents at the beginning, and of a classified list of the publications relating to the subject of electrochemistry for the year 1897, and the alphabetical index, at the end. The subject-matter is classified as to arrangement, as follows:—SECTION I. ELECTROCHEMICAL SCIENCE.—General, Educational. Theories. Lecture Experiments. Apparatus and Methods. Conductivity and Dissociation. Electrical Energy. Polarisation and Electrolysis. Dielectric Conductivity. Various (Danneel). Electro-analysis (Küster). Section II. Applied Electrochemistry.—Production of Electrical Energy. Electrical Energy from Chemical Energy. Accumulators. Other Sources of Energy (Elbs). Electromagnetic Preparation. Electrothermic Apparatus and Processes. Metalloids. Metals. A. Special Metallurgy. B. Investigations and Proposals of Universal Applicability. C. Working of Metals, Galvanoplastic, &c. Inorganic Compounds (Borchers). Organic Compounds (Elbs). Bleaching and Disinfecting (Borchers).

A Manual of Chemical Analysis, Qualitative and Quantitative. By G. S. Newth, F.I.C., F.C.S., Demonstrator in the Royal College of Science, London. Longmans, Green, and Co., 39, Paternoster Row, London; also in New York and Bombay. 1898. Price 6*s.* 6*d.*

Small 8vo volume, containing preface, table of contents, subject-matter filling 455 pages, and an alphabetical index. The text is illustrated with 100 well-executed engravings. The importance of the work may be judged from the following excerpt of the contents:—

Book I. Qualitative Analysis. I. Preliminary Exercises. II. Analytical Classification. III. Reactions of the Metals of Group V. IV., V., and VI. Reactions of the Metals of Groups IV., IIIa., and IIIb. VII. The Phosphates. VIII. and IX. Reactions of the Metals of Group II., Divisions 1 and 2. X. Reactions of the Metals of Group I. XI to XVII. Non-Metals and their Acids. The Halogens. Sulphur. Nitrogen and Phosphorus. Carbon, Silicon, and Boron. Detection of Acids. Preliminary tests and operations in a systematic analysis. The results of a qualitative analysis. Book II. Quantitative Analysis. Part I. Gravimetric Analysis. Sect. I. Preliminary manipulations. II. Typical gravimetric determinations of Metals. III. Typical gravimetric determinations of Acids. IV. Exercises in gravimetric analysis. V. Electrolytic methods of analysis. Part II. Volumetric Methods. Sect. I. Preliminary manipulations. II. Volumetric methods based upon saturation. III. Methods based upon oxidation and reduction. IV. Methods based upon precipitation. V. Gas analysis. Part III. Sect. I. Estimation of Carbon, Hydrogen, Nitrogen, Chlorine, Sulphur, and Phosphorus in organic compounds. II. Miscellaneous physico-chemical determinations. Appendix, with Tables of Elements and Atomic Weights. Reagents. System of standard strengths. Table of hardness. Expansion of water between 0° and 25°. Tension of aqueous vapour from 5° to 25°, and Table of factors for the correction of gaseous volumes.

# Trade Report.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

### Canada.

#### *Preferential Tariff.*

*Bd. of Trade J., August*, 1898, 194.

The following information has been received at the Board of Trade from the High Commissioner for Canada:—

By an Act of the Parliament of Canada, assented to on the 13th of June, 1898, it is provided:

"1. That all articles except wines, malt liquors, spirits, spirituous liquors, liquid medicines and articles containing alcohol; tobacco, cigars, and cigarettes, which are the growth, produce, or manufacture of any of the following countries, and which are declared to be dutiable upon importation into Canada, may, when imported direct into Canada from any of such countries, be entered for duty or taken out of warehouse for consumption in Canada, on and after the first day of August, 1898, at a reduction of one-fourth from the general tariff rates of duty."

The Preferential Tariff will apply to the United Kingdom, Bermuda, the British West Indies, British Guiana, British India, Ceylon, New South Wales, and the Straits Settlements. It may also be extended by Order in Council to any other British colony or possession, the customs tariff of which on the whole is as favourable to Canada as the British Preferential Tariff of Canada is to such colony or possession.

Raw sugar, including sugar drainings or pumpings drained in transit, melado or concentrated melado, tank bottoms and sugar concrete, the produce of any British colony or possession, may, when imported direct from any British colony or possession, be entered for duty or taken out of warehouse for consumption in Canada at the reduced rate of duty provided in the British Preferential Tariff.

The reduction under the Preferential Tariff will only apply to refined sugars when evidence satisfactory to the Minister of Customs is furnished that such refined sugar has been manufactured wholly from raw sugar produced in the British colonies or possessions.

Copies of the "Memorandum of Information and Instructions respecting the British Preferential Tariff," issued by the Canadian Customs Department, of the "Regulations for the entry of goods under the British Preferential Tariff of Canada," and of the "Schedule of forms" that are to be used, as referred to in the regulations, may be obtained from the High Commissioner for Canada, 17, Victoria Street, London, S.W., or from any of the Canadian Government Agents in the United Kingdom.

### Netherlands.

#### *Sodium and Calcium Acetates.*

A despatch, dated 14th July last, has been received from Her Majesty's Minister at The Hague, transmitting copy and translation of a Royal Decree, according to which "exemption from import duty shall be granted for sodium and calcium acetates required in the work of dyeing and printing textile fabrics, provided that those acetates by mixture with one or more other substances are rendered incapable of having vinegar extracted from them for household consumption."

### Russia.

#### *Traumatol.*

The Russian Customs Department notifies that "Le Traumatol," a chemical product (iodocresol), not specially named in the Customs tariff, may be imported duty free, under section 112 in the form of goods, but no description of its medicinal qualities or its application to diseases must appear on the labels of the packages or explanatory matter accompanying such package.

### United States.

#### *Duty on Sheep Dips, &c.*

Japanese needle antimony is dutiable at 20 per cent. *ad valorem* as "metal, unwrought," under paragraph 183, Act of 1897.

Hides sent from India to a European port for shipment to the United States require disinfection at the port of shipment if no evidence of disinfection in India is produced by the skipper.

Coal-tar sheep-dip is dutiable as a chemical compound, under paragraph 3 of Act of 1897, at 25 per cent. *ad valorem.*

Tobacco sheep-dip is assessed for duty at the rate of 20 per cent. *ad valorem* as an unenumerated manufactured article, under section 6 of the Act of 1897.

Sheep dip composed of sulphide of arsenic is entitled to free entry, under paragraphs 479 and 657 of the Act of 1897.

#### Duty on Coal-Tar Products.

*Eng. and Mining J., July* 23, 1898, 94.

Assistant Secretary Howell, of the Treasury Department, has just issued a new decision, reversing one of March 31st, 1898, which is important to manufacturers and dealers in dyestuffs, anilines, and chemicals. The decision is based on representations on behalf of domestic dealers and manufacturers that the indigo artificially prepared from coal-tar products, referred to in the instructions of March 31st, was not, in fact, definitely, uniformly, or generally designated as indigo by merchants and importers. It differs in essential particulars, and materially in its chemical properties, from the Bengal indigo derived from plants. The Department, therefore, revokes the order of March 31st last, and makes indigo artificially prepared from coal-tar dutiable at 30 per cent. *ad valorem*, as a coal-tar colour or dye, under paragraph 15 of the Act of 1897, thereby giving importers an opportunity to carry the matter before the Board of General Appraisers.

### SPANISH EXPORT DUTIES IN 1898–99.

*Board of Trade J., Aug.* 1898, 181.

The Board of Trade have received through the Foreign Office copy of a translation of the tariff of Spanish export duties imposed by the Spanish Budget law of 28th June last, as follows:

| Tariff No. | Articles. | | Valuation for 1897. | Duties. |
|---|---|---|---|---|
| | CLASS I. | | | |
| | STONES, EARTHS, MINERALS, GLASS AND GLASSWARE AND CERAMIC PRODUCTS. | | | |
| | *Group 1. Stones and Earths employed in Buildings, Arts, and Industry.* | | Pesetas. | Pesetas. |
| 4 | Steatite | Per 100 kilos. | 8·0 | 0·20 |
| 4A | Wash-ball (*jaboncillo*) | ,, | 16·0 | 0·40 |
| 5 | Lime | ,, | 4·0 | 0·10 |
| 6 | Lime, hydraulic and cement. | ,, | 3·50 | 0·19 |
| 7 | Gypsum | ,, | 2·0 | 0·05 |
| 8 | Kaolin | ,, | 5·0 | 0·10 |
| 8A | Fluor-spar, heavy spar | ,, | 2·0 | 0·05 |
| 8B | Other stones and earthen substances employed in building, in the arts, and in industry. | ,, | 2·0 | 0·05 |
| 10 | Mineral waters | Hectolitre | 80·0 | 2·00 |
| | *Group 2.—Coal, &c.* | | | |
| 11 | Coal and coke | Ton of 1,000 kilos. | 27·0 | 0·65 |
| 12 | Jet | ,, | 76·0 | 1·90 |
| 13 | Tar, pitch, creosote, bitumen, and products derived therefrom. | Per 100 kilos. | 25·0 | 0·60 |
| | *Group 3.—Metallic Minerals.* | | | |
| 14 | Antimony or galena, non argentiferous. | Ton of 1,000 kilos. | 225·0 | 5·50 |
| 15 | Antimony or galena, argentiferous, when exported to France, Algeria, or Portugal. | ,, | 600·0 | 15·00 |
| 15A | Antimony or galena, argentiferous, when exported to other countries. | ,, | 300·0 | 7·50 |
| 16 | Other lead ores | ,, | 190·0 | 4·75 |
| 17 | Blende | ,, | 40·0 | 1·00 |
| 18 | Calamine | ,, | 48·0 | 1·20 |
| 19 | Phosphorus | ,, | 10·0 | 0·25 |
| 20 | Antimony | ,, | 300·0 | 7·50 |
| 21 | Copper | ,, | 34·0 | 0·85 |
| 22 | Copper blende ("*mata bronce*"). | ,, | 520·0 | 13·00 |
| 23 | Iron | ,, | 11·0 | 0·25 |
| 24 | Iron pyrites | ,, | 12·0 | 0·30 |
| 25 | Carbonate of manganese. | ,, | 20·0 | 0·50 |
| 25A | Other mineral manganese. | ,, | 57·0 | 1·40 |
| | *Group 4.—Glass and Glassware.* | | | |
| 26 | Common or ordinary glassware. | Per 100 kilos. | 35·0 | 0·85 |
| 27 | Crystal, and glass imitations thereof. | Per kilo. | 165·0 | 4·10 |
| 28 | Plate glass | ,, | 80·0 | 2·00 |
| 29 | Looking glasses and mirrors. | ,, | 350·0 | 8·75 |
| 30 | Broken glass and crystal. | ,, | 25·0 | 0·60 |
| | CLASS II. | | | |
| | METALS AND METAL MANUFACTURES. | | | |
| | *Group 3.—Copper and its Alloys.* | | | |
| 49 | Copper shell | Per 100 kilos. | 80·0 | 2·00 |
| 50 | Black copper and old copper, bronze, or brass. | ,, | 125·0 | 3·10 |
| 51 | Black copper and old copper, bronze, or brass, in "*torales*". | ,, | 160·0 | 4·00 |
| 52 | Black copper and old copper, bronze, or brass, in bars. | ,, | 150·0 | 3·75 |
| 53 | In sheets and nails | ,, | 185·0 | 4·60 |
| 54 | Brass, in sheets | ,, | 175·0 | 4·35 |
| 55 | Copper, brass, and bronze, manufactured in any form. | ,, | 500·0 | 12·50 |
| | *Group 4.—Other Metals.* | | | |
| 56 | Quicksilver or mercury. | Per 100 kilos. | 540·0 | 13·50 |
| 57 | Tin | ,, | 200·0 | 5·00 |
| 58 | Argentiferous lead in ingots, when exported to France, Algeria, Italy, or Portugal. | ,, | 35·0 | 1·75 |
| 58A | Argentiferous lead in ingots, when exported to other countries. | ,, | 35·0 | 0·85 |
| 59 | Argentiferous lead in ingots, when exported to other countries, poor. | ,, | 26·0 | 0·65 |
| 60 | Lead, in tubes | ,, | 40·0 | 1·00 |
| 61 | Lead, manufactured in any form. | ,, | 38·0 | 0·95 |
| 62 | Zinc, in bars and plates | ,, | 55·0 | 1·35 |
| 63 | Other metals and their alloys. | ,, | 200·0 | 5·00 |
| | CLASS III. | | | |
| | SUBSTANCES EMPLOYED IN PHARMACY, PERFUMERY, AND CHEMICAL INDUSTRIES. | | | |
| | *Group 1.—Simple Drugs.* | | | |
| 64 | Oil of almonds | Per 100 kilos. | 360·0 | 9·00 |
| 65 | Seed oils | ,, | 109·0 | 2·70 |
| 66 | Refuse of olive oil | ,, | 38·0 | 0·95 |
| 67 | Bark and other tanning materials. | ,, | 14·0 | 0·35 |
| 68 | Pea nuts, monkey nuts ("*cacahuet*"). | ,, | 44·0 | 1·10 |
| 69 | Liquorice | ,, | 35·0 | 0·85 |
| 70 | Liquorice, in extract or paste. | ,, | 115·0 | 2·85 |
| 71 | Other vegetable products. | ,, | 110·0 | 2·75 |
| | *Group 2.—Colours.* | | | |
| 72 | Ochres and natural earths, for painting. | Per 100 kilos. | 8·0 | 0·20 |
| 72A | Colours in powder or lumps. | ,, | 30·0 | 0·75 |
| 73 | Colours, prepared, paints and varnishes. | ,, | 130·0 | 3·25 |
| 74 | Saffron | ,, | 250·0 | 6·25 |
| 75 | Cochineal | ,, | 400·0 | 10·00 |
| | *Group 3.—Chemical Products.* | | | |
| 76 | Sulphur | Per 100 kilos. | 13·0 | 0·30 |
| 77 | Chloride of sodium (common salt). | ,, | 1·50 | 0·05 |
| 78 | Tartar, crude, and lees of wine. | ,, | 54·0 | 1·35 |
| 79 | Cream of tartar | ,, | 185·0 | 4·60 |
| 80 | Red lead | ,, | 45·0 | 1·10 |
| 81 | Argentiferous litharge | ,, | 65·0 | 1·50 |
| 82 | Non-argentiferous litharge. | ,, | 45·0 | 1·10 |
| 83 | Common glue | ,, | 85·0 | 2·10 |
| 84 | Glycerine | ,, | 100·0 | 2·50 |
| 85 | Chemical products, not specified. | ,, | 65·0 | 1·60 |
| 86 | Chemical products, pharmaceutical. | Per kilo. | 8·0 | 0·20 |

| Tariff No. | Articles. | | Valuation for 1897. | Duties. |
|---|---|---|---|---|
| | CLASS III.—*cont.* | | | |
| | SUBSTANCES EMPLOYED IN PHARMACY, PERFUMERY, AND CHEMICAL INDUSTRIES—*cont.* | | | |
| | *Group* 4.—*Various.* | | | |
| 87 | Starch.................. | Per 100 kilos. | 55·0 | 1·35 |
| 88 | Common soap......... | „ | 52·0 | 1·30 |
| 89 | Stearine and wax, in lumps. | „ | 150·0 | 3·75 |
| 90 | Stearine and wax, in candles. | „ | 165·0 | 4·10 |
| 91 | Perfumery and essences. | Per kilo. | 8·0 | 0·20 |

## *DIPLOMATIC AND CONSULAR REPORTS.*

### EMPLOYMENT OF ARTIFICIAL SWEETENING MATERIALS IN ARTICLES OF FOOD IN GERMANY.

*Board of Trade J., Aug.*, 1898, 201.

Her Majesty's Chargé d'Affaires at Berlin transmits text of a Law respecting the trade in artificial sweetening materials of 6th July, 1898.

*Section* 1.—Artificial sweetening materials in the sense of this Law are all substances obtained by artificial means, and possessing a higher sweetening power than refined cane or beetroot sugar, without a correspondingly nourishing value.

*Section* 2.—The use of artificial sweetening materials in the preparation of articles of food or refreshment shall be considered as adulteration in the sense of Section 10 of the Law respecting the traffic in articles of food, refreshment, or general use of the 14th May, 1879.

Articles of food or refreshment, in the preparation of which artificial sweetening materials have been employed, may only be sold or offered for sale under a denomination which distinctly indicates the fact of such employment.

*Section* 3.—It is prohibited :—

1. To employ artificial sweetening materials in the wholesale preparation of beer, wine, or beverages resembling wine, fruit cordials, preserves, liqueurs, and syrups.
2. To sell or offer for sale beverages of the kind described under 1 to which artificial sweetening materials have been added.

*Section* 4.—Any person who intentionally contravenes the provisions of Section 3 shall be punished by imprisonment up to six months, and by a fine up to 1,500 marks (75*l.*), or by one of these penalties.

If the contravention has been due to negligence it shall be punished by a fine of 150 marks (7*l.* 10*s.*) or detention.

Besides the above punishments, the confiscation of the articles prepared for sale in contravention of the prohibition may be ordered. If the prosecution or conviction of a particular person is not practicable, the confiscation can be ordered independently thereof.

The provisions of Sections 16 and 17 of the Law of May, 1879, apply in this case also.

### CHEMICALS, &c. EXPORTED FROM YOKOHAMA IN 1897—1896.

*Foreign Office Annual Series, No.* 2165,

| Articles. | | 1897. | | 1896. | |
|---|---|---|---|---|---|
| | | Quantity. | Value. | Quantity. | Value. |
| | | | £ | | £ |
| Drugs, dyes, and paints. | Lb. | 385,722 | 8,035 | .. | 7,335 |
| Manganese ....... | Tons | 10,546 | 15,912 | 13,398 | 20,386 |
| Menthol crystal .. | Lb. | 32,823 | 10,295 | 70,757 | 30,725 |
| Sulphur .......... | „ | 4,131,644 | 6,060 | 8,667,672 | 9,403 |
| Soy ............... | Galls. | 206,966 | 8,894 | .. | 6,091 |
| Porcelain and earthenware. | | .. | 57,122 | .. | 79,455 |
| Paper .................. | | .. | 24,319 | .. | 34,182 |

### CHEMICALS, &c. IMPORTED INTO YOKOHAMA IN 1896—1897.

| Articles. | | 1897. | | 1896. | |
|---|---|---|---|---|---|
| | | Quantity. | Value. | Quantity. | Value. |
| | | | £ | | £ |
| Alcohol .......... | Lb. | 13,741,323 | 86,222 | 6,420,321 | 41,594 |
| Bismuth subnitrate | „ | 26,512 | 5,624 | 43,584 | 6,851 |
| Dyes: | | | | | |
| Alizarine ....... | „ | 92,824 | 10,631 | 97,556 | 13,158 |
| Aniline ......... | „ | 774,316 | 58,413 | 860,104 | 68,.39 |
| Drugs and chemicals.... | | .. | 103,519 | .. | 76,016 |
| Dyes and paints ........ | | .. | 18,539 | .. | 16,645 |
| Dynamite ........ | Lb. | 471,304 | 20,399 | 197,133 | 8,962 |
| Indiarubber ware....... | | .. | 16,949 | .. | 20,463 |
| Logwood extract.. | Lb. | 1,020,844 | 16,667 | 1,376,022 | 23,158 |
| Oil and wax ............ | | .. | 22,031 | .. | 20,098 |
| Paint in oil ....... | Lb. | 2,159,483 | 20,199 | 1,790,056 | 15,477 |
| Paper, printing... | „ | 9,634,829 | 66,817 | 5,929,219 | 47,779 |
| Paraffin wax....... | „ | 1,147,277 | 7,539 | .. | .. |
| Perfumery and cosmetics | | .. | 5,169 | .. | .. |
| Portland cement.. | Tons | 22,815 | 35,415 | .. | .. |
| Potassium chlorate | Lb. | 536,372 | 8,079 | .. | .. |
| Soda, bicarbonate. | „ | 1,829,865 | 5,219 | 2,016,767 | 5,669 |
| Soda, caustic...... | „ | 3,942,996 | 10,602 | .. | .. |

### MINERALS AND CAOUTCHOUC IN NEW CALEDONIA.

*Foreign Office Annual Series, No.* 2157.

Mineral products continue to be the principal articles of export, and show an increase in value of over 16,000*l.* The monthly export of mineral is shown in the following table :—

*Quantity of Minerals and Ores Exported in* 1897.

| Nature of Ores. | From January to August. | In September. | In October. |
|---|---|---|---|
| | Tons. | Tons. | Tons. |
| Nickel................ | 6,675,646 | 9,062,927 | 10,230,705 |
| Cobalt................ | 781,219 | 127,000 | 343,896 |
| Chrome .............. | 196,000 | 387,000 | 586,366 |
| Argentiferous lead.... | .. | 2,000 | .. |
| Copper ............... | 2,840 | .. | .. |
| Iron.................. | .. | .. | .. |
| Total of monthly shipments. | 7,655,705 | 9,578,927 | 11,160,967 |

| Nature of Ores. | In November. | In December. | Total, |
|---|---|---|---|
| | Tons. | Tons. | Tons. |
| Nickel................ | 2,240 000 | 5,562,024 | 33,771,302 |
| Cobalt................ | 859,266 | 89,500 | 2,200,881 |
| Chrome .............. | 138,000 | 150,250 | 1,457,616 |
| Argentiferous lead.... | .. | .. | 2,000 |
| Copper ............... | .. | .. | 2,840 |
| Iron.................. | 25,000 | .. | 25,000 |
| Total of monthly shipments. | 3,262,266 | 5,801,774 | 37,459,639 |

NOTE.—All the nickel ores were shipped to Europe, the cobalt to Europe and Australia, the chrome solely to Sydney, and lead copper, and iron to Sydney as samples.

In last year's report mention was made of the floating of certain mining companies. An Anglo-French society has been formed, who are now opening out mines on the north of the island, mostly gold, copper, and lead ores. Nickel mining is still showing increased activity, and new works are being opened in all parts of the colony. The French "Société Le Nickel" have sent out engineers to report with a view to the construction of smelting works. Great hopes are entertained of the mining industry in New Caledonia attracting foreign capital, and thus opening out the valuable mineral deposits of the country.

Several attempts have been made to exploit the coal deposits found at a short distance from Noumea. There is no doubt that coal of a fair quality exists on the western coast of the island, but no real attempt on a large scale has been made to show whether a paying coalfield has been reached.

M. le Gouverneur Feillet, returning from leave, brought with him 10,000 seeds of the Caoutchouc Ceara. These he distributed through the "Union Agricole" to several of the settlers for trial. The result has been marvellously successful. With a few exceptions most of the seeds have sprouted, and the trees, already of a respectable height, have withstood the late cyclone very well, and all the settlers who have made a trial of this tree are quite agreed as to its future, and although the experience is somewhat short, it nevertheless shows that the C. Ceara does not require a hot and moist climate for its development, as is the case with other species of the india-rubber tree. The demand for india-rubber increases every day, and the supply falls far short of it.

### Coke Imports of California in 1897.

*Foreign Office Annual Series, No.* 2152.

The total imports of coke in 1897 were 30,320 tons, being 5,812 tons less than in 1896. Of this, 2,573 tons came from British Columbia and 1,889 tons from Australia; these are two new sources of supply for this market. It is understood that the plant in British Columbia, where some of the above-mentioned coke was manufactured, is being enlarged to double its present capacity, and it is likely that the imports from England and Belgium will be affected by this enterprise as soon as it gets into full running order.

### Tin-plate in California in 1897.

*Foreign Office Annual Series, No.* 2152.

The imports of tin plates show a heavy decline as compared with 1896, 65,418 boxes less than that year having been received. These articles are being manufactured in the Eastern States in increasing quantities, and as heavy consignments are shipped to this market they are rapidly superseding the imported plates. It is said they can be laid down here for 1 dol. per box less than the foreign ones, and it is only a question of time when they will entirely displace them. It is understood that the imported article is now principally used in packing goods intended for export, in which case a drawback on the duty is allowed.

### Cement in California in 1897.

*Foreign Office Annual Series, No.* 2152.

The imports of cement show a decrease of 117,845 barrels as compared with 1896. Belgium supplied the greatest amount, as will be seen from the following figures taken from the custom-house records:—

| Country. | Quantity. |
|---|---|
| | Lbs. |
| Belgium | 40,015,610 |
| Great Britain | 30,191,656 |
| Germany | 17,694,800 |
| France | 1,764 |
| Total | 87,903,830 |

The past year cannot be said to have been a very active one, although there was a fairly steady demand for most descriptions during the year. It is expected that the market will take an increasing quantity, but there is always enough on hand to meet any demand that may be made. As will be noticed from the above figures, Great Britain has fallen to the second place in supplying this market. This is said to be entirely owing to the course pursued by the producers. It is alleged that after supplying a certain quantity at a price agreed upon they will ask an advance on a further consignment of the same brand. Dealers here have become so disgusted with this method of doing business that they have transferred their orders to Belgian and German firms, who can be relied on to supply any quantity of a certain quality at a fixed price. The quality of the British article is said to be quite satisfactory, and the only reason given for its losing ground is the unsatisfactory way it is handled by the British producer.

### Chemicals in California in 1897.

*Foreign Office Annual Series, No.* 2152.

Owing to the extended operations of the chemical manufacturers of the Eastern States, and the increase in the duty under the new tariff, the importation of chemicals into this market is likely to fall off materially.

### Sugar Beet Culture in California in 1897.

*Foreign Office Annual Series, No.* 2152.

Sugar beet culture is expected to become one of the leading industries of this State in the near future. Last year there were four large factories in operation, their capacity being given as follows:—

| Location. | Daily Capacity. |
|---|---|
| | Tons of Beets. |
| Alvarado (refined sugar) | 800 |
| Watsonville (raw sugar) | 1,000 |
| Chino (refined sugar) | 800 |
| Alamitos (refined sugar) | 350 |

The following factories are under construction and are expected to commence operations this year:—

| Location. | Daily Capacity. |
|---|---|
| | Tons of Beets. |
| Salinas | 3,000 |
| Crockett | 1,000 |
| Hueneme | 2,000 |

The mills at Alvarado and Alamitos are in process of enlargement, and it is stated that a factory will be erected at Carritos, near Alamitos, by those interested in the latter concern. The large amount of money required to build and equip one of these factories has hitherto militated against the increase of the industry, as capitalists were naturally unwilling to invest until assured of success. Now that sugar has received increased protection under the new tariff, and it has been demonstrated that it can be profitably produced on this coast, there is likely to be a considerable increase in the business.

The president of the sugar beet factory at Alvarado, one of the oldest established factories in the State, caused a count of the farmers to be made who were growing beets under contract for his establishment in 1896. The number was found to be 120, some of whom grew a few acres and some many, the average being 15 acres per farmer. A great deal has been said about the desirability of having more small farms in California, and this industry certainly promises to be a factor in bringing about that result.

### Adulteration of Foodstuffs in California.

*Foreign Office Annual Series, No.* 2152.

Under an increased appropriation granted to that department, the Board of Health has been enabled to establish and equip two laboratories: one for the examination of food and the other for bacteriological investigation. An official chemist with an assistant has been placed in charge of the chemical laboratory, and is constantly engaged in the analysis of food-stuffs. The chemist renders monthly reports, and where his analysis shows articles purchased to be adulterations, or substitutions, warrants are promptly issued for the arrest of the offending dealers. All brands of condensed milk, catsups, and jellies have been inspected and analysed, and many palpable and startling adulterations have been made known to the public. Out of 31 samples of tomato catsup only two were found to be free from antiseptics, the remainder were found to contain either salicylic acid or boracic acid, and 16 were coloured with aniline dyes. 14 samples of condensed milk and evaporated cream were examined and seven found to be approximately pure condensed milk, representing a standard of about three-parts of pure milk condensed to one part. The remainder seemed to have been made either from skim-milk or milk which was very poor in butter fat. Manufacturers of these goods are now said to be making laudable efforts to bring their products up to a uniform standard.

The analytical data regarding fruit jellies, jams, and marmalades shows that in no line of food products has adulteration and substitution been carried to such an alarming extent. Of 87 samples analysed, 28 only had the chance of being called pure, 20 were adulterated and 39 fraudulent substitutes. A majority of those, although passed as pure jellies made from their respective fruits, were not made from choice fruit; some were made from immature fruit, and some from over-ripe fruit that had already passed into a state of incipient putrefaction.

### Wine Storage in California.

*Foreign Office Annual Series, No.* 2152.

An experiment in handling red wine was tried last year at the Italian-Swiss colony's vineyard situated at Asti in this State. The grapes handled by the colony were far in excess of the cooperage facilities it possessed, and some means had to be devised to care for the surplus. Among the different plans suggested was that of building a concrete cistern, and this idea was finally adopted. An excavation was first made in a rocky hillside in the rear of the establishment. Next, walls of concrete 2 ft. in thickness were put in, and the floor and top were added in as substantial a manner, the latter being supported by 15 steel girders. Then the entire surface was covered with a lining of pure cement, and finally this was glazed to the impermeability of glass. The whole cistern was buried beneath 3 ft. of earth, the object of all these precautions being to preserve the wine at a uniform temperature. This cement tank is 104 ft. long, 34 ft. wide, and 24 ft. high, and is capable of holding 500,000 galls. The wine was kept in this reservoir for four months or more, and the experiment is said to have been entirely successful. It was then drawn off by gravitation into wooden tanks in which it will be aged previous to being placed in barrels for shipment. There are said to be several advantages derived from treating the wine in this manner. One is that it can be maintained at a cool even temperature; another is the equal blending of such a large quantity of wine at one time; and a third is the great saving in insurance which is expected to repay the cost of the construction of the tank in five years.

### Camphor in South Formosa.

*Foreign Office Annual Series, No.* 2149.

The camphor trade has, so far as concerns foreign merchants in South Formosa, almost entirely stopped, owing, among other causes, to the disturbed state of the country and the difficulty and danger of sending money into the camphor districts. The roads continued throughout the year to be infested with armed robbers, who on the approach of the military or police fled to the hills (where it was, apparently, impossible to pursue them) only to reappear at the first favourable opportunity. Robberies became of such frequent occurrence that no foreign or native merchant would venture to send money into the interior. The Japanese authorities on their part did not see their way to allow the tax to be paid in the treaty port on arrival of the camphor, and business was consequently brought to a standstill.

In the raids and skirmishes, too, which have taken place in the camphor-producing districts numbers of stills have been destroyed. Their destruction was perhaps inevitable, but as they were almost entirely erected with money advanced or loaned by foreign merchants in South Formosa, the losses incurred by the latter have been very considerable. It is estimated that not one-third of the stills in existence two years ago, in which foreigners in South Formosa are interested, are now available for camphor production.

The hope expressed by Her Majesty's Consul in last year's report on the trade of this district, that the camphor trade might revive and assume large proportions has not been realised; in fact, far from this being the case, the camphor export business, as far as South Formosa is concerned, has now (April, 1898) almost stopped.

These remarks, of course, apply exclusively to the export of camphor by foreign merchants in this district (South Formosa) who have in the past invested considerable sums of money in the business. The production of camphor in the districts of Rinkipo and Shu Shu (Hunlin and Chip Chip), the principal districts whence the drug came to South Formosa, still, I am informed, continues, though to nothing like the same extent as formerly; but all the camphor so produced finds its way, *viâ* the port of Rokko (Lokkang), to Tamsui, whence it is shipped to Hong Kong and Japan. The roads north of Rokko are said to be perfectly safe, so that dealers can reach the neighbourhood of Chip Chip, and buy up any camphor that, under other circumstances, should and would go to the foreign firms in Tainan, with whose money the business was first started. Things may remedy themselves in course of time, but the outlook at present is certainly not very bright.

The following table shows the export of camphor from this port since, practically, the commencement of the trade:—

| Year. | Number of Boxes Exported. |
|---|---|
| 1892 | 4,315 |
| 1893 | 6,691 |
| 1894 | 12,157 |
| 1895 | 10,145 |
| 1896 | 8,001 |
| 1897 | 3,057 |

Note.—One box contained about 1 picul (133⅓ lb.) of camphor.

### Camphor in North Formosa.

*Foreign Office Annual Series, No.* 2148.

With regard to camphor, at present no British firm in North Formosa seems to interest itself in this important product, the handling of which is in the hands of German and Chinese merchants, acting probably in combination. There is a slight decrease in the quantity exported, and a large decrease in value, from 194,221*l.* in 1896 to 121,938*l.* in 1897.

### Wood Pulp at Gothenburg.

*Foreign Office Annual Series, No.* 2161.

Sulphate, as well as sulphide, has occupied an excellent position all through 1897; demand very lively at satisfactory prices, and makers can consequently congratulate themselves at having experienced a good year. The best evidence of prosperity in an industry is, without doubt, the possibility of enlargement and extension, and this, as regards the wood-pulp factories, has been manifest generally. On the other hand, as is the case nowadays throughout the world, the establishment of new factories in the same line is likely, at no distant date, to occasion over-production and the return to an unremunerative market.

This observation principally refers to the sulphite works, of which there are many building in this country at present. Under such circumstances it is quite natural that prices should already have moderated in some degree. As regards prices over the year, the following should be looked upon as correct: for unbleached sulphite, 8*l.* 6*s.* 8*d.* to 8*l.* 17*s.* 9*d.* per ton; for unbleached sulphate, 7*l.* 10*s.* to 8*l.* 1*s.* 1*d.* per ton f.o.b. Swedish seaport, dependent, of course, also on quality and terms of delivery.

Mechanical wood-pulp has maintained its advantageous position from the autumn of 1896, and on the whole experienced a satisfactory market. There have been no new establishments of importance built or planned in this branch of the manufacture, and consequently no influence from such cause has been felt; there certainly is some sharp competition threatening from North America, chiefly Canada, whence already considerable export of mechanical wood-pulps have come into the market of Europe, but it has not yet been proved that Americans, except in isolated cases, can compete advantageously with European makers, although they certainly prevent prices from reaching any extraordinary figure as well as exclude the possibility of lowness in the European stocks to affect the market.

Prices during during the year have been: for dry, 3*l.* 8*s.* 11*d.* to 4*l.* 3*s.* 4*d.* per ton; for wet, 1*l.* 15*s.* 7*d.* to 2*l.* 2*s.* 3*d.* per ton f.o.b. Swedish seaport, likewise dependent upon quality and terms of delivery.

### Chemicals at Gothenburg.

*Foreign Office Annual Series, No.* 2161.

The requirements of alkali mainly depend on the description of glass that the bottle and glass works have to manufacture; if white glass is on the order list, alkali must be procured, otherwise soda-ash takes its place. England supplies the bulk of these articles to the Swedish works, which in their turn export large quantities of bottles to Great Britain. This year alkali has been predominant in the import of these two articles.

Each year has brought an increase in the import of fertilisers. Much comes from England, or, as in the case with phosphates, from the United States, is carried here in British ships.

Glauber salts is an article that is annually increasing among imports. The glass works as well as cellulose manufacturers are the most prominent buyers.

Germany principally supplies potash (caustic), which is now mostly imported in liquid form.

The guano and oil mills on this coast, 23 in number, have been unable to work to any extent worth mentioning, owing to the high price and scarcity of fish. Their production was, however, mostly exported to France and Great Britain.

There is a factory established lately for the purpose of manufacturing lubricating oils, by a patent process, from fish-oil, which is reported to be a complete success.

### Electrical Soda at Uddevalla (Sweden).

*Foreign Office Annual Series, No.* 2161.

It was mentioned in last year's report that a factory had been established for the purpose of producing caustic soda and bleaching powder. This undertaking, laid out on a very extensive scale, has, however, up to date but proved an experimental affair, for none of these products have appeared in the market, and nothing has been done beyond putting up new machinery. It is hoped that success will finally attend this electrical process; if so, it will be colossal, as the power and raw material are there in unlimited quantity.

### Phosphates in Algeria.

*Foreign Office Annual Series, No.* 2164.

The following numbers indicate the quantity of phosphates extracted in the region of Tebesa during the year 1896:—

| Proprietors. | Situation of Mines. | Quantity Extracted. |
|---|---|---|
| | | Tons. |
| Crookston | Djebel Dyr | 66,678 |
| Jacobsen | Djebel Kouif | 80,127 |
| Societe Francaise | Ain Kissa | 18,000 |
| Total | | 164,805 |

The number of workmen employed in these mines is:—

| At | Number. | | |
|---|---|---|---|
| | Interior. | Exterior. | Side Works. |
| Djebel Dyr | 150 | 60 | .. |
| Djebel Kouif | 101 | 98 | 130 |
| Societe Francaise | 120 | .. | 30 |

Phosphates have lately been discovered at three places: (1) Tocqueville; (2) Bordj Redir (near Setif), and (3) Rio-Salado. At Tocqueville 212 tons have been extracted, transported, and shipped at Bougie. At Rio-Salado 738 tons were produced during five months with only 100 workmen. The quantity sold amounted to 520 tons, the lowest price realising 40 frs. per ton.

During the past year the total amount of phosphates shipped was 199,104 tons, showing an increase of 16,226 tons on the preceding year (from bone).

A mine of calamine recently discovered near Tébassa is now being worked by a Belgian company.

1,700 tons of zinc and 2,000 tons of lead were shipped for Belgium (from bone).

A French firm proposed to erect in the neighbourhood of Philippeville, on the sea shore, a manufactory for sulphuric acid, but for divers reasons it appears that the project has been abandoned, and that Bôna has been chosen instead of Philippeville. If this is true it is greatly to be deplored, as the manufactory would have given work to many, and the iron-pyrites mines in this neighbourhood, and at Ain Sedma, near Collo, would have found an outlet for their mineral, which, from its low grade, cannot stand high rates of carriage.

### Paper Exports from Yokohama.

*Foreign Office Annual Series, No.* 2165.

The export of paper in 1897 shows an increase of nearly 12,000*l.* over that of the previous year. In former reports all descriptions of Japanese paper have been included under one general heading in the tables, but it has been considered desirable to give the export more in detail in the present report. The Japanese paper known as gampishi is finding great favour, both in Europe and America, as copying paper, it being more durable and capable of receiving a larger number of impressions than the home-manufactured article. It is made from the fibres of a climber called gampi, botanically known as Wikstrœmia gampi, which grows wild in many of the hilly districts of Japan. The export of Japanese wall-paper also shows a considerable increase over last year.

### Mineral Products of Angora (Turkey).

*Foreign Office Annual Series, No.* 2166.

Some 687,000 lb. of saltpetre, of a value of some 11,000*l.*, are annually supplied to the Government factory which has been established at Konieh. It is there subjected to a cleansing process, and the residue, about 650,000 lb., is sent to Constantinople to the Government powder magazines.

Of all the mines in the province, only some four which produce chrome are being worked by an English company established at Smyrna. There is also an argentiferous lead mine near Bulgar-Maden, work at which is being carried on by Government. These and similar sources of wealth would be opened up at once if the Anatolian Railway were worked on really commercial principles.

### Sulphate of Copper in Algeria.

*Foreign Office Annual Series, No.* 2164.

Sulphate of copper is used in large quantities in the composition of "Bouillie Bordelaise" and for sprinkling on the vines. The price is from 22 to 24 fr. per 100 kilos. according to purity, and the duty from 2 fr. 75 c. to 3 fr., also according to purity. About 1,000 tons are annually imported into this colony, of which some 478 tons arrive *viâ* Marseilles from Great Britain. The English sulphate of copper is preferred in the market, it being slightly cheaper and also purer.

### Esparto at Oran (Algeria).

*Foreign Office Annual Series, No.* 2164.

The exportation of esparto grass is as usual about 10,000 tons per annum, and it now seems to have touched its lowest price. As usual it is shipped almost exclusively to the United Kingdom. There is no doubt but that the great misery existing amongst the Arabs through the failure of their crops for the last three years is almost the only reason by which it is possible to explain the continuation of the production at such low prices, and this year, although crops promise exceedingly well, there will still be great misery amongst the natives, but few having been able to till, having in most cases been obliged to use for food the grain given them by the Government for tilling purposes. Esparto production is not, therefore, likely to fall off for at least another year, notwithstanding its low prices.

### Alfa Pulp in Algeria.

*Foreign Office Annual Series, No.* 2164.

There has recently been some correspondence in a consular journal as to the feasibility, from a commercial point of view, of making " pâte d'alfa," or alfa pulp in this country. This question was gone fully into some 16 or 18 years ago, when esparto grass was three times its present value, and the cost of transport some six times greater than now. It was then found to be impracticable. Algeria is not, and cannot for some time become an industrial country. There was also another reason, and apparently an unsurmountable one, *viz.*, that British paper makers, who are almost alone in the employment of esparto grass, have their own plant for the fabrication from esparto grass itself; their motive power would cost them just as much, and they do not care to purchase any article partially made up if they can avoid it, preferring to make their own pulp, and thus be certain of the quality of the materials they employ.

There is, however, no reason why a paper manufactory, for the supply of local requirements, should not be established, as Algeria consumes some 6,000 tons of paper of different kinds annually.

## OFFICIAL REPORTS.

### Correspondence relating to the Conference at Brussels on the Question of Sugar Bounties—Commercial No. 6 (1898). (C. 8938.) Price 11½*d*.

*Board of Trade J., Aug.*, 232.

The present paper contains the appointment, dated 31st May 1898, of Sir F. Plunkett, Sir H. Bergne, Lord Ampthill, and Mr. Ozanne to act as British delegates at the International Conference on the question of Sugar Bounties at Brussels. It adds that Mr. Nevile Lubbock and Mr. G. Martineau will attend at Brussels in the capacity of experts, and gives general instructions to the delegates for their guidance.

It also gives the report of the delegates, in which they offer the following general observations upon the present position of the question from the international point of view:—

Austria-Hungary, Germany, Belgium, and Holland desire to effect a complete abolition of the bounties, and no opposition to an arrangement to this effect is to be apprehended from Spain and Sweden. France, however, whilst willing to abolish the direct bounty on export under her Law of 1897, wishes to retain the advantage of the indirect export bounty created by her internal Law of 1884; and Russia declines even to discuss whether her existing system amounts to a bounty on export or not.

Germany grants only a direct export bounty, which is, roughly speaking, about equal in amount to the direct export bounty granted under the French Law of 1897, and although sugar can be produced cheaper in Germany than in France, it is not to be expected that Germany will consent to abolish the whole of her bounty whilst France retains that created by her Law of 1884, which is about three times as much as the direct export bounty granted under her Law of 1897. Austria-Hungary, on the other hand, contends that the Russian system does in fact amount to the grant of a bounty on exportation, and as Russia is her chief competitor in the sugar markets of Italy and the Levant, the Austro-Hungarian Government are not prepared to abolish their bounties unless some modification can be obtained in the Russian system.

It seems clear that in these circumstances there are at present but two methods of securing the suppression of the bounty system:—

1. By coming to some arrangement for such modifications or limitations in the French and Russian systems as may be acceptable to the other sugar-producing States, in return for the suppression of their bounties; and

2. By the conclusion of a Convention between a certain number of the sugar-producing States providing for the total suppression of sugar bounties within their dominions, and engaging that they will either impose countervailing duties on, or prohibit the entry of bounty-fed sugar coming from States which cannot be induced to become parties to the Convention.

The market of the United States is already rendered unprofitable by this means to all bounty-fed sugar. All the continental sugar-producing States, by means of customs duties and internal legislation, reserve the entire supply of the home market to the home producer of sugar; and the English, and to a rapidly increasing extent the Indian market thus becomes essential for the surplus sugar production of the European countries. Any steps by which these markets might be closed to bounty-fed sugar must therefore have a decisive effect in securing the speedy abolition of the bounty system.

That system is, however, now felt to press heavily on the economic resources of those States which have recourse to it, and it is not impossible that a further exchange of views may lead to some concessions by France and Russia which would form the basis of a general arrangement acceptable to all the sugar-producing States, as at least a mitigation of the unsatisfactory system which at present prevails. If no solution can be obtained, it is possible that a still worse state of affairs may result by the increase of bounties in various countries.

In the appendix are given the *procès-verbaux* of the various sittings held by the conference; summary and statistics of sugar legislation in different countries for the years 1883–97; correspondence exchanged between the Belgian Government and various countries respecting the programme of the conference, and notes on the legislation affecting the refinement of sugar in various countries.

## GENERAL TRADE NOTES.

### Manganese from Bog Ore.

*Eng. and Mining J., July* 23, 1898, 94.

Bog iron ore is worked in the province of Quebec, Canada, and arrangements are now being made to extract manganese from bog ore deposits in the province of New Brunswick by the Mineral Products Company. The ore is a soft, wet stuff, containing 50 per cent. of water, and is covered by a thin coating of vegetable earth. The depth of ore varies from 5 ft. to 30 ft. When dried the residuum is a fine black powder, too fine to be treated in the blast furnace, and this has therefore to be made into briquettes. The cementing material used is kept secret. The ore is brought in small tram cars into the building and shoveled into the feed hopper of a drier, which is an inclined iron cylinder 5 ft. diameter and 28 ft. long, revolving slowly in a brick chamber or furnace 44 ft. long, 10 ft. wide, and 30 ft. high. The dried ore falls from the end of the cylinder into the trough of a spiral conveyer, and is carried to a vertical bucket conveyor, which hoists it to the top of the building and feeds it into a revolving screen. The coarser material passes out at the end of the screen and goes to a pulverizer, from which it is led back to the vertical conveyor. The very fine material which passes off as dust is caught by a dust chamber and also carried to the vertical conveyor.

The screened ore dust is stored in bins and fed to a mixing machine, where it is mixed with some binder, which forms it into a plastic material, which is taken to the briquettes pressing machine, from which it is delivered in the form of cylindrical briquettes, 3 ins. diameter, and 2½ ins. long. These are taken to the company's blast furnace in Nova Scotia, where they are converted into ferro-manganese. An analysis of the dried ore at 212° F. is as follows: Metallic manganese, 48·210 per cent.; metallic iron, 5·700 per cent.; sulphur, 0.096 per cent.; phosphorus, trace; silica, 1·88 per cent.

### THE SUGAR INDUSTRY OF THE UNITED KINGDOM.

*The Times, August 8th, 1898. From the Bulletin of the French Ministry of Agriculture, 1898, 1—130.*

The whole of this bulletin is devoted to the recent international conference "Sur le Régime des Sucres." It includes a verbatim report of the proceedings at the sittings held in Brussels between June 7th and June 25th, and an appendix, in which are enumerated the legislative measures relating to the sugar industry in the different European countries, as well as details of the sugar trade of the United Kingdom. The quantities are enumerated of the raw cane sugar which has reached this country during the last 15 years from British possessions, including the West Indies, India, Ceylon, and the Straits Settlements, and Mauritius; also from foreign countries, including Brazil, Central America, the West and East Indies. A table is given showing the quantities of raw beet sugar which has been imported by this country from France, Germany, Belgium, the Netherlands, and other countries. The following is a summary from two tables of the quantities of refined cane and beet sugar:—

*Annual Imports of Sugar into the United Kingdom, 1883 to 1897.*

| Year. | Raw Sugar. | | Refined Sugar. | | Total. |
|---|---|---|---|---|---|
| | Cane. | Beet. | Cane. | Beet. | Cane and Beet. |
| | Cwt. | Cwt. | Cwt. | Cwt. | Cwt. |
| 1883...... | 11,949,337 | 8,417,299 | 139,793 | 3,144,127 | 23,650,556 |
| 1884...... | 11,640,201 | 7,082,475 | 1,066,187 | 3,197,686 | 23,886,552 |
| 1885...... | 11,226,918 | 8,189,611 | 2,282,239 | 3,046,807 | 24,745,605 |
| 1886...... | 9,339,523 | 6,774,138 | 1,125,613 | 4,946,437 | 22,505,711 |
| 1887...... | 8,786,510 | 9,223,856 | 775,859 | 6,220,453 | 25,006,678 |
| 1888...... | 11,483,052 | 6,371,117 | 11,680 | 6,830,271 | 24,729,423 |
| 1889...... | 9,403,194 | 8,146,243 | 17,531 | 8,960,729 | 26,528,407 |
| 1890...... | 5,649,610 | 10,067,868 | 293,285 | 9,683,380 | 25,694,141 |
| 1891...... | 6,275,190 | 9,226,958 | 537,969 | 10,791,152 | 27,531,579 |
| 1892...... | 7,709,051 | 8,586,596 | 39,658 | 10,581,013 | 26,916,318 |
| 1893...... | 7,354,890 | 8,677,214 | 19,049 | 11,501,491 | 27,582,653 |
| 1894...... | 6,489,435 | 7,816,769 | 19,712 | 13,925,080 | 28,250,796 |
| 1895...... | 7,760,237 | 9,219,760 | 21,415 | 14,123,728 | 31,155,140 |
| 1896...... | 7,617,366 | 8,126,310 | 11,659 | 14,765,270 | 30,520,605 |
| 1897...... | .. | .. | .. | .. | 29,384,319 |

It will be observed that over the period embraced by the above statement the imports of sugar of all kinds have expanded to the extent of 25 per cent. The great decline in the quantity of raw cane sugar imported is very noteworthy. Still more remarkable is the large increase—fully four fold—in the quantity of refined beet sugar which we annually import, and which comes exclusively from foreign countries that reap all the benefit involved in the demand for labour in the work of refining. In 1883 we imported 20,366,636 cwt. of raw sugar and only 3,283,920 cwt. of refined, whereas in 1896 the quantites were approximately equal. Then, again, in 1883 our total imports comprised 12,089,130 cwt. of cane sugar and 11,561,426 cwt. of beet sugar; but in 1896 they were 7,629,025 cwt. of cano sugar and 22,891,580 cwt. of beet sugar, showing an increase of three times as much beet- as cane-sugar.

The United Kingdom is entirely dependent for its sugar supply upon extraneous sources. It is, however, pointed out that an appreciable fraction of the quantity which we import is eventually re-exported to Sweden and Norway, Denmark, Holland, Belgium, Portugal, Italy, &c., these countries obtaining a portion of their supply in this manner.

The following table is combined from two tables in the original bulletin:—

*Annual Quantities of Raw Sugar Retained, and of Refined Sugar Imported and Retained, for Consumption in the United Kingdom, with the Annual Average Prices, 1883 to 1897.*

| — | Raw Sugar. | | | | Refined Sugar. | | | |
|---|---|---|---|---|---|---|---|---|
| | Total Quantity. | Per Head of Population. | Price per Quintal. | | Total Quantity. | Per Head of Population. | Price per Quintal. | |
| | Cwt. | Lb. | s. | d. | Cwt. | Lb. | s. | d. |
| 1883 | 18,516,116 | 58·5 | 20 | 1 | 3,138,088 | 9·9 | 27 | 2 |
| 1884 | 17,841,380 | 55·9 | 15 | 6 | 4,039,778 | 12·7 | 20 | 11 |
| 1885 | 17,991,974 | 53·9 | 13 | 10 | 5,109,507 | 15·9 | 18 | 2 |
| 1886 | 14,621,114 | 45·1 | 13 | 0 | 6,145,932 | 19·0 | 16 | 8 |
| 1887 | 16,823,490 | 51·5 | 12 | 1 | 6,704,832 | 20·5 | 15 | 8 |
| 1888 | 16,042,933 | 48·7 | 13 | 5 | 6,684,414 | 20·3 | 17 | 6 |
| 1889 | 16,058,761 | 48·4 | 15 | 5 | 8,809,669 | 26·5 | 19 | 8 |
| 1890 | 14,317,889 | 42·9 | 12 | 6 | 9,444,789 | 28·2 | 16 | 4 |
| 1891 | 15,213,249 | 45·1 | 12 | 10 | 11,115,151 | 32·9 | 16 | 6 |
| 1892 | 15,166,205 | 44·6 | 13 | 0 | 10,419,955 | 30·6 | 17 | 1 |
| 1893 | 14,556,836 | 42·4 | 11 | 2 | 11,385,544 | 33·2 | 18 | 4 |
| 1894 | 12,874,665 | 37·2 | 11 | 5 | 13,813,492 | 39·9 | 15 | 6 |
| 1895 | 15,813,354 | 45·3 | 9 | 7 | 14,003,191 | 40·1 | 13 | 4 |
| 1896 | 14,125,250 | 40·9 | 10 | 5 | 11,634,792 | 41·5 | 13 | 7 |
| 1897 | 12,274,121 | 34·5 | 9 | 0 | 15,615,680 | 43·9 | 12 | 3 |

Finally, in the table shown below, is the total consumption of sugar in the United Kingdom for each of the years under notice, the aggregates being in effect those of raw and refined sugar in the table just given. In other words the following figures represent imports less exports. Disregarding minor fluctuations, the table justifies the assertion (*loc. cit.*) that in the years 1883 to 1897 our annual consumption of sugar increased by fully 25 per cent.:—

*Annual Consumption of Sugar in the United Kingdom, 1883 to 1897.*

| Year. | Cwt. | Year. | Cwt. |
|---|---|---|---|
| 1883 | 21,654,204 | 1891 | 26,328,400 |
| 1884 | 21,881,158 | 1892 | 25,586,160 |
| 1885 | 23,101,481 | 1893 | 25,942,380 |
| 1886 | 20,727,346 | 1894 | 26,688,157 |
| 1887 | 23,528,322 | 1895 | 29,817,548 |
| 1888 | 22,727,347 | 1896 | 25,760,042 |
| 1889 | 24,868,430 | 1897 | 27,889,801 |
| 1890 | 23,762,678 | | |

—J. L. B.

## BOARD OF TRADE RETURNS.

### SUMMARY OF IMPORTS.

| Articles. | Month ending 31st July. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals.............................. | 1,983,090 | 1,847,311 |
| Chemicals and dyestuffs ........... | 430,792 | 418,269 |
| Oils................................. | 759,037 | 657,845 |
| Raw materials for non-textile industries. | 5,985,226 | 5,323,559 |
| Total value of all Imports .... | 36,123,523 | 35,903,039 |

### SUMMARY OF EXPORTS.

| Articles. | Month ending 31st July. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) .... | 3,023,282 | 2,719,593 |
| Chemicals and medicines........... | 629,405 | 637,031 |
| Miscellaneous articles............... | 2,887,761 | 2,756,039 |
| Total value of all exports..... | 21,501,452 | 20,089,878 |

### IMPORTS OF METALS FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Copper:— | | | | |
| Ore ............ Tons | 12,209 | 13,232 | 41,728 | 39,812 |
| Regulus ........ „ | 7,669 | 4,897 | 213,808 | 123,480 |
| Unwrought ..... „ | 7,939 | 6,311 | 390,557 | 320,003 |
| Iron:— | | | | |
| Pig and puddled „ | 11,224 | 16,460 | 37,107 | 56,495 |
| Ore............. „ | 576,337 | 522,416 | 432,879 | 391,088 |
| Bolt, bar, &c. ... „ | 6,878 | 5,315 | 56,175 | 42,649 |
| Steel, unwrought.. „ | 2,589 | 3,433 | 14,896 | 19,344 |
| Lead, pig and sheet „ | 13,346 | 18,544 | 155,702 | 244,194 |
| Pyrites ........... „ | 51,829 | 64,677 | 83,799 | 111,186 |
| Quicksilver ....... Lb. | 59,125 | 17,700 | 5,551 | 1,769 |
| Silver ore....... Value £ | .. | .. | 130,202 | 96,170 |
| Tin ............... Cwt. | 26,406 | 28,829 | 80,185 | 96,431 |
| Zinc .............. Tons | 5,854 | 6,497 | 93,455 | 113,923 |
| Other articles ...Value £ | .. | .. | 247,556 | 190,767 |
| Total valne of metals | .. | .. | 1,983,600 | 1,847,311 |

### IMPORTS OF OILS FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut......... Cwt. | 12,892 | 9,217 | 13,211 | 10,505 |
| Olive.............. Tuns | 1,245 | 2,556 | 42,366 | 81,749 |
| Palm ............. Cwt. | 95,477 | 105,357 | 95,446 | 110,098 |
| Petroleum ........ Gall. | 21,096,814 | 12,538,203 | 343,389 | 242,923 |
| Seed .............. Tons | 2,998 | 1,813 | 51,647 | 35 366 |
| Train, &c. ........ Tuns | 2,255 | 2,271 | 38,083 | 38 377 |
| Turpentine ....... Cwt. | 101,402 | 42,463 | 99 632 | 43,930 |
| Other articles .. Value £ | .. | .. | 75,263 | 94,897 |
| Total value of oils... | .. | .. | 759,037 | 657,845 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,710 | 1,429 | 3,322 | 3,079 |
| Bristles.......... Lb. | 362,732 | 368,571 | 43,389 | 46,722 |
| Caoutchouc ...... Cwt. | 23,280 | 35,515 | 273,561 | 421,521 |
| Gum:— | | | | |
| Arabic......... „ | 6,261 | 2,648 | 10,872 | 6,705 |
| Lac, &c. ....... „ | 14,918 | 7,357 | 58,273 | 28,819 |
| Gutta-percha .... „ | 2,411 | 5,844 | 22,512 | 57,736 |
| Hides, raw:— | | | | |
| Dry............ „ | 46,071 | 35,383 | 116,846 | 100,615 |
| Wet ........... „ | 66,159 | 50,341 | 146,569 | 107,313 |
| Ivory ............ „ | 1,308 | 1,059 | 49,252 | 46,312 |
| Manure:— | | | | |
| Guano ......... Tons | 421 | 1,295 | 2,339 | 5,194 |
| Bones ......... „ | 4,069 | 1,760 | 13,122 | 7,421 |
| Nitrate of soda... „ | 3,249 | 8,842 | 24,083 | 68,585 |
| Phosphate of lime „ | 21,411 | 21,263 | 30,545 | 30,912 |
| Paraffin.......... Cwt. | 47,105 | 58,803 | 39,470 | 47,145 |
| Linen rags....... Tons | 1,879 | 1,933 | 18,316 | 16,958 |
| Esparto.......... „ | 15,121 | 7,693 | 60,707 | 29,593 |
| Pulp of wood:— | | | | |
| Chemical ...... „ | 28,510 | 13,025 | 149,390 | 89,684 |
| Mechanical .... „ | | 23,405 | | 60,588 |
| Rosin........... Cwt. | 140,504 | 194,623 | 35,551 | 43,289 |
| Tallow and stearin „ | 264,983 | 220,524 | 249,060 | 228,447 |
| Tar ............Barrels | 13,867 | 12,514 | 8,709 | 8,050 |
| Wood:— | | | | |
| Hewn ......... Loads | 358,344 | 242,634 | 762,973 | 584,304 |
| Sawn .......... „ | 1,197,110 | 906,183 | 2,862,409 | 2,144,094 |
| Staves......... „ | 14,615 | 20,909 | 45,817 | 67,148 |
| Mahogany ..... Tons | 7,297 | 2,862 | 62,786 | 26,036 |
| Other articles....Value £ | .. | .. | 895,353 | 1,047,289 |
| Total value ....... | .. | .. | 5,985,226 | 5,323,559 |

Besides the above, drugs to the value of 67,221*l*. were imported, as against 88,509*l*. in July 1897.

### IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............. Cwt. | 18,658 | 23,268 | 8,470 | 12,298 |
| Bark (tanners', &c.) „ | 52,455 | 38,499 | 21,837 | 20,702 |
| Borax ............ „ | .. | 4,645 | .. | 2,126 |
| Brimstone ........ „ | 35,880 | 24,340 | 7,897 | 6,158 |
| Chemicals...... Value £ | .. | .. | 111,237 | 124,942 |
| Cochineal ......... Cwt. | 482 | 433 | 2,991 | 2,599 |
| Cutch and gambier Tons | 2,800 | 1,771 | 42,847 | 31,375 |
| Dyes:— | | | | |
| Alizarin ....... Value £ | .. | .. | 14,653 | 15,671 |
| Anilin and other „ | .. | .. | 37,491 | 49,550 |
| Indigo ........... Cwt. | 1,684 | 1,081 | 25,013 | 18,833 |
| Nitrate of potash . „ | 25,950 | 19,755 | 19,149 | 14,123 |
| Valonia........... Tons | 3,580 | 3,088 | 36,006 | 32,555 |
| Other articles... Value £ | .. | .. | 103,151 | 87,337 |
| Total value of chemicals | .. | .. | 430,792 | 418,269 |

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 8,732 | 7,213 | 38,522 | 34,018 |
| Copper:— | | | | |
| Unwrought.... „ | 33,487 | 52,640 | 87,063 | 139,121 |
| Wrought ...... „ | 24,533 | 19,133 | 73,565 | 60,312 |
| Mixed metal... „ | 18,541 | 13,552 | 43,898 | 33,848 |
| Hardware and cutlery. Value £ | .. | .. | 173,918 | 160,892 |
| Implements ..... „ | .. | .. | 124,268 | 111,485 |
| Iron and steel ... Tons | 331,553 | 281,194 | 2,136,401 | 1,955,027 |
| Lead ............ „ | 4,735 | 2,732 | 60,791 | 40,608 |
| Plated wares ... Value £ | .. | .. | 34,266 | 33,853 |
| Telegraph wires . „ | .. | .. | 136,547 | 45,600 |
| Tin ............. Cwt. | 7,725 | 7,447 | 25,561 | 26,679 |
| Zinc ............ „ | 12,835 | 21,674 | 10,446 | 19,720 |
| Other articles .. Value £ | .. | .. | 78,036 | 88,435 |
| Total value ....... | .. | .. | 3,023,282 | 2,749,593 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 953,700 | 1,082,200 | 20,061 | 23,668 |
| Military stores.. Value £ | .. | .. | 136,461 | 179,140 |
| Candles.......... Lb. | 2,022,600 | 2,204,100 | 27,177 | 29,218 |
| Caoutchouc ..... Value £ | .. | .. | 99,153 | 106,574 |
| Cement.......... Tons | 38,039 | 30,053 | 62,387 | 56,270 |
| Products of coal Value £ | .. | .. | 121,458 | 111,927 |
| Earthenware ... „ | .. | .. | 150,761 | 144,459 |
| Stoneware ...... „ | .. | .. | 13,392 | 14,749 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 140,196 | 124,903 | 7,360 | 7,830 |
| Flint........... Cwt. | 9,285 | 7,287 | 20,892 | 16,625 |
| Bottles......... „ | 69,676 | 58,587 | 33,067 | 27,836 |
| Other kinds.... „ | 18,322 | 18,363 | 14,433 | 16,586 |
| Leather:— | | | | |
| Unwrought .... „ | 13,350 | 11,671 | 123,292 | 107,720 |
| Wrought ..... Value £ | .. | .. | 35,043 | 27,444 |
| Seed oil.......... Tons | 3,688 | 3,064 | 64,017 | 53,762 |
| Floorcloth ..... Sq. Yds. | 2,127,300 | 1,699,900 | 90,110 | 75,490 |
| Painters' materials Val. £ | .. | .. | 148,985 | 132,953 |
| Paper ........... Cwt. | 87,712 | 76,885 | 129,548 | 119,252 |
| Rags............. Tons | 5,538 | 5,224 | 28,238 | 26,197 |
| Soap ............. Cwt. | 57,338 | 71,116 | 56,239 | 75,988 |
| Total value .......... | .. | .. | 2,887,761 | 2,756,039 |

EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST JULY.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 71,391 | 282,400 | 104,509 | 75,100 |
| Bleaching materials „ | 81,468 | 91,246 | 27,013 | 25,335 |
| Sulphate of copper Tons | 1,684 | 1,745 | 27,065 | 27,677 |
| Chemical manures „ | 41,517 | 48,758 | 158,874 | 195,396 |
| Medicines....... Value £ | .. | .. | 91,177 | 84,886 |
| Other articles... „ | .. | .. | 220,767 | 228,628 |
| Total value.......... | .. | .. | 629,405 | 637,031 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

16,100. A. J. Boult.—From H. J. Peron, Belgium. Improvements in or relating to liquid fuel furnaces. Complete Specification. July 23.

16,220. O. Guttmann. *See* Class VII.

16,594. H. Lyon and J. B. Talbot-Crosbie. Improvements in refrigerating apparatus. July 30.

16,672. A. Lecomte and I. Loeser. Improvements in apparatus for regulating the pressure of and distributing gas. Complete Specification. Filed July 30. Date applied for 11 July 1898, being date of application in Belgium.

16,769. Brewster, Smith, and Co. Improved test tube stand. Aug. 3.

16,808. J. Week. Improvements in air-tight provision receptacles suitable for use with sterilising apparatus. Complete Specification. Aug. 3.

16,957. C. S. Cohen. Improved apparatus for cooling purposes. Aug. 5.

17,042. A. C. Harris. Improvements in machines for making tablets of certain chemical powders by compression. Aug. 8.

17,084. C. Maquet. Improvements in heating apparatus. Aug. 8.

17,100. F. C. Hart. Improved apparatus for testing anthracene, viz., measuring and adding acetic acid, adding chromic acid solution, and a filter pump for same. Aug. 9.

17,101. F. C. Hart. An improved form of apparatus for the testing of ammoniacal gas liquor for free and fixed ammonia, and also density of same. Aug. 9.

17,201. G. Zschocke. Improvements in apparatus for precipitating the solid particles of the top-gases of a blast furnace from out of the clear water. Aug. 10.

17,216. F. E. V. Baines. Improvements in and apparatus for the evaporation of solutions of solid matter. Aug. 10.

17,259. M. Otto. A titrating and mixing apparatus, especially applicable to the distribution and regulation of mixtures of ozone and air. Complete Specification. Aug. 10.

17,296. J. Metzger. Improvements in and relating to filtering presses. Complete Specification. Aug. 10.

17,110. W. Eckardt. Improvements in kilns. Complete Specification. Aug. 12.

17,523. M. Otto. Improvements in meter for apparatus for measuring and registering liquids and gases. Complete Specification. Aug. 13.

17,524. M. Otto. An improved colorimeter, more especially applicable for use as an ozonometer. Complete Specification. Aug. 13.

### COMPLETE SPECIFICATIONS ACCEPTED.*

1897.

15,402. E. Sterné. *See* Class II. Aug. 3.

16,787. W. Lockwood. Method of generating steam and other vapours. July 27.

19,344. T. Swift and G. H. Swift. A liquid composition for the removal and prevention of scale from corrosion on inside of boilers. Aug. 10.

22,026. A. W. Stokes and R. A. Lister and Co., Ltd. Apparatus for automatically measuring off quantities of liquids, specially applicable for testing the amount of fat in milk. Aug. 3.

22,027. A. W. Stokes and R. A. Lister and Co., Ltd. Apparatus for testing the acidity of milk and other liquids. Aug. 17.

22,141. P. F. C. Willcox, H. W. Willcox, and R. J. N. Willcox. Apparatus for heating or evaporating liquids by means of steam. Aug. 17.

22,965. D. Cameron, F. J. Commin, and A. J. Martin. Apparatus for the treatment of sewage or other liquids. Aug. 10.

24,014. P. E. M. Jamain. Apparatus for compressing powdered substances. Aug. 3.

1898.

1087. H. Beyer and F. Reeke. Economical kiln. Aug. 10.

9005. H. Rockling. Apparatus for compressing coal or similar material. Aug. 3.

9605. I. Fokchaner. Filters for water and other liquids. Aug. 10.

10,100. A. J. Boult.—From The Nitrogen Processing Company, United States. Drying and pulverising apparatus. July 27.

11,826. C. Butters. Manufacture of discharge doors for vats. Aug. 3.

13,619. S. S. Bromhead.—From Möller and Pfeifer, Germany. Cell cylinders for drying apparatus. Aug. 10.

14,718. J. B. Bomseau. Metallic capsules for storing liquids or gases under pressure. Aug. 10.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

15,713. J. H. H. Duncan. Improvements in the manufacture of incandescence bodies suitable for use with gas and other burners. July 18.

15,731. M. Shettel. Improvements in the manufacture of mantles or equivalent means of lighting by incandescence. July 18.

15,818. A. J. Boult.—From E. Salzenberg, Germany. Improvements in or relating to incandescent gas lighting. July 19.

15,957. G. Kohl. Improvements in the manufacture of glow-bodies for incandescence burners. July 21.

15,958. G. Kohl. Improvements in the manufacture of glow bodies of incandescence burners. July 21.

16,009. J. Fraser. Process of manufacturing gas for illuminating and other purposes. July 22.

16,026. H. Hawkins. A new or improved apparatus for generating acetylene gas. July 22.

16,090. H. Dant. Improvements in acetylene gas generators. Complete Specification. July 23.

16,234. R. J. Moss. An improvement connected with the generation of acetylene. July 26.

16,266. A. Engelhardt. Improvements in or relating to the manufacture of gas. July 26.

16,392. S. Biheller. Improved method of producing gas from calcium carbide, and in the means or apparatus therefor. July 27.

16,457. H. R. Berger. Improved apparatus for generating acetylene gas in a pure state. July 28.

16,559. R. Williams. Improvements relating to the generation of acetylene. July 29.

16,613. C. Testelin and C. Roseaux. Improvements in apparatus for generating and burning acetylene gas. July 30.

16,631. C. J. Yarnold. Improvements in or relating to the manufacture of hydrogen peroxide and its application to bleaching purposes. July 30.

16,634. C. V. G. Schmidt. New or improved means and apparatus for the generation and regulation of acetylene gas in lamps. July 30.

16,670. H. H. Böhndel and C. C. Yeaton. An improved process for the production of gas for illuminating and other purposes. July 30.

16,712. W. L. Wise.—From J. H. Moss, United States. Improvements in process and apparatus for generating and burning gas from hydrocarbon oils. Complete Specification. Aug. 2.

16,884. J. Varon. An improved apparatus for the production of acetylene gas. Complete Specification. Aug. 4.

16,894. C. Vautrin and A. Brocard. Improvements in apparatus for producing acetylene gas. Aug. 4.

16,903. M. L. J. R. L. de Montais. Improvements in acetylene gas generators. Aug. 4.

17,007. J. F. Duke. Improvements in incandescent gas lighting. Aug. 6.

17,008. R. Holliday and Sons, Ltd., and R. Holliday. Improvements in or connected with apparatus for generating acetylene gas. Aug. 6.

17,079. F. Gehlert. Improvements in or relating to apparatus for producing acetylene gas. Complete Specification. Aug. 8.

17,101. F. C. Hart. *See* Class I.

17,127. J. W. Bailey and J. Clapham. Improvements in apparatus for use in generating acetylene gas. Aug. 9.

17,199. F. P. Berange. Improvements in or relating to acetylene gas generators. Aug. 9.

17,230. W. Smith. An improved water jacket for acetylene gas generators. Aug. 10.

17,260. M. Otto. Improvements in apparatus for producing ozone. Complete Specification. Aug. 10.

17,414. F. Priestley. Improvements in apparatus applicable to steam boiler and other furnaces for consuming smoke and economising fuel. Aug. 12.

17,427. C. Davies, H. Arrowsmith, and S. W. Dod. A new or improved manufacture of fuel, applicable also as fire-lighters. Aug. 12.

17,504. G. W. Emmerson. Improvements in apparatus for generating acetylene gas. Aug. 13.

17,516. J. Fielding. Improvements in apparatus for producing and carburetting hydrogen. Complete Specification. Aug. 13.

17,518. T. G. Allen, jun. Improvements in apparatus for generating and storing acetylene gas. Aug. 13.

### Complete Specifications Accepted.

1897.

15,402. E. Sterné. Capsules or containers for containing compressed or liquefied gases. Aug. 3.

17,090. E. Barnard. Apparatus for the generation of acetylene gas or other gases. July 27.

19,590. F. M. Mooney. Treatment of oxide of iron used for the absorption of sulphuretted hydrogen in the purification of illuminating or other gases or otherwise. Aug. 10.

19,887. L. G. Harris. Manufacture of gas from garbage and other refuse. July 27.

21,114. H. R. Bean and H. Ringwood. Apparatus for the generation of acetylene and other gases. Aug. 3.

21,115. T. C. Palmer, of the firm of W. J. Fraser and Co. *See* Class XII.

21,508. R. P. Pictet. *See* Class VII.

22,918. C. J. Bailey and J. H. Nicklin. Apparatus for the generation of acetylene gas. Aug. 17.

27,065. J. Benjamin. Gasometer for generating acetylene gas. Aug. 3.

29,304. M. H. Hawes, R. Farrington, and W. H. Hand. Gas and oil lighting. Aug. 10.

1898.

1625. W. Owens. Manufacture and storage of acetylene gas. Aug. 3.

2174. J. S. Goodwin. Acetylene generating apparatus. July 27.

2444. A. E. N. Yeadon and S. N. Yeadon. Apparatus for the purification of gas or other analogous purposes. Aug. 3.

6860. L. Stoecklin. Process for the manufacture of self-igniting mantles for glow-light. Aug. 17.

7592. J. Coyne. Manufacture of gas. Aug. 10.

8813. W. L. Wise.—From A. Piatti and Co., Italy. Manufacture of gas. Aug. 17.

10,008. J. Loeser and A. Lecomte. Gas burner for incandescent lighting. July 27.

11,261. F. A. Keiffer. Acetylene gas generators. Aug. 17.

13,135. G. Hilgenstock. Apparatus for opening and closing the doors of coke ovens. July 27.

13,387. C. D. Abel.—From P. Lipcke, Germany. Apparatus for generating acetylene. Aug. 3.

14,197. W. P. Thompson.—From H. Sez, France. Apparatus for producing acetylene. Aug. 17.

14,713. H. H. Lake.—From La Compagnie Continentale d'incandescence et de chauffage, Belgium. Apparatus for generating acetylene gas. Aug. 17.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Application.

16,045. P. Tack. Improvements in and apparatus for distilling tar or oil or the like, and for distilling and concentrating other liquid or semi-liquid matters. July 22.

## IV.—COLOURING MATTERS and DYES.

### Applications.

15,708. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of a new product suitable for use as a colouring matter. July 18.

15,709. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new colouring matter. July 18.

16,034. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new nitro derivatives of the anthracene series. July 22.

16,199. A. Goldberg, W. Siepermann, and H. Flemming. A process for the preparation of a yellow sulphur containing dye from sulpho cyanide salts. Complete Specification. July 25.

16,201. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for the production of indigo. July 25.

16,474. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new colouring matters of the acridine series. July 28.

16,475. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of acridine derivatives. July 28.

16,564. G. W. Johnson.—From Vereingte Chininfabriken Zimmer and Co., Gesellschaft mit Veschränkter Haftung, Germany. The manufacture and production of diquinone carbonic ether. July 29.

16,650. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of azo-colouring matters, and materials thereof. July 30.

17,155. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Manufacture and production of aromatic paroamido aldehydes and their derivatives of substitution. Aug. 9.

17,366. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Improved manufacture of acetyl compounds. Aug. 11.

### Complete Specifications Accepted.

### 1897.

15,498. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Manufacture of aldehydonaptholsulphonic, aldehydonaptholcartoxylic, and aldehydonaptholsulphocarboxylic acids and of new blue and green colouring matters or dyes therefrom. Aug. 3.

20,126. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Bruning, Germany. Manufacture of dyestuff directly dyeing cotton black. Aug. 3.

20,278. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Manufacture of new polyazo colouring matters or dyes. Aug. 17.

20,333. W. H. Claus, A. Rée, and L. Marchlewski. Production of new derivatives of alkyl-hydroxy-paraamidobenzenes. Aug. 10.

20,649. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of colouring matters derived from anthraquinone. Aug. 3.

21,451. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of blue to green dyestuffs soluble in water. Aug. 17.

22,242. O. Imray.—From The Farbwerke, Meister, Lucius, and Bruning, Germany. Manufacture of blue basic safranine azo-dyestuffs. Aug. 17.

23,017. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of chemical compounds of the aromatic series. Aug. 17.

24,527. C. D. Abel.—From The Actien Gesellschaft fur Anilin Fabrikation, Germany. Manufacture of black diazo colouring matters. Aug. 17.

24,793. J. C. Mewburn.—From The Chemische Fabrik von Heyden, Gesellschait mit beschrankter Haftung, Germany. Production of phenolic carbonates. Aug. 17.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

16,010. K. T. Sutherland. Improvements connected with the preparing or cleansing or bleaching of leaf and stalk fibres and materials made from them. July 22.

16,720. H. H. Lake.—From the firm of M. T. Stevens and Sons, United States. Improvements in apparatus for drying wool and other textile fabrics. Complete Specification. Aug. 2.

16,823. C. D. Abel.—From the firm of Joh. Kleinewefers, Söhne, Germany. Improvements in apparatus for mercerising cotton in the form of hanks. Aug. 3.

16,931. T. F. Wilson and P. Kestner. Improvements in the conditioning of cotton and like yarns, and apparatus for that purpose. Aug. 5.

17,010. W. C. Wood. Improvements in machinery for the "mercerising" of cotton and other textile fabrics. Aug. 8.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

16,390. B. J. B. Mills.—From Mercier and Chaumartin, France. An improved process for dyeing fabrics, yarns, and threads. July 27.

16,579. R. W. Kemp. A combined dyeing machine for dyeing cloth yarns (cotton, worsted, woollen, silk, and all kinds of yarn and slubbing). July 30.

16,893. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Bruning, Germany. Method for the production of fast colours on the wool fibre from picramic acid monazo dyestuffs. Aug. 4.

17,075. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Method for the production on the fibre of the azo colours soluble in water in tints varying from violet-black to dark-black tints. Aug. 8.

### Complete Specifications Accepted.

### 1897.

18,487. G. B. Ellis.—From J. Heilmann et Cie. Manufacture and production of coloured designs on woven fabrics. Aug. 10.

22,150. E. Cabiati. Process of bleaching fabrics. July 27.

### 1898.

9885. J. R. Hope, W. T. Galey, and T. McConnell. Machines for heating yarns for mercerising, dyeing, and the like. July 27.

12,183. W. Shaw. Method of and apparatus for colouring yarns. Aug. 3.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

15,719. C. A. Jensen.—From J. Maemecke, Germany. An improved process for the production of alkali compounds of albuminous substances. Complete Specification. July 18.

15,807. F. F. Myles. Bath salt. Complete Specification. July 19.

15,947. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of sulphuric anhydride and sulphuric acid, and apparatus to be employed therein. July 21.

15,948. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of sulphuric anhydride. July 21.

15,949. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in and apparatus for the manufacture of sulphuric anhydride. July 21.

15,950. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of sulphuric anhydride and apparatus for use therein. July 21.

16,220. O. Guttmann. Improvements in apparatus for the concentration of sulphuric acid and other liquids by evaporation. July 26.

16,536. O. Imray.—From G. E. Junius, France. Process for extracting baryta from barium ferrite. July 29.

16,541. B. Pitfard. Improvements in the manufacture of gaseous ammonia. July 29.

16,577. B. Piffard. Improvements in the manufacture of gaseous ammonia. July 29.

16,639. B. H. Thwaite and F. L. Gardner. A method of utilising the carbon monoxide evolved in the production of calcic carbide and apparatus for that purpose. July 30.

16,939. C. Wülfing. Improvements in the manufacture of oxalic acid. Aug. 5.

16,978. I. Carr. Improvements in or connected with apparatus or plant for the manufacture of sulphate of ammonia from ammoniacal liquors. Complete Specification. Aug. 6.

17,243. A. S. Bower. Improvements in the manufacture of sodium carbonate, caustic soda, sodium, and sodium carbide for the manufacture of acetylene gas from common salt or from sodium sulphate or sulphite. Aug. 10.

17,266. G. W. Johnson.—From Verein Chemischer Fabriken, Germany. Improvements in the manufacture of sulphurous acid, sulphuric acid, and sulphuric anhydride. Aug. 10.

17,415. J. C. Graham. Improvements in apparatus for and in the method of making caustic alkali and sodium. Aug. 12.

### Complete Specifications Accepted.

1897.

21,508. R. P. Pictet. Process and apparatus for the production of carbide of calcium. July 27.

25,007. H. S. Elworthy. Process for the manufacture of carbonic acid, and apparatus therefor. Aug. 17.

25,042. E. M. Taylor. Alkali phenate compound for bleaching, scouring, and stripping. Aug. 10.

27,049. L. H. Frentz. Manufacture of conglomerates containing alkaline oxidising bodies for bleaching linen and other objects. Aug. 17.

1898.

10,290. C. S. Bradley and C. B. Bradley. Process of manufacturing phosphorus and other materials. Aug. 3.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### Applications.

16,295. W. P. Rix. Improvements in moulding pottery and in apparatus therefor. July 26.

16,417. H. Burridge. Improvements in kilns or ovens for firing bricks and other ceramic goods. July 28.

16,550. A. J. A. Berthelier. Improvements in furnaces for baking enamels, glassware, and porcelain. Complete Specification. Filed July 29. Date applied for, 1 March 1898, being date of application in France.

16,665. V. F. L. Smidth and E. Rüsäger. Improvements in kilns for burning cement and other substances. July 30.

16,666. F. Ruppel. Improvements in kilns for cement, lime, and the like. July 30.

17,071. E. Offenbacher. An improved process for the manufacture of decorated glass. Complete Specification. Aug. 8.

17,140. T. Pfister. An improved manufacture of mosaics, tiles, bricks, and the like. Complete Specification. Aug. 9.

17,141. Improvements in cloisonné work and mosaics. Complete Specification. Aug. 9.

17,206. B. G. Davies-Cooke. Improvements in the manufacture of fancy and ornamental glazed bricks, tiles, blocks, and other glazed goods. Aug. 10.

17,209. T. Pfister and E. Barthels. Improvements in cloisonné work. Complete Specification. Aug. 10.

17,381. W. W. Pilkington. Improvements in machinery for rolling sheets of glass. Aug. 12.

### Complete Specifications Accepted.

1897.

20,414. W. Thompson. Improved glass tiles. Aug. 10.

23,080. W. S. Hadley, F. W. Sephton, and R. Mills. Method of and means for producing indelible designs upon the surface of glass, marble, metal, and similar material. Aug. 3.

1898.

10,128. G. W. Tooker. Gilding of articles of china, earthenware, porcelain, and the like. Aug. 7.

13,260. F. Schuman. Apparatus for the manufacture of wire-glass.

13,310. F. O'Neill. Machine for manufacture of glassware. Aug. 10.

13,706. J. M. Humphreys. Manufacture of glassware. Aug. 3.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

15,872. F. M. David. The manufacture and production of a new material more especially intended for use as a substitute for wood. July 20.

15,929. R. Stone. Improvements in the construction of blast furnaces for the manufacture of cements. July 21.

16,022. G. A. Kiefer and W. T. Herbst. Improvements in the manufacture or production of bricks or blocks suitable for building purposes. July 22.

16,173. O. Imray.—From La Société Anonyme des Ciments et Plâtres de Vilvorde a Vilvorde, Belgium. Improvements in the manufacture of Portland cement. Complete Specification. July 25.

16,184. C. Schneider and W. F. L. Smidth. Improvements in kilns for burning Portland cement and other substances. July 25.

16,489. W. Olschewsky. Improvements in the process of and appliances for producing artificial stone. July 29.

16,697. R. A. Rossborough and J. P. Pile. Improvements in the process of treating or preparing clay having large quantities of stones in it for the manufacture of bricks, tiles, and such like. Aug. 2.

16,777. A. Patrick. Improvements in the manufacture of cement. Aug. 3.

17,158. W. Courtenay. Improvements in and relating to artificial stone known as reconstructed granite and in process of making the same. Complete Specification. Aug. 9.

### Complete Specifications Accepted.

1897.

21,141. J. A. Jones. Manufacture of plaster for walls ceilings, and the like. Aug. 17.

1898.

5351. P. A. Winkler. New method for the production of hard stones, especially lithographic stones. Aug. 10.

## X.—METALLURGY, MINING, Etc.

### Applications.

16,312. J. Armstrong. Improvements in or appertaining to the smelting of complex sulphide ores containing zinc and lead. July 26.

16,338. J. Armstrong. Improvements in the extraction of zinc from its ores. July 27.

16,399. J. J. Hood. Improvements in gold and silver extraction. July 27.

16,622. J. Ranald. A humid process for the extraction of the metals, bismuth, and the like from their sulphide ores. July 30.

16,685. C. J. Robinson. Improvements in the treatment of ores and in apparatus therefor. Aug. 2.

16,735. W. Feit. Improvements in treating lead containing precious metals. Complete Specification. August 2.

16,865. E. L. Rhead and F. A. Burgin. A process for the extraction of and recovery of antimony and other metals from ores preliminary to the extraction of gold or other metals. Aug. 4.

17,186. T. Terell. Improvements in coating metals, also applicable to soldering metals. Aug. 9.

17,220. T. A. Ullathorne and F. Bedford. Improvements in the method of and apparatus for hardening, tempering, and tinning steel wire. Aug. 10.

17,353. W. Florence. Process and apparatus for separating zinc, cadmium, and other volatile elements from their alloys or mixtures with less volatile metal. Aug. 11.

### Complete Specifications Accepted.

1897.

18,135. T. Doherty. Method and means for manufacturing steel direct in a cupola. Aug. 10.

18,217. W. J. Wigg and E. G. Ballard. Separation and recovery of zinc from liquors containing zinc in solution with iron and other metals. July 27.

19,650. Kynoch, Ltd., and A. T. Cocking. Improvements relating to blasting. Aug. 17.

1898.

937. E. Martin. Manufacture of a compound metal of steel and silver. Aug. 10.

7377. J. A. Hunter. Method of converting iron into steel. July 27.

11,002. C. H. Pead. Separating liquids from fine slimes of gold-bearing and other ores, and apparatus therefor. Aug. 3.

11,180. J. Colquhoun. Treating copper matte. July 27.

11,265. W. P. Thompson.—From W. J. Wilder, United States. Process and preparation for coating iron or steel. July 27.

11,689. W. M. Theobald. Process of finishing sheet iron or steel plates. Aug. 3.

13,123. L. Budzinski and B. Schouwaloff. Treatment of steel. Aug. 10.

13,332. W. J. Clapp. Furnaces and apparatus for and in the recovery and purifying of iron metals, slag, and cinder tap ores, and other kinds of waste products, and for other purposes. Aug. 17.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### Applications.

15,703. E. Giglio. Improvements in primary batteries. July 18.

15,724. O. Imray.—From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Production of chromic acid by electrolytic oxidation of solutions of chromium oxide salts. July 18.

15,801. E. Leiner. Improvements in electric accumulators. Complete Specification. July 19.

15,802. E. Leiner. Improvements in electric accumulators. Complete Specification. July 19.

15,803. E. Leiner. Improvements in electric accumulators. Complete Specification. July 19.

15,967. J. W. Kynaston. Improvements in cells applicable for the production of chlorine and alkali metal amalgams by electrolytic decomposition of solutions of alkaline chlorides. July 21.

16,012. E. Bailey and T. G. Hodgson. An improved method in the production of oxide of zinc by the use of the electric arc and compound gases. July 22.

16,129. O. Imray.—From Oesterreichischer verein für Chemische und Metallurgische Production, Bohemia. Improvements in the electrolysis of solutions of alkali salts, especially alkali chlorides. Complete Specification. July 23.

16,172. H. de Rufz de Lairson. A voltaic battery cell with hot-air depolarisation. July 25.

16,461. A. Henneton. Improvements in the construction of electrodes for secondary batteries. Complete Specification. July 28.

16,556. A. J. Benedict.—From W. S. Doe, United States. Improvements in galvanic batteries. July 29.

16,590. C. Levetus. Improvements in and relating to secondary batteries and the like. July 30.

16,659. Dr. E. Stilberg. Electrolytic process for extracting metals from their halogen combinations. Complete Specification. July 30.

16,807. W. D. Gooch. Improved methods of controlling and directing electro deposition of metals and alloys. Aug. 3.

16,876. E. J. Clark. Improvements in secondary electric batteries or accumulators. Aug. 4.

16,999. E. Andreas. Improvements in or relating to electric accumulators. Aug. 6.

17,162. L. Lejeune. An improved electric accumulator. Filed Aug. 9. Date applied for, 25 Jan. 1898, being date of application in France.

17,268. O. Behrend. Improvements in secondary batteries or electrical accumulators. Aug. 10.

17,359. D. Martini. Improvements in electrodes for electrically obtaining ozone. Aug. 11.

17,458. A. A. Beadle. Improvements in primary electric batteries. Aug. 13.

17,488. L. A. Timmis. Improvements in electric secondary batteries. Aug. 13.

### Complete Specifications Accepted.

1897.

17,315. C. A. Meygret. Process for electrolitically purifying and filtering saccharine liquids or solutions. July 27.

18,796. W. M. McDougall. Secondary or storage batteries. Aug. 17.

23,332. E. A. Paris. Secondary battery plates or electrodes. Aug. 3.

24,591. A. Heil. Galvanic cells. Aug. 3.

1898.

4903. J. Ensign Fuller. Primary batteries. July 27.

13,960. H. Damm. Electrode for accumulators. Aug. 3.

14,112. A. Peters. Primary batteries. Aug. 3.

## XII.—FATS, OILS, AND SOAP.

### Applications.

15,773. H. A. Tilcock. Improvements in the manufacture of soap. July 19.

15,828. F. L. Bartelt. A washing compound for vegetable fibre or textiles. July 20.

15,838. H. Hadfield. Improvements in and connected with soap frames used in the manufacture and slabbing of soap. July 20.

16,098. H. Smith. An improved manufacture of soap. July 23.

17,087. M. Ekenberg. Improvements in the method of refining Oils. Complete Specification. Aug 8.

17,510. H. L. Haas. Improvements in cleansing compositions containing ammonia. Complete Specification. Aug. 13.

### Complete Specifications Accepted.

1897.

21,115. T. C. Palmer, of the firm of W. J. Fraser and Co. Distillation of petroleum and other oil, and apparatus therefor. July 27.

1898.

6464. S. G. Rosenblum and The Commercial Ozone Syndicate, Ltd. Oxidising of oils. Aug. 3.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

15,721. E. G. Scott. Improvements in the distillation of rosin. July 18.

16,472. E. Schliemann. Manufacture of a wax-like substance by the action of oxidising agents upon a mixture of resin and paraffin. Complete Specification. July 28.

16,607. A. E. Parker. Process for the manufacture of peroxide of lead. July 30.

17,178. Matthews, Lancashire, Cheshire, and North Wales District White Lead Company, Ltd., S. Pope, and G. V. Barton. Improvements in or connected with the production of lead oxide, and in means or apparatus employed therein. Aug. 9.

17,180. Matthews, Lancashire, Cheshire, and North Wales District White Lead Company, Ltd., S. Pope, and G. V. Barton. Improvements in or connected with the treatment of litharge. Aug. 9.

17,329. R. Matthews. Improvements in the production of lead oxide. Aug. 11.

17,330. R. Matthews. Improvements in the production of lead oxide. Aug. 11.

17,333. A. Taylor and W. S. Taylor. Improved rubber compounds. Aug. 11.

17,478. C. Weygang. Improvements relating to siccative oils and varnishes. Aug. 13.

### Complete Specifications Accepted.

1897.

19,729. R. J. Friswell and Brooke, Simpson, and Spiller, Ltd. Obtaining gutta-percha and the utilisation of by-products arising therefrom. Aug. 10.

1898.

10,263. T. Turnbull. Anti-corrosive damp-resisting paint. Aug. 17.

12,348. Saxby's White Lead Syndicate, Ltd., and H. G. Percival. Chambers for the manufacture of white lead. Aug. 10.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

16,369. J. H. Curling. Improvements in tanning hides and skins. Complete Specification. July 27.

16,419. S. Thomson and J. A. Todd. An improved method of tanning hides and skins. Complete Specification. July 28.

16,545. W. Turner. Improvements in machinery for dressing or working hides, skins, and the like. July 29.

16,857. W. Douglas. A new or improved process for accelerating the tanning and curing of hides. Aug. 4.

### Complete Specifications Accepted.

1897.

19,356. A. E. E. Cantley and E. Springborn. Artificial imitation leather. Aug. 17.

1898.

12,549. J. T. Wood. Means used for the bating of hides and skins. July 27.

## XV.—AGRICULTURE AND MANURES.

### Application.

15,749. A. J. Oxford. Converting brewers' yeast into a portable and workable manure. July 19.

### Complete Specification Accepted.

1897.

21,175. W. Walters. Manufacture of artificial manures.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

16,292. E. W. Gerbracht and F. G. Wiechmann. Improvements in the purification of saccharine juices or sugar solutions. Complete Specification. July 26.

16,540. J. Robin-Langlois. Improved process and apparatus for the rapid refining of sugar. July 29.

16,748. C. Boeye. *See* Class XVII.

16,949. S. Pitt.—From The Pharmaceutisches Institut Ludwig Wilhelm Gans. Germany. The production of soluble albumen compounds. Aug. 5.

16,979. F. M. Spence and D. D. Spence. Improvements in the manufacture of syrups and beverages. Aug. 6.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

15,711. N. van Laer. Improvements in apparatus for use in connection with the filtration of beer. July 18.

15,749. A. J. Oxford. *See* Class XV.

15,771. D. Striebeck. Improvements in cooling beer. July 19.

15,833. E. A. Hamlyn. Improvements in and relating to apparatus for drying brewery and distillery bye-products and the like materials. July 20.

16,667. R. Genge, V. Ragon, and L. Doulers. Improved method and apparatus for producing pure ethyl alcohol direct from fermented mash. July 30.

16,748. C. Boeye. Improved process for treating malt starch and other residues. Aug. 2.

16,896. A. Munsche. Improvements in and connected with the fermentation and manufacture of perfumed malt-wines. Complete Specification. Aug. 4.

17,159. F. R. Coudert, jun. Improvements in the manufacture of ethyl alcohol. Complete Specification. Aug. 9.

17,357. J. Declercq. An improved malting apparatus. Aug. 11.

### Complete Specifications Accepted.

1897.

20,955. J. E. J. Johnson. Treatment of alcoholic or fermented liquids. Aug. 17.

1898.

12,660. G. Kreutzer and J. Schmitz. Brewing and apparatus therefor. Aug. 17.

13,053. A. Collette, fils. Process for the manufacture of alcohol by saccharification and fermentation by mucedineae, and apparatus therefor. Aug. 3.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

#### *A.—Foods.*

15,815. A. M. Nathan.—From F. W. Stockhausen, West Indies. Improvements in the manufacture and preservation of butter and other products from cocoanut fruit. July 19.

16,195. A. Rehse. Improvements in or connected with preserving food. Complete Specification. July 25.

16,699. W. B. Hart and C. A. McKerrow. Improvements in or connected with the purification treatment of white or black peppers in the corn or berry or other state. Aug. 2.

17,278. J. Peeters. Improvements in the manufacture of extracts from yeast suitable for use as condiments and for other analogous purposes. Filed Aug. 10. Date applied for, Feb. 25, 1898, being date of application in Belgium.

#### *B.—Sanitation.*

16,217. F. M. Spence and D. D. Spence. Improvements in the treatment of sewage. July 26.

16,767. D. B. Cumming. Improvements in or connected with the destruction of distillery refuse. Aug. 3.

16,970. C. J. Lomax. A machine for the extraction of liquid from sewage sludge and other substances. Aug. 6.

17,257. M. Otto. A process of and apparatus for the purification of water and other liquids. Complete Specification. Aug. 10.

#### *C.—Disinfectants.*

15,819. A. E. Berry and A. B. Roberts and Co., Ltd. Manufacture of a new chemical salt of valuable antiseptic properties. July 19.

15,867. G. A. Galetto. Improvements relating to the production of formaldehyde vapours. July 20.

16,046. W. P. Thompson.—From J. G. Brissonnet, France. Improved antiseptic and disinfectant compound. July 22.

### Complete Specifications Accepted.

#### *A.—Foods.*

1897.

19,357. H. Higgins. Proteid breads, biscuits, and cakes. Aug. 17.

19,980. C. Estcourt. Production of a new food for human consumption. July 27.

20,527. A. W. Rothwell.—From T. T. Rothwell, Victoria. Elimination of tannin from tea in infusion. Aug. 17.

1898.

11,414. L. Buchhold. Process for preserving preparations or objects. Aug. 3.

### *B.—Sanitation.*

1897.

22,965. D. Cameron, F. J. Commin, and A. J. Martin. *See* Class I.

27,349. D. Cameron, F. J. Commin, and A. J. Martin Apparatus for the treatment of sewage or other liquids. Aug. 17.

### *C.—Disinfectants.*

1897.

18,178. W. L. Wise.—From Knoll and Co., Germany. Manufacture of antiseptic substances. July 27.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

15,673. W. R. Meggeson, G. Pelmear, and W. H. Cummings. Uninflammable celluloid. July 18.

16,780. J. F. Bennett and W. Appleyard. An improvement in the treatment of cardboard, paper pulp and other substances of a fibrous nature. Aug. 3.

16,996. L. Lityñski, A. Rodakiewicz, and F. Kurowski. Improvements in rendering fireproof wood, textile fabrics, paper pulp, straw, and the like. Aug. 6.

17,052. W. Wild and A. H. Wild. Oleo parchment. Aug. 8.

### Complete Specifications Accepted.

1897.

15,601. A. Von Feszty. Production of paper pulp from peat. Aug. 3.

1898.

6651. F. Buhler. Preparation of cellulose. Aug. 3.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

15,907. J. L. Hawliczek. Improvements in the manufacture of acetone. July 21.

16,122. M. Otto. Improvements in the production of iodoform. Complete Specification. July 23.

16,965. L. Lilienfeld and F. Silberstein. Process for the synthetic production of proteids or of compounds having the chemical properties of proteids. Aug. 5.

16,994. H. S. Wellcome and S. B. Schryver. Improvements in the manufacture of fine chemical products for use in pharmacy and other purposes. Aug. 6.

17,304. W. P. Thompson.—From J. C. Kreis, Germany. An improved process for producing pyrotartaric diphenylester as a preparation for medicinal purposes. Aug. 10.

### Complete Specifications Accepted.

1897.

14,614. A. Verley. Manufacture and production of perfumes. July 27.

18,478. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Manufacture or production of pharmaceutical compounds. Aug. 10.

19,204. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Process for producing aromatic aldehydes. July 27.

23,004. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or preparation of adenin. Aug 17.

23,005. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or preparation of hetroxanthine, paraxanthine, and methylated hypoxanthines. Aug. 17.

1898.

7740. S. S. Bromhead.—From The Chemische Fabrik, Germany. Improved process for the production of iodized albumen and iodised peptones. July 27.

## XXI.—PHOTOGRAPHY.

### Applications.

16,959. A. J. Boult.—From E. Goldschmid, Germany. Improved method of producing photographic pictures. Aug. 5.

17,167. C. Dicker. Improvements in and relating to the production of coloured photographs. Aug. 9.

17,437. G. Selle. Improvements in the production of photographs in natural colours. Aug. 12.

### Complete Specification Accepted.

23,739. H. W. Cottrell. Method of producing coloured photographs. Aug. 17.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

16,037. The firm of Bohy, Gallay and Co. Improvements in the manufacture of matches. Filed 22 July. Date applied for, 11 March 1898, being date of application in Switzerland.

16,314. H. Sévène and E. D. Cahen. Improvements in the manufacture of inflammable pastes suitable for matches. Filed 26 July. Date applied for, Feb. 1, 1898, being date of application in France.

16,403. W. L. Wise.—From the Maschinenfabrik Oerlikon, Switzerland. Improvements in time fuses. July 27.

16,480. W. Hope. Improvements in pellets and grains of gunpowder of any kind. July 28.

16,481. W. Hope. Improvements in gunpowders and other explosives. July 28.

16,953. O. Imray.—From F. W. Slack, South African Republic. Improvements in the manufacture of explosives. Aug. 5.

17,097. A. Minter. Phosphorusless matches. Aug. 8.

17,181. A. Riechwald.—From the firm of F. Krupp, Germany. Improvements in or relating to time fuses. Aug. 13.

### Complete Specifications Accepted.

1897.

19,418 S. Neuberg and I. Neuberg. Manufacture of matches. Aug. 10.

20,223. E. S. Clark. Safety tamping for explosives used for blasting operations in coal and other mines, and apparatus for use in connection therewith.

23,340. G. Beneké. Manufacture of explosive compounds. Aug. 17.

25,806. O. Hohmrodt. Fog-signal for railways. Aug. 10.

1898.

9190. U. Alvisi and E. Pultici. Manufacture of explosives. Aug. 10.

11,671. E. S. Clark. Safety tamping for explosives used for blasting operations in coal and other mines, and apparatus for use in connection therewith. Aug. 10.

## PATENTS UNCLASSIFIABLE.

### Applications.

15,758. C. A. Allison.—From L. Silverman, United States. Obtaining products from the pith of cornstalks and utilising same. Complete Specification. July 19.

16,158. J. Baier and E. Clark. Improved imitation whalebone. July 25.

17,258. M. Otto. An improved product obtained by the dissolution of ozone in petroleum.. Aug. 10.

17,166. W. S. Hill and W. P. Ingham. Improvements in the manufacture of slag wool or silicate cotton. Aug. 13

17,497. G. N. Dane. Improvements in fireproofing compositions. Complete Specification. Aug. 13.

### Complete Specifications Accepted.

1898.

12,416. A. Krefting. Improved system or apparatus for treating seaweed for the manufacture of products therefrom. Aug. 3.

13,289. A. Krefting. Manufacture of organic products from seaweed. July 20.

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C. for the Society of Chemical Industry.

THE JOURNAL

OF THE

# Society of Chemical Industry.

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## NOTICES.

In consequence of the lamented death of the Hon. Treasurer, Mr. E. Rider Cook, all remittances should be made payable to the General Secretary until further notice.

### Collective Index.

In reply to numerous inquiries, the compilation of the Collective Index is in active progress, the bulk of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer and Committee who have the work of correction in hand.

The prices are as follows:—

To Members (see Rules 25 and 27) and Past Members (see Rule 30); Libraries, Corporations, and Exchanges on the Society's List. Each copy 10*s.*
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Notice is hereby given, for the information of members and advertisers, that the advertisement columns of this Journal have been contracted for by Messrs. Eyre and Spottiswoode, the Society's printers and publishers, to whom all communications respecting them should be addressed. The circulation of the Journal is now more than 3,500 per month.

Authors of communications read before the Society, or any of its Local Sections, are requested to take notice that under Rule 43 of the Bye-laws the Society has the right of priority of publication for three months of all such papers. Infringement of this Bye-law renders papers liable to be rejected by the Publication Committee, or ordered to be abstracted for the Journal, in which case no reprints can be furnished to the author.

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Tripp, Dr. E. Howard, l/o Margate; The Grammar School, Swansea.

Vanderpoel, Dr. F., l/o Newark; 153, Center Street, Orange, N.J., U.S.A.

## Deaths.

Owen, Thos., M.P., 5, Whitehall Gardens, London, S.W. July 10th.

Trimble, Prof. H., 145, North 10th Street, Philadelphia Pa., U.S.A. Aug. 24th.

Verel, W. A., Fairholm, Larkhall, N.B. Sept. 8th.

# Obituary.

## EDWARD RIDER COOK, J.P., F.I.C.,

### TREASURER OF THE SOCIETY OF CHEMICAL INDUSTRY, AND PRESIDENT OF THE SOCIETY FOR THE YEAR 1890–91.

WITH extreme regret the death is herewith announced of Mr. E. Rider Cook, the treasurer of this Society. The sad event occurred on Sunday morning, August 21st, at his residence, Woodford House, Woodford Green. The immediate cause of death was cerebral hæmorrhage, and there is no doubt that the extreme heat of the week preceding mainly contributed to that result.

Edward Rider Cook was the eldest son of the late Edward Cook, of Crix, Hatfield Peveril, and of Bow, and was born on the 4th June 1836. He received education at the well-known St. Saviour's Grammar School, and the City of London School, afterwards matriculating at the University of London. Always studious and reflective, science claimed him, but at the age of 17 he dutifully abandoned a scientific career in order to assist his father in business, the soap works, which bear his father's name, being then comparatively small. So great was his progress and general aptitude for business, that at the early age of 21 he was taken into partnership in the firm of Edward Cook and Co. Their works were then at Goodman's Yard, Minories, and afterwards at Bow.

In 1881, when the Society of Chemical Industry was formed, Mr. Rider Cook heartily supported the movement, and was elected first Hon. Treasurer of the Society, at the well-remembered Burlington House meeting of April 4th in that year. This post he continued to fill without intermission until his death.

In 1890, his conspicuous services to the Society received suitable recognition by his election as President, and his Presidential Address was delivered at Trinity College, Dublin, in 1891 (this Journal, 1891, 598—602).

The Imperial Institute, having just then completed its constitution and having recognised the status of the Society of Chemical Industry, Mr. Cook, by virtue of his office, became the first representative on the Board of Directors. This election was repeated by special resolution at every subsequent Annual Meeting, up to that at Nottingham in July last.

Through good report and ill, Mr. Cook has been one of the truest friends East London has ever had to serve its interests. With indomitable courage, patience, and courtesy, he laboured for the general good. He lived to see much that he worked for accomplished, the condition of East London materially improved, and many abuses swept away.

In 1856, when the constitution of a definite form of local administration for the metropolis was inaugurated, he first came to the front as a member of the Poplar District Board of Works, where he established a reputation for patient and persevering work.

Mr. Cook devoted attention to all schemes for the benefit of East and South-East London, and he lived to see in one direction a greatly needed improvement, *viz.*, the Thames communications below London Bridge. He was among the first to move in this matter, and was a member of the first local committee with the Right Hon. C. T. Ritchie, then M.P. for the Tower Hamlets, which agitated the question. He was one of the principal witnesses before the Select Committee of the House of Commons, and it was largely due to the importance of his evidence, and the impression which he created, that the Bill authorising the construction of the Tower Bridge, and later that for the Blackwall Tunnel, was passed. Mr. Cook, in conjunction with a few other East End representatives, and Sir Joseph Hazalgette, devoted great time to the tunnel scheme, and it was owing as much to his efforts as to those of anyone else, that this great highway has at last been provided for the people and traders of East London. Had it not been for the persistence of his representations, the City would never have taken up the Tower Bridge scheme. Akin to these movements was the Woolwich Free Ferry, which was completed under the Metropolitan Board.

Mr. Cook was a Justice of the Peace of three counties, Essex, Middlesex, and London, and the public bodies with which he was connected are numerous. He was a member of the Dagenham Sewers Commission, the Poplar and Metropolitan Board of Works, Chairman of the Urban Council for Woodford, and for some years Chairman of the Lee Conservancy Board, in which position he did good work in assisting to purify East London's waterway. In this administrative duty he took a keen interest, and up to the time of his death was personally concerned in new schemes for the improvement of the drainage system of the Lee Valley.

In the great cholera epidemic of 1868 he was Chairman of the Bow Committee, and he worked day and night till the plague was stopped. His experiences at this time specially directed his attention to the importance of sanitary science, which has since always found in him a warm supporter. His early leanings towards science were later encouraged, especially as applied to manufacture and commerce, whilst it was always a favorite idea of his that scientific method brought to bear upon sanitary questions would result in a more healthy mode of living among the poor. If further evidence were needed of his practical interest in the people's lives it could be found in his connection with the Shadwell Fish Market, of which company he was chairman. As one of its founders, he had great difficulty in obtaining the necessary Parliamentary powers, the influence of the Billingsgate dealers being so great. But they were obtained at last, and with Mr. Spencer Charrington, M.P., he stood by the company through all its fortunes, for he firmly believed that its success was imperative, in order to open the market to the East End poor, who needed to be supplied with cheap fish. Year after year the directors worked, up to recently, with financially poor results, in spite of the fact that fish has been cheaper, fresher, and more readily obtainable in the East End. Better reports are now presented every year, and Mr. Cook had the satisfaction of knowing that his time and labour had not been spent in vain.

It was to be expected that such an energetic spirit should have political opinions. He was an advanced Liberal, but he did not allow political opinions to sway him unduly, nor did political considerations enter into his local or administrative work. In fact, there were many who did not know his politics until in 1881 he stood as the Liberal and Radical candidate for the North Division of West Ham, where in 1885 he was successful.

That a public record such as this should have its corollary in private life is natural. Mr. Cook was kindness itself. As a personal friend he has unostentatiously helped many struggling men. He was an ardent and a munificent Nonconformist, and a firm believer in religious liberty and equality. In business, Mr. Cook carried out the principles which he applied to public affairs. He and his partners built up the East London Soap Works at Bow until they took a high rank amongst similar industrial concerns. He believed that concern for the labouring element ought not to stop at the platform and end with an election address. He saw in his workpeople men and women who were helping him to fortune, and decided, as many other employers have done, that as far as possible they should share in the prosperity of the concern. So a profit-sharing scheme was formed, and has been working in connection with the firm for a number of years with success. Quite recently Mr. Cook presided over the annual distribution of profits, when all grades of the workers were assembled. In this way the East London Soap Works has obtained an enviable reputation, and the happy responsibility rests upon him whose death is genuinely mourned throughout a large and varied circle, including many an humble home.

# Scottish Section.

## ERRATUM.

## THE REDUCTION OF NITRIC OXIDE BY COPPER.

BY THOS. GRAY, B.SC.

(*This Journal, Aug.* 1898, p. 741.)

In table 3, col. 2, p. 742, for "percentage of nitrogen" read "percentage of nitric oxide."

# Journal and Patent* Literature.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Storing Liquids or Gases under Pressure, Impts. in Metallic Capsules for.* J. B. Bourseau, Paris. Eng. Pat. 14,718, July 4, 1898.

"HITHERTO, in order to discharge capsules or cartridges of this type, it was necessary to puncture them in order to let the contents escape." According to the present invention, a capsule is constructed which can be opened without puncturing. The claim is for "A metallic capsule for storing liquids or gases under pressure, consisting in the combination of two tubes sliding one within the other, one of which may have an enlarged end the upper rim of which is bent inwards, the joint being formed by a cap and a ring or washer, the internal pressure causing this washer to press against that portion of the outer tube, which has been bent inwards."—R. S.

*Capsules or Containers for Containing Compressed or Liquefied Gases, Impts. in.* E. Sterné, Paris. Eng. Pat. 15,402, June 28, 1897.

"A capsule or container for holding compressed or liquefied gases, which has a small hole in the body" or closing appliance, and "which, before charging with gas, is closed by means of any suitable soluble or decomposable" substance, "capable of being dissolved or transformed by contact with the medium in which the capsule or container is placed," thus "automatically discharging the contents thereof."—R. S.

*Filters for Water and other Liquids, Impts. in.* I. Fokchaner, Brussels. Eng. Pat. 9605, April 26, 1898.

THE filter consists of an outer vertical casing through the bottom of which passes the exit tube, which extends upwards into a central hollow of an otherwise solid carbon block, surrounded on all sides by pieces of carbon, which are retained in place by a tin cylinder. The latter is closed at the top but open at the bottom, and is perforated around its sides. Between this cylinder and the casing is packed pulverised carbon. A sheet of woven asbestos entirely covers the latter, and supports a layer of animal charcoal on which is a spreader or distributor for the liquid. The cover, which is dome-shaped and securely bolted on, is provided internally with a sheet of woven asbestos covered with a layer of asbestos fibre. The liquid to be filtered passes downwards through the filter. When it is required to clean out the filter, water is passed upwards, entering at the usual outlet, a connecting pipe joining the charging and discharging pipes, and suitable valves being arranged on them.—R. S.

*Compressing Powdered Substances, Impts. in Apparatus for.* P. E. M. Jamain, Dijon, Côte d'Or. Eng. Pat. 24,014, Oct. 18, 1897.

REFERS to the apparatus for compressing powdered substances described in this Journal, 1898, 744, and consisting of modifications whereby as the powdered substance is brought between the stamps, it takes up the whole free space, and when the stamps are set in motion, it becomes evenly and energetically compressed all over the surface of the tablet or lozenge.—R. S.

*Cooling Liquids, and Condensing; Impts. in Apparatus suitable for.* W. R. Renshaw, Queen Anne's Mansions, Westminster. Eng. Pat. 18,819, Aug. 13, 1897.

WITHIN a vertical cylindrical casing is a vertical rotating shaft, carrying a fan and also circular dishes, which do not extend to the sides, and beneath each of which is a series of overlapping annular shelves. The liquid enters at the top by a tube, and is thrown from each dish outwards, flowing back to the next dish over the annular shelves.

At the bottom of the shaft is a fan.

The liquid to be cooled is delivered by a pipe into the upper dish, and is thrown by the centrifugal force over its edge in a convolvulus-like jet. When the jet strikes the casing, the fluid runs down on to the top shelf of the first series, from whence it falls to the next shelf, and so on, flowing downwards and inwards until it drops from the bottom shelf into the second dish, from which it is similarly ejected in a convolvulus-shaped jet to fall on to the second series of shelves, and so on. The fan draws air or vapour into the apparatus, and forces it upwards between the shelves and through the convolvulus-shaped jets.—R. S.

*Separation of Liquid from Bodies containing or Mingled with the Same, Impts. in or relating to the.* W. A. Koneman, Battersea, and W. H. Hartley, Moorfields. Eng. Pat. 17,848, July 29, 1897.

THE bodies to be treated are more particularly what are termed "slime-bearing materials," but paper pulp or other industrial products from which it is desired to remove solutions or wash-water, may also be similarly treated. The essential feature consists in compacting the material by means of fluid pressure above, and withdrawing the expressed liquid from the top. The slime, &c. is charged into a vertical closed cylinder having a filter near the bottom, beneath which is a space which can be put into connection with an exhaust, whilst positive gaseous pressure is applied to the top. Near the top is a surface filter connected with a vacuum apparatus, and having a slack and weighted filter fabric, and also a removable agitator. Only a little water runs away from the bottom, the mass beginning to compact, the heavier particles first, and then the lighter ones, most of the water passing off from the top through the surface filter. Both quantities of water are passed to an auxiliary filter. The compacted contents may be discharged by removing the coned bottom and moving the bars supporting the mass as required, the bars being fitted so as to make this possible; or the compacted mass may be subjected to pressure from below, for the purpose of breaking up the mass.—R. S.

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* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Covering and Preservation from Rust of Exposed Ironwork, A New or Improved Process and Composition for the.* K. B. Skeet, Chichester. Eng. Pat. 14,595, July 2, 1898.

*See under* XIII. B., *page* 854.

## II.—FUEL, GAS, AND LIGHT.

*Coal, Indian; Chemical Examination of.* W. R. Dunstan. Imp. Inst. (Indian Sect.) Annual Report, 1897–98, 40—44.

The examination extended to the estimation of fixed carbon, volatile matter, ash, sulphur, and calorific power, together with the general characteristics and caking quality of each sample.

*Sampling.*—The large blocks (½ to 1 cwt.) were cut, and pieces of several pounds weight were taken, broken up, and averaged.

*Technical Analysis.*—The *volatile matter* was determined by heating about 0·2 grm. of powdered sample in a platinum capsule for two minutes over a No. 8 Fletcher-Bunsen burner, and for a further two minutes at bright red heat in a blow-pipe flame, the loss being ascertained by weighing, after cooling in a desiccator. The residue was then incinerated in a muffle furnace and the *ash* weighed, the *fixed carbon* being estimated by difference.

*Sulphur.*—About 1·5 grm. of the sample was fused in a platinum dish with 30 grms. of a mixture of sodium chloride 4 parts, potassium nitrate 3 parts and dry sodium carbonate 1 part, the fused cake being dissolved in boiling water, and the solution filtered, acidified with hydrochloric acid, and precipitated hot with barium chloride,—the whole being kept warm in order to produce a granular precipitate. A modified Gooch filter, in the shape of asbestos felt, contained in a perforated platinum crucible, was employed. The method is more rapid than the ordinary one and is equally accurate.

*Ultimate Analysis.*—The usual method was pursued, the sample being first dried at 120° C. in a current of hydrogen for ½ hour, and then burned in a combustion tube containing copper oxide and lead chromate.

The larger percentage of ash obtained in some cases, in comparison with the results of the technical analysis, is ascribed to the higher state of oxidation of the iron, &c., occasioned by heating in oxygen.

*Calorific Equivalent.* — The Thompson calorimeter (a standardised intrument) was employed.

*Results.*—The tabulated statement occupies several pages of the report. In addition, the characteristics of the coals of each district form the subject of the following comments:—

*Assam.*—The samples examined do not bear out the reputation of Makum coal as a steam coal. Ash content low (1·02—3·65 per cent.); sulphur high (1·07—4·87 per cent.); fixed carbon (47·84—53·28 per cent.) lower than recorded by Mallet in 1876 (75·7 per cent.). One sample gave a soft coke rich in sulphur, the other was non-caking.

The Cherrapunji coal, though poor in ash (1·74 per cent.), has a high sulphur content (3·98 per cent.), but is a hard caking coal, burning like cannel. The Maoflung sample resembles the other two, and that from the Dikhu Valley is a non-caking bituminous coal.

The Assam coals are probably serviceable as steam coals, but the samples are too rich in sulphur to use for iron-smelting.

*Baluchistan.*—The Khost coal is tertiary and somewhat bituminous, the sample from the Khost seam being probably a fair caking steam coal, though rich in sulphur (4·82 per cent.). That from the Killa-Hakim seam is low in sulphur (0·74 per cent.), and appears to be a fair caking coal of greater calorific power (6,936 cals.) than the analytical results would imply.

*Bengal.* — Fifty-three samples were examined. The quality varies greatly, much being excellent with a content of 50—60 per cent. of fixed carbon, a calorific value of more than 6,000 cals., and seldom over 10 per cent. of ash, with, frequently, a low percentage of sulphur. A good deal is serviceable steam coal, whilst many samples cake well, are poor in sulphur and the coke is therefore suitable for smelting.

Karharbari coal is largely used for coking for locomotive use, and in the East India Company's Jamalpur factory.

Barakar coal is in good repute as a steam coal, and yields a coke poor in sulphur. Both Sanctoria and Giridih coals are regarded favourably for gas-producing.

*Burmah.*—Both the samples examined were of poor quality, the one—from the roof of a seam—being an inferior caking coal, difficult to burn; whilst the other—from the floor—is valueless, being almost incombustible and little better than shale.

*Central India.*—None of the Rewah samples are as good as Bengal coal, the ash being high (14·94 to 26·60 per cent.), and the fixed carbon (43·42 to 63·63 per cent.) and calorific power (5,247 to 5,940 cals.) low. The sulphur content is not excessive (0·32 to 2·03), and the picked coal might do for locomotive firing, but is unsuitable for coking, being a non-caking coal.

*Central Provinces.*—The samples from Mohpani are fair, non-caking coals, low in sulphur (0·47—1·03 per cent.), but, in two cases, rich in ash (up to 24·3 per cent.). The Warora samples are similar, but contain less fixed carbon and more sulphur (up to 2·37 per cent.). The existence of iron ore and limestone is reported in this district, which might be worked if a good caking coal could be found there.

Gadawarra coal is similar in fixed carbon content, but differs in that it cakes well. Sulphur, about 0·42 per cent.; ash, 19—20 per cent. in two samples, but lower (9·23 per cent.) in the third.

*Nizam's Dominions.*—Two samples from Hyderabad are of fair quality as steam coals, one caking but little and the other not at all. In the latter, the sulphur is low (0·29 per cent.) and the ash high (12·71 per cent.), in the former, the sulphur is high and the ash low, these being respectively 1·28 and 8·61.

*Punjab.*—The Dandot sample is a good non-caking steam coal of relatively high calorific power (5,995 cals.), though low in fixed carbon (38·32 per cent.). Pidh coal is similar but better, sulphur content rather high (3·20 per cent.). Baghanwalla is a poorer, slightly caking coal. The sample from Shabrig is a bituminous caking coal, poor in fixed carbon (38·74 per cent.), but of high calorific power (6,730 cals.), due to solid hydrocarbons; is probably useful, but fast-burning.

*Shale.*—Two samples, from Pidh and Dandot, were examined, but found unsatisfactory, the yield of oil being inappreciable, and the production of gas (of poor quality, moreover), only ¼—⅓ that furnished by good English coal.
—C. S.

*Photometric Standards.* W. Grafton. J. Gas Lighting, 1898, **72**, [1836], 150—151.

The author has come to the following conclusions after a prolonged investigation :—

*Dibdin Ten-Candle Standard.*—This so-called 10-candle standard is stated to be really equal to 10·47 certified sperm candles, and is not reliable or constant, being affected by (*a*) the percentage of pentane vapour in the air; (*b*) small differences in the height of the flame; (*c*) to a great extent by a close atmosphere; (*d*) moisture also largely affects it without going over a great range of temperature, that of the testing stations being 50°—84° F., while occasionally a lower temperature may be found in winter. In using this standard, the following precautions should be observed:—A correction for moisture should be made, and the aperture in the screen reduced by $3\frac{1}{2}$ mm., *i.e.*, the opening in the screen above the top of the steatite should be 51 mm. in height. The standard should be allowed to burn at least 20 minutes before use, in order to cool the carburetter to a normal point. The air should always be thoroughly saturated when leaving the carburetter; on passing it over more pentane of the same temperature, no more of the latter should be taken up. When the standards are alike, they should agree to within 1 per cent. of each other, irrespective of the end of the photometer in which they may be fixed. It should be noted "that the quantity of pentane in the carburetter affects the composition of the resulting carburetted air; that coal-gas is practically an equivalent of air as a carrier of pentane vapour at all temperatures above 60° F., very little difference in light value being observable. For temperatures under 60° F., less gas is required than air; also the greater the rate of gas or air, the higher is the power of the standard."

*Methven Standard.*—This standard is stated to be reliable, and practically constant; it shows only $\frac{1}{2}$ per cent. variation due to temperature and pentane vapour, whilst moisture has no appreciable influence upon the portion of the flame used as the standard. The light passing through the small slot from a portion of a $2\frac{1}{2}$-in. pentane-gas flame is equal to two certified sperm candles. There is no real difficulty in setting the flame to $2\frac{1}{2}$ ins. when the atmosphere is still, as it is in a properly constructed photometer and room. The readings or observations of the disc made by the use of this standard are much more delicate than by one having a higher standard. The author considers that the principle of the standard, as proved by experiment, is sound, and as near perfection as can be hoped for; and he states that no whole or greater part of a flame can be constant.

*Pentane Air-Gas Standard.*—It is shown that this standard is only reliable between 60° and 70° F., and that if it agrees with the 10-candle standard having a 2·15-in. opening in the cut-off screen, it must be of greater value than one average certified sperm candle.

*Harcourt Ten-Candle Lamp.*—This standard in its present form is said to be "not reliable." Its power is variable, it is too greatly affected by temperature, and the central air current is insufficient. The discs, as forming part of the apparatus, should be certified, as required from time to time, by the gas referees.—A. S.

*Carburetted Water-Gas.* W. Leybold. J. für Gasbeleucht. **41**, [32], 509—512.

Two-thirds of the gasworks of the United States, which was the birthplace of carburetted water-gas, now supply that gas, and its manufacture has spread thence to England, and is now being introduced in Germany. In London, the Gas Light and Coke Company have plant for the production of $16\frac{1}{4}$ millions of cubic feet of carburetted water-gas per diem; but here and elsewhere in Europe—with the exception of the Garston Works, at Liverpool, which supply this gas undiluted—water-gas is used for admixture with coal-gas. Carburetted water-gas has lately been introduced into Germany, where it has been adopted at Bremen. The advantages of carburetted water-gas in the United States were stated by Bunte in 1894 to be: (1.) The utilisation of petroleum, which is exceedingly cheap throughout that country. (2.) That the gas may be made of 25 to 32 candle-power, while the candle-power of coal-gas does not usually exceed 16. (3.) Economy in wages, since fewer workers are required than for coal-gas manufacture. There are, however, other causes which favour the introduction of this gas in England and European countries, where petroleum is not a native product. Among these are: (1.) The small cost of the installation. For instance, carburetted water-gas plant for a daily make of 50,000 cb. m. (say $1\frac{3}{4}$ million cubic feet) costs only 600,000 marks, whereas coal-gas plant of the same capacity costs 3,000,000 marks. (2.) The small area occupied by the plant. The generators, washers, and condensers take up only one-third the space needed for coal-gas plant of the same capacity. (3.) The utilisation of coke, which could not be disposed of as fuel for so high a price. A production of 25 per cent. of coal-gas and 75 per cent. of carburetted water-gas, would leave no coke to be otherwise disposed of. (4.) 100 kilos. of petroleum are equivalent in illuminating value to 400 to 500 kilos. of good cannel; hence considerably less space is required to store oil than cannel for the enrichment of coal-gas to a given extent. Oil is more easily discharged from barge than is cannel. (5.) The freedom of the gas from by-products, such as ammonia, and consequently absence of the necessity for washing it carefully. The tar, amounting to 10 to 12 per cent. of the oil used, is either added to the coal-tar or used as fuel. (6.) The small amount of sulphuretted hydrogen in the gas, so that less purification is needed than with coal-gas. (7.) The small amount of other sulphur compounds in the gas. The amount even falls below the maximum permitted in the gas supplied to London, and no steps need be taken to reduce it. (8.) Unless the removal of carbonic acid is considered necessary, only oxide of iron need be used for the purification of the gas. (9.) The illuminating power of the gas made can be changed from that of ordinary coal-gas to 40 candles in a few minutes. (10.) The generator can be made ready for gas-making in less than four hours, whilst two to four days are required to raise retort settings to the proper heat for making gas. Gas-making may be discontinued during the night or on Sunday, and recommenced at half to three-quarters of an hour's notice. The system is therefore specially adapted to cope with fogs, or any emergencies which may call for a sudden increase in production.

The small number of men required to work a water-gas plant make it valuable in case of strikes of workmen. The labour also is light, except that the removal of clinker from the generator fire bars is difficult if the ash fluxes readily. This labour is, however, not more arduous than the drawing and charging of retorts, and the tending of retort furnaces. It has been observed at Belfast that the admission of carburetted water-gas to the distributing system abolished naphthalene stoppages, which had previously been very troublesome. Carburetted water-gas burns in batswing burners with a beautifully white and steady flame; in Argand burners the flame is white but short. Its mixture with coal-gas burns in batswing burners just like coal-gas enriched with cannel gas, but in Argand burners the flame appears to be rather shorter.

The disadvantage of carburetted water-gas is the large proportion of carbonic oxide which it contains. The percentage of carbonic oxide in coal-gas is from 8 to 10; in carburetted water-gas of 25 candle-power from 26 to 30. In most cases an addition of 20 per cent. of carburetted water-gas to coal-gas is the greatest that will be made, and this addition increases the percentage of carbonic oxide in the gas from about 9 to 13.

The report of a deputation from Berlin, which visited several British installations, recommends the erection of carburetted water-gas plant at all the municipal gasworks, in order that the exceptional demand in the winter months may be thus provided for. The amount paid for wages at the five municipal gasworks in Berlin in 1893–94 was 23·4 per cent. of the total cost of producing the coal-gas, while the wages would amount to only 3 to 9 per cent. of the cost of carburetted water-gas of similar illuminating value. Therefore carburetted water-gas renders a works more independent of the cost of labour.

Coke for the manufacture of carburetted water-gas should contain little ash, and that should not flux readily. Gas-coke serves equally as well as smithy coke. It is economical to use the red hot coke as it is drawn from the retorts. The amount of coke used is about 560 kilos. per 1,000 cb. m. (about 35 lb. per 1,000 cb. ft.) of 15—16-candle gas, or 800 kilos. per 1,000 cb. m. (about 50 lb. per 1,000 cb. ft.) of 25-candle gas. The cost of the gas therefore fluctuates with the current value of coke.—J. A. B.

*Spirit for Illuminating Purposes.* M. Hayduck. Zeits. für Spiritusind. **21**, [33], 289.

By the addition of certain hydrocarbons to spirit (Zeits. für Spiritusind. 1898, [2]), it is possible to obtain a flame equal in brightness to that of petroleum, but the cost and consumption of the illuminant for an equal light is considerably greater than when petroleum is used.

The same statement holds for the preparation lately introduced by Guttmann under the name of "Lucin" and for the Illuminating-spirit of Hempel.

In the present paper the author tabulates the results of some experiments, comparing the two last-mentioned illuminants with ordinary petroleum, but with no improvement in the results already above stated.—J. F. B.

*Strontium Sulphides, Composition of the Phosphorescent.* J. R. Mourelo. Comptes Rend. 1898, **127**, [4], 229—231. (See also this Journal, 1897, 704; 1898, 189, 282, 613.)

The phosphorescent strontium sulphides are mixtures, rich in strontium sulphide, and always produced at high temperatures. In using celestine containing 98·65 per cent. of strontium sulphate, the product obtained by reduction with lampblack contains 0·017 per cent. of strontium sulphate, at the most, in recent preparations. This proportion may reach 10—12 per cent. by long exposure to air. If the air is damp, sulphuretted hydrogen is evolved and the phosphorescent property lost. Strontianite, containing 89·15 per cent. of carbonate, 8·37 per cent. of calcium carbonate, and 2·04 per cent. of barium sulphate, when reduced by sulphur, gave a phosphorescent sulphide, as also did artificial strontium carbonate containing only traces of sodium chloride and carbonate. Pure strontia reduced with sulphur in the entire absence of air produced a non-phosphorescent sulphide, which became phosphorescent when mixed with very small quantities of sodium carbonate and chloride, 0·02 grm. and 0·001 grm. respectively, to 53 grms. of sulphide, and heated for three hours with some excess of air.—A. C. W.

*Briquette Factory at Langenberg, Province Pomerania.* Foreign Office Ann. Ser., No. 2177, Aug. 1898.

*See under* Trade Rep., *page* 884.

## PATENTS.

*Gas, Impts. in and Apparatus for the Manufacture of Illuminating.* V. B. Lewes, Greenwich. Eng. Pat. 17,749, July 28, 1897.

Coke or other carbonaceous fuel is raised to incandescence in a furnace fitted with air blasts, and converts the tar or other heavy liquid hydrocarbon, and steam injected into the glowing fuel through suitable jets, into a mixture of methane, hydrogen, and carbon monoxide (or water gas). The evolved gases are delivered to a vertical superheater of chequer brickwork and iron ingots, where a fresh quantity of water gas is generated by the contact of injected steam with any undecomposed tarry matter that may have passed over from the furnace. Thence the gaseous mixture is conducted to a scrubber, and subsequently to an acetylene generator containing calcium carbide, through which the moist gas ascends, liberating acetylene in its passage. Further enrichment of the gas by acetylene is provided for by imparting a shaking motion to the perforated hopper containing the carbide, whereby variable quantities of the latter can be discharged into the water in the bottom of the generator.

The inventor obtains satisfactory results as regards illuminating power, when about 10 per cent. of acetylene is mixed with a gaseous mixture containing hydrogen, 52 per cent.; carbon monoxide, 16 per cent.; methane, 32 per cent.—C. S.

*Oxide of Iron [Gas Purification, &c.] used for the Absorption of Sulphuretted Hydrogen in the Purification of Illuminating or other Gases, or otherwise; Impt. in the Treatment of.* F. M. Mooney, Dublin. Eng. Pat. 19,590, Aug. 25, 1897.

The residue from the burning of "spent oxide" may be rendered available for use over again, by quenching with water whilst still hot from the kiln—a procedure which thoroughly impregnates it with water and extracts the free acid present—and, after straining, incorporating it with some fibrous organic matter, such as peat-mould, sawdust, &c., so as to render the whole sufficiently porous to admit the free passage of the gas to be purified.—C. S.

*Gas, Impts. in the Manufacture of, from Garbage and other Refuse.* L. G. Harris, Toronto, Canada. Eng. Pat. 19,887, Aug. 28, 1897.

The garbage or refuse employed is incorporated by sprinkling and agitating with about one-twentieth part by weight of a mixture compounded of: alcohol, 2 quarts (or water, 2 galls.); caustic alkali, 4 lb.; chloride of lime, 8 lb.; powdered "soap bark," $\frac{3}{4}$ lb.; hydrocarbon "gas oil," 100 galls. The whole is then distilled in any suitable retort, the evolved gas being passed through a superheater, and purified in the usual manner.

The use of the above-named mixture is said to equalise the distillation of the materials, enable the process to be carried on more rapidly and at a lower temperature than would otherwise be possible; and, finally, to ensure the production of a pure gas of high illuminating power at a low cost.—C. S.

*Gas for Illuminating and Heating Purposes; Impts. in the Production of, and of certain Bye-products.* W. Dancer, Old Trafford. Eng. Pat. 20,184, Sept. 2, 1897.

The inventor proposes to manufacture oil gas and ammonia from the fatty (lime soaps) and nitrogenous matters in sewage sludge, by distilling the dried sludge at a high temperature in a gas retort, and completing the decomposition of the oil, by passing the products of distillation through a second retort, filled with glowing coke "or other inert substance," "so that any oil distilling over from the first retort is at once converted into gas on coming into contact with the heated substance in the second retort."

As an alternative method, the sludge may be distilled at a lower temperature, in retorts of the type used for distilling Scotch shale, the oil thus recovered being afterwards gasified in the usual manner. Any gas generated in the still is utilised as fuel, after the recovery of the ammonia.
—C. S.

*Gas, An Improved Apparatus for the Purification of, or other analogous Purpose.* A. E. N. and S. N. Neadon, Leeds. Eng. Pat. 2114, Jan. 31, 1898.

In this apparatus the gas to be purified is introduced at the rear of a revolving horizontal cylinder containing a spiral chamber partly filled with lime, &c., through which the gas passes, and in so doing is deprived of its impurities. The lime, &c. is fed into the front end of the cylinder by a spiral conveyer supplied by a hopper of sufficient height to prevent any escape of gas. Drag chains in the spiral chamber prevent the clogging of the purifying material, which is discharged at the further end into gas-tight conveyers for removal.—C. S.

*Gas, Impts. in Manufacture of.* J. Coyne, Pittsburg (Pa.), U.S.A. Eng. Pat. 7592, March 9, 1898.

The inventor employs two chambers, employed alternately as generating and fixing chambers, in such a manner that there is no loss of gas in blowing up the furnace after the initial heating; consequently the production of gas is continuous.

When the generator is charged with carbonaceous fuel, air is blown in from below, the result being to produce a mixture of carbon monoxide and volatile hydrocarbons, which are then passed into the lower part of the fixing chamber and ascend, in admixture with injected steam, through the incandescent fuel therein. By this treatment carbon monoxide, hydrogen, methane, ethylene, and ammonia are formed, in consequence of the gradually decreasing temperature to which the gases are exposed during their ascent in the fixing chamber. When the charge of fuel in the latter is cooled, the valves are reversed and the chamber acts as a generator, the former generator now serving as a fixing chamber.—C. S.

*Gas, Illuminating and Heating, An Improved.* E. Tooth, Redfern, New South Wales. Eng. Pat. 8555, April 12, 1898.

"An improved illuminating and heating gas, the combustion of which gives a brilliant light and great heat," is yielded, according to the inventor, by the action of water on a mixture of about 40 per cent. by volume of calcium carbide, 50 per cent. of pure unslaked lime, and 5—10 per cent. of "terebine," or "a solution of resin in molten naphthalene," with or without the addition of paraffin as a coating, which "forms not only an efficient protector of the carbide of calcium from damp, but also forms an essential ingredient of the mixture."—C. S.

*Phosphorus and other Materials, Impts. in the Process of Manufacturing.* C. S. Bradley, Avon, New York, and C. B. Jacobs, New Jersey, U.S.A. Eng. Pat. 10,290, May 5, 1898.

*See under* XXII., *page* 872.

*Calcium Carbide, An Improved Method of, and Apparatus for the Production of, applicable also to other Purposes.* [*Polyphase Currents.*] H. Maxim, London, and W. H. Graham, Trowbridge. Eng. Pat. 18,988, Aug. 17, 1897.

The furnace, which is made of firebrick, consists of a long open box, having a large upright carbon slab forming the common electrode fixed along one side, and separated electrodes consisting of six equi-distant circular carbon rods extending into the furnace horizontally in the same plane through the opposite side. The outer end of each electrode is electrically connected, respectively, by a clamp, a cable, or switch, and an ammeter on a switchboard, to a lead or cable of a polyphase generator, the number of whose phases is equal to the number of the electrodes. Between each electrode and the common electrode, a small carbon rod is held by the pressure of a spring, and these rods form incandescent conducting cores on the passage of the current. The furnace is covered with a hood, and over each separate electrode is a hopper to supply fresh material as required, and means are provided for removing the formed calcium carbide at each conductor independently. Any number of electrodes may be employed, and the furnace may be divided up into a number of separated compartments, each of which contains a pair of opposed electrodes, both being adjustable. The electrodes along one side are connected together by a cable, and form a common electrode.—G. H. R.

*Carbide of Calcium, Impts. in the Process and Apparatus for the Production of.* R. P. Pictet, Geneva, Switzerland. Eng. Pat. 21,508, Sept. 20, 1897. (Under Internat. Convent.)

In this process, which is an improvement on that described in Eng. Pat. 9358, 1896 (this Journal, 1896, 582), some substance which has a reducing action at high temperatures, such as coal, pitch, and the like, is mixed with the powdered lime and carbon, with or without the addition of fluxes, and the mixture is fed into a furnace into which air is brought by tuyeres. The oxygen of the air combining with the hydrogen and the carbon heats the substances intensely before their arrival at the electric arc. The furnace is arranged in such a manner that the liquid carbide protects the most exposed portion of the casing, and that the gases formed in the central portion are utilised for heating the outer casing.—G. H. R.

*Acetylene Gas or other Gases, An Improved Apparatus for the Generation of.* E. Barnard, Christchurch, Hants. Eng. Pat. 17,090, July 20, 1897.

A carbide chamber fixed on the top of a gasholder bell contains a cylinder having holes in its base, through each of which a carbide cartridge can pass, when, by the rotation of the cylinder, a hole is brought over a shoot leading to a basket within the tank of the gasholder. The generation of gas causes the bell to rise. When it descends the cylinder is rotated so that a second hole is brought over the shoot. Various devices for causing rotation of the cylinder in correspondence with the movements of the bell, and several modifications of the apparatus, are described.

—J. A. B.

*Acetylene Gas Generators, Impts. in.* N. Reggiani and A. Chrisini, both of Rome. Eng. Pat. 17,482, July 24, 1897.

A generator containing carbide in compartments, which water may enter by overflowing the vertical partition walls in series, is supplied with water from a vessel between which and the generator is a water-seal corresponding in depth to the pressure of gas within the bell gasholder, to which the gas passes as it is generated. When the bell descends, owing to withdrawal of gas, an inverted cone attached to its crown enters the water and causes a diminution of the pressure within the bell, and thus disturbs the equilibrium, and permits water to pass to the generator until by the rise of the bell the pressure is again equalised throughout the apparatus. The pressure within the bell may be reduced by means of two spiral springs instead of by the inverted cones.—J. A. B.

*Acetylene Gas, Impts. in Heating Burners or Stoves for Use with.* F. P. J. Ackermann, Marseilles. Eng. Pat. 19,536, Aug. 24, 1897.

A Bunsen burner, suitable for the combustion of acetylene, and applicable to all heating purposes, is constructed with a single gas orifice or jet protruding into a chamber into which air enters from the atmosphere. A mixing tube, attached to the top of the chamber, is throttled at its lower end, and at its upper end terminates in a jet or a number of short tubes or jets, at which the gas burns with a blue flame. A sheet of wire gauze may be fixed in the tube to prevent lighting back.—J. A. B.

*Acetylene and other Gases, Impts. in Apparatus for the Generation of.* H. R. Bean, Plaistow, Essex, and H. Ringwood, Poplar, Middlesex. Eng. Pat. 21,114, Sept. 14, 1897.

The invention relates to improvements in the method of generating acetylene claimed in Eng. Pat. No. 5756 of 1897 (this Journal, 1897, 527). Cocks are provided in the pipes which convey the gas from several generating chambers to the sliding gasholder, so that the generators may be successively isolated from the gasholder for recharging, while the apparatus is in use.—J. A. B.

*Acetylene Gas, An Improved Gasometer for Generating.* J. Benjamin, Swansea. Eng. Pat. 27,065, Nov. 19, 1897.

A bell gasholder floating in a tank to the sides of which are attached generating cylinders containing baskets of carbide, controls, by means of rods and a cog-crank, cocks on the pipes which admit water to the cylinders from the tank. The generation of gas is thus governed by the movements of the gas-holder.—J. A. B.

*Acetylene Gas, Impts in or connected with the Manufacture and Storage of.* W. Owens, Pontardawe, South Wales. Eng. Pat. 1625, Jan. 20, 1898.

An inclined cylindrical generator receives through its lower end a tray comprising a number of compartments charged with carbide. Water is supplied to the upper end of the generator through a pipe provided with a valve, which is operated by a catch attached to the bell of the gas-holder, which receives the gas from the generator after it has passed through a purifier. A telescoping safety device for the escape of surplus gas from the holder, and an arrangement for causing the water to flow to a second generator when the first is exhausted, are described.

—J. A. B.

*Acetylene Generating Apparatus, Impts. in.* J. S. Goodwin, London. Eng. Pat. 2174, Jan. 27, 1898.

The upper compartment of a vertical cylinder contains water, and the lower compartment a carbide-holder. Water passes by a pipe to the bottom of the lower compartment, and rises therein until it reaches carbide in the carbide-holder, which has a perforated conical lower end. The gas escapes through a lateral perforation in the pipe which rises from the top of the carbide-holder into a central cavity in the upper compartment, and thence through the top of the latter to the burners.—J. A. B.

*Acetylene Gas Apparatus, Impts. in.* A. E. R. Söhnel and A. R. Zehner, both of Hamburg. Eng. Pat. 8030, April 4, 1898.

A water-container above an interchangeable carbide-container communicates with the latter by a perforation in the barrel of an intervening cock, through another perforation in which the gas escapes to a pipe which traverses the water-container. By turning the cock, the communications are interrupted, but a transverse perforation in the cock allows gas subsequently generated to escape to the air.
—J. A. B.

*Acetylene, Impts. in Apparatus for Generating.* C. D. Abel, London. From P. Lipcke, Charlottenburg, Germany. Eng. Pat. 13,387, June 15, 1898.

On the top of a generator, within which is water, is fixed a gas-tight slide containing a compartment which is first charged with carbide, and is then, by rotation of the slide, shut off from the air, and brought over a shoot down which the carbide falls into the water in the generator. Air is expelled from the generator before it is put in action by a stream of acetylene gas from the gasholder.—J. A. B.

*Coke Ovens, Impts. in.* F. J. Collin, Dortmund, Germany. Eng. Pat. 11,439, May 19, 1898.

The inventor claims "coke ovens with horizontal coking chambers, having, at the ends of the heating flues, upright heating chambers, into which the combustibles are introduced either directly from the coking chambers or after being purified, and whence they pass into the heating flues, after entering partially into combustion with an air supply."
—C. S.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Pentachlorethane ($CCl_3.CCl_2H$) and Perchlorethylene ($CCl_2:CCl_2$). Action of, on Benzene in Presence of Aluminium Chloride.* [*Synthesis of Anthracene.*] A. Mouneyrat. Bull. Soc. Chim. 1898, **19**, [13], 557—559.

The author dissolved 150 grms. of pentachlorethane in 1,200 c.c. of benzene, warmed the solution to 70° C., added 150 grms. of aluminium chloride little by little, and heated the liquid until 155 grms. of hydrochloric acid had been collected, *i.e.*, the quantity corresponding to the equation—

$$CCl_3.CCl_2H + 5C_6H_6 = (C_6H_5)_3{:}C.C(C_6H_5)_2H + 5HCl.$$

At this point the mass was thrown into water containing hydrochloric acid, washed with water and potash, dried over calcium chloride and distilled. After the benzene had been removed, the temperature rose rapidly, and between 250° and 370° C. a large quantity of a colorless liquid, which solidified on cooling, distilled. Between 370° and 500° C., a yellow oil passed over, whilst the residue in the flask was an insoluble carbonaceous mass. The substance distilling between 250° and 370° was crystallised from hot acetic acid, from which it was deposited in fine needles melting at 92°, and was identified as triphenylmethane. In this reaction the pentachlorethane must have behaved as chloroform would with benzene—

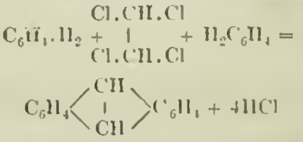
$$CHCl_3 + (C_6H_6)_3 = CH(C_6H_5)_3 + 3HCl$$

and its behaviour is an instance of the variability of the reactions brought about by aluminium chloride.

The product distilling between 370° and 500° was dried on a porous tile, crystallised from a mixture of alcohol and benzene, and purified by sublimation. It melted at 213° C., and was identified as anthracene. The formation of this substance is accounted for as follows. In a former paper (Bull. Soc. Chim. **19—20**, 182) the author showed that under the influence of aluminium chloride, pentachlorethane is converted into perchlorethylene and hydrochloric acid—

$$CCl_3.CCl_2H = HCl + CCl_2{:}CCl_2$$

Friedel and Crafts showed in their synthetical experiments that compounds with a double ethylene bond can absorb two atoms of hydrogen. In the present instance, therefore, $CCl_2{:}CCl_2$ would combine with two atoms of hydrogen to form $CHCl_2.CHCl_2$, which, acting on benzene under the conditions of the experiment, would yield anthracene—



$$C_6H_4.H_2 + \begin{matrix} Cl.CH.Cl \\ | \\ Cl.CH.Cl \end{matrix} + H_2C_6H_4 = C_6H_4\left\langle\begin{matrix} CH \\ | \\ CH \end{matrix}\right\rangle C_6H_4 + 4HCl$$

This hypothesis was verified by treating the product formed by the action of perchlorethylene on benzene in the presence of aluminium chloride, in the same way as that described above. A large quantity of anthracene was thus obtained.—C. A. M.

### PATENT.

*Distillation of Petroleum and other Oil, and in Apparatus therefor; Impts. in the.* T. C. Palmer, London. Eng. Pat. 21,115, Sept. 14, 1897.

The patentee claims the employment of superheated steam for distilling crude petroleum oil, the operation being conducted in a vacuum. A further claim comprises the still, "catch-all," vacuum pump, condensers, and "fore-warmer," drawings of which accompany the specification, together with their combination, arrangement, and method of working.—T. A. L.

## IV.—COLOURING MATTERS AND DYES.

*Saffron, Adulteration of.* J. Wauters. Bull. Assoc. Belge des Chim. **12**, [3], 103—107.

A preparation known as "féminelle" is employed, both in a crude condition and also mixed with colour for the adulteration of saffron. The following table gives the results of analyses, made by the author, of samples of pure and adulterated saffron, as also of the adulterant and colour in question :—

| — | Feminelle. | | Colouring Matter. | Saffron. | | | | |
|---|---|---|---|---|---|---|---|---|
| | Crude. | Prepared. | | Pure. | Whole. T. | Powdered. T. | Whole. F. | Powdered. F. |
| Moisture | 10·85 | 22·15 | .. | 9·35 | 8·90 | 6·20 | 13·35 | 6·05 |
| Total ash | 8·25 | 13·05 | 81·75 | 6·13 | 21·02 | 21·00 | 10·45 | 15·35 |
| Ash insoluble in water | 2·23 | 3·40 | 0·10 | 2·45 | 16·83 | 1·00 | 3·75 | 0·70 |
| " " " HCl | 1·55 | 0·98 | 0·05 | 1·50 | 15·18 | 0·25 | 0·90 | 0·20 |
| Substances insoluble in water | .. | .. | .. | 36·50 | 46·70 | 54·60 | 38·15 | 66·60 |
| Crocetine | .. | .. | .. | 6·55 | 2·60 | 1·60 | 1·05 | 1·95 |
| Chlorine (as NaCl) | 0·23 | 7·44 | 78·00 | 0·23 | Traces | 16·38 | 1·22 | 11·11 |

The colouring matter—an orange-yellow powder—consists of an amido-azo sulphonated coal-tar compound, itself adulterated with 75 per cent. of sodium chloride. The colour of the aqueous solution exactly resembles that of pure saffron, and does not alter under the influence of hydrochloric acid at any temperature, though decolorised when treated by this acid in presence of tin. Caustic potash or ammonia causes it to become deep orange-red, and in presence of the latter reagent it gives up but a very small quantity of colouring matter to amyl alcohol. Baryta water renders the substance insoluble in acetic ether. Dissolved in water and mixed with tartaric acid, this dyestuff stains wool a red-brown, and silk a fine orange-yellow, whilst cotton takes only a pale yellow tinge.

The crude "féminelle" consists of the flowers and ligulæ of the marigold, an old adulterant for saffron, together with a colouring matter which in presence of tartaric acid dyes wool an orange-red, and silk, a rose colour, but is not fixed by cotton.

When prepared by impregnation with the artificial colour already mentioned, the "féminelle" furnishes the same result in test dyeings. The amount of colouring matter fixed, can be approximately determined from the sodium chloride in the ash.

The determination of crocetine affords an indication of the degree of adulteration practised in any case, assistance being also furnished by microscopical examination.

The dyeing test gives very clear indications, a solution of pure saffron imparting, in presence of tartaric acid, a fine pale citron-yellow shade to both wool, silk, and cotton, the colour being unchanged by subsequent exposure to caustic potash; whereas with adulterated saffron, the dyed colours are different and are altered by alkali, as already stated. The reaction is sufficiently delicate to detect 5 per cent. of féminelle. Again, the solution left when the crocetine is thrown down by hydrochloric acid is, in the case of pure saffron, of a very pale yellow colour, and only capable of very slightly staining wool or silk, whilst the brown liquid resulting in the case of adulterated saffron has greater tinctorial properties, and is also changed by an addition of caustic alkali.

The samples in powder, T and F, represent mixtures of féminelle and 25—30 per cent. of saffron; the whole sample F is rather richer in saffron, whilst the whole sample T, though free from NaCl and therefore from the artificial colour in question, contained 14·12 per cent. of $BaSO_4$, of the "pure saffron" employed.—C. S.

### *Myrticolorin, Notes on.* H. G. Smith. J. and Proc. Roy. Soc. of N. S. Wales, **31**, 377—380.

The author communicates further particulars of the dyestuff "myrticolorin," which he obtained from the dried leaves of the *Eucalyptus macrorhyncha*, known as the "Red Stringy Bark," which abounds in New South Wales and Victoria (J. and Proc. Roy. Soc. of N. S. Wales, 1897, Aug. 4).

Myrticolorin breaks up, on boiling with dilute sulphuric acid, into a body which was recognised as quercetin by means of the acetyl derivative and its other reactions, and into a sugar or sugars of the glucose type, which reduces Fehling's solution on heating, is readily fermentable by yeast, and crystallises partially from water in minute prisms, probably monoclinic. Hence it is concluded that myrticolorin is a glucoside of quercetin, and this view is shared by A. G. Perkin, who has also examined a sample.

The species of tree which yields this dyestuff is widely distributed, and the leaves are very readily obtainable, whilst the process of manufacture is of the simplest description. The dried leaves of *E. macrorhyncha* contain no less then 10 per cent. of myrticolorin, which crystallises with 7 per cent. (probably 3 molecules) of water, and yields 48 to 50 per cent. of quercetin, on which its value as a dye depends.

The fresh leaves are carefully dried under conditions which prevent any heating or fermentation, and then ground as finely as possible; the powder is boiled with water and the extract filtered hot through cloth. The yellow myrticolorin crystallises out on cooling and is filtered off, washed, and dried. The dyestuff is not very soluble, even in boiling water, and a satisfactory extraction can only be obtained by grinding the leaves to an extremely fine powder. Three extractions are generally found to be sufficient.

The collection of the eucalyptus leaves and the manufacture of myrticolorin can easily be conducted on a small scale. The dried product keeps well and can be accumulated for export. The industry is thus eminently suitable for the conditions prevailing in the colonies. No skill is necessary, and the only capital and plant required are a mill for grinding the leaves, and a few ordinary vessels, which must not be iron ones.

Quercitron bark is estimated by Perkin to contain about 2 or 3 per cent. of quercetin, and its value in England is about 6*l.* 10*s.* per ton, and that of fustic 4*l.* 10*s.* per ton.

When it is considered that the dried leaves of the above species of eucalyptus contain 5 per cent. of quercetin, and that the process of grinding and extracting the leaves is so much simpler than working up bark, it will easily be recognised that this discovery is likely to be of great commercial importance to the Australian colonies.

—J. F. B.

### *Aniline, The Discovery of.* E. Knecht. J. Soc. Dyers and Colourists, **14**, [8], 162—163.

The discovery of aniline is usually attributed to Unverdorben, who obtained it in 1826 as one of the products of the distillation of indigo (Pogg. Annalen, Vol. VIII., p. 397). However, the author, in looking up the English translation, published in 1789, of the work of Hellot, "*L'Art de la Teinture des Laines*," published in 1750, finds among his remarks anent the theory of indigo dyeing, the following passages:—

"If indigo be distilled in a retort, alone, or with the addition of a fixed alkali, either saline or terreous, you will thereby procure a liquor which in all chemical experiments produces the effect of the volatile spirit of urine."

"The sal-ammoniac from which the chemists obtain their most penetrating volatile spirit, has not, when dissolved and boiled in water, this strong urinous smell."

This evidence that Hellot detected in indigo a product distinct from ammonia, is, in the author's opinion, sufficient to justify the assumption that he was the discoverer of aniline.—J. E. H.

### *Indigo, Action of Aniline on; Note on the.* E. Knecht and G. A. Parkes. J. Soc. Dyers and Colourists, **14**, [8], 163.

It has been pointed out by Brylinski that aniline has a very destructive action on indigo (this Journal, 1898, 609). Thus, if indigo be boiled from 4—16 hours with aniline, a loss occurs, varying from 32—43 per cent.

The authors have carried out a series of experiments to determine what becomes of the indigo when acted upon by aniline. The following mode of procedure was adopted. The finely divided indigo was boiled for a prolonged period with pure, freshly distilled aniline oil. After filtering and washing the residue with boiling water, it was extracted with strong hydrochloric acid and again filtered. A dark brown filtrate was obtained, which, on being rendered alkaline with caustic soda, yielded a copious precipitate. This was filtered, washed, dried, and crystallised from alcohol. The crystals thus obtained were small, of a bright yellow colour, and melted at about 300° C. They were insoluble in water, but soluble in alcohol, ether, and benzene. They possessed a basic character.—J. E. H.

### *Tetramethyldiamidobenzophenone, Some Derivatives of.* E. Grimaux. Bull. Soc. Chim. 1898, **19**, [14], 608—610.

The dinitro-derivative of this ketone—

$$CO[C_6H_3(NO_2).N(CH_3)_2]_2,$$

is obtained by the addition of potassium nitrate to a solution of the ketone in sulphuric acid, pouring into water, addition of soda, and recrystallisation of the precipitate from acetone. The crystals melt at 165°—166° C., and resemble ammonium bichromate in appearance. Reduction with stannous chloride and hydrochloric acid furnishes an uncrystallisable base melting at 66°—69°. The base yields a crystalline picrate.

The dibromo-derivative is formed by the action of 2 mols. of bromine on the chloroform solution of the ketone. After evaporation of the solvent, the residue is dissolved in hydrochloric acid (1:1), precipitated by ammonia, and the precipitate recrystallised from alcohol. It forms hard shining prisms melting at 130°—131° C.

New colouring matters are obtained by the condensation of the dibromo- and dinitro-derivatives with dimethylaniline and phenyl-α-naphthylamine by the action of phosphorus chlorides. They are duller than the corresponding non-substituted colours, but appear to have more resistance to soap.—A. C. W.

### *Nitro- and Amido-Naphthonitriles and Naphthoic Acids.*

H. Heilpern and M. Spielfogel. Mitt. des tech. Gewerbe Museums in Wien, 1898, **8**, [11 and 12], 316—323.

Two methods are described in the paper for the production of these compounds, the one by converting the nitronaphthylamines into nitronaphthonitriles (by means of the Sandmeyer reaction), which then on saponification yield the acid; whilst the other method consists in distilling certain naphthylamine sulphonic acids with potassium cyanide, when they are converted into amidonaphthonitriles, from which the amidonaphthoic acids can be obtained. An improved method is given for nitrating β-acetnaphthalide:—90 grms. of finely powdered β-acetnaphthalide suspended in 120 grms. of acetic acid are treated with 70 grms. of nitric acid (sp. gr. 1·4) at about 30°—40° C. The crystalline magma is washed with 59 per cent. acetic acid. In order to purify the crude product, which melts at 101°—102° C., it is boiled with three times its weight of benzene, and filtered warm, when yellow crystals are deposited from the filtrate which melt at 119°—120° C., and consist of almost pure 1.2-nitro-acetnaphthalide. The residue is extracted with xylene and the insoluble portion is crystallised from acetone or chloroform, when greenish-yellow crystals are obtained melting at 195° C. and having the constitution of a 1.2′-nitro-acetnaphthalide. On boiling the latter with dilute sulphuric acid, (1 : 1), nitronaphthylamine melting at 104° C. is obtained.

The 1.2-nitro-acetnaphthalide is hydrolysed with alcoholic potash and the 1.2-nitronaphthylamine is diazotised in an alkaline solution which, after diluting and filtering, is added to a potassium copper cyanide solution and gently warmed until the evolution of nitrogen ceases. The precipitate is then steam-distilled, when brownish needles of the nitrile pass over, melting at 101° C. and easily soluble in alcohol, benzene, glacial acetic acid, and acetone. The conversion into nitronaphthoic acid is best effected by heating with baryta water, the solution being precipitated by an acid. The 1.2-nitronaphthoic acid crystallises from chloroform in red needles melting at 182° C.

1.3′-*nitronaphthonitrile* is obtained by grinding together 20 grms. of 1.3′-nitronaphthylamine, melting at 143·5° C., with 10 grms. of concentrated sulphuric acid and diazotising with 8 grms. of sodium nitrite in solution. The filtrate is poured into a solution of 25 grms. of copper sulphate, 30 grms. of potassium cyanide, and 150 c.c. of water. When the evolution of nitrogen has ceased, the precipitate is extracted with 25 per cent. acetic acid, when the nitrile separates out in yellowish-brown needles melting at 168° C. When heated with 3—4 times its weight of sulphuric acid (1:1) to 140°—150° C., it yields a brownish-yellow product melting at 263° C., probably the amide of the nitronaphthoic acid, since it is insoluble in alkalis. When boiled with 10 per cent. caustic soda it dissolves, and is precipitated from the filtrate in a mass of thin white needles crystallising from alcohol in yellowish-white needles melting at 287° C.

In a similar manner, corresponding derivatives were obtained from 1.2′-nitronaphthylamine melting at 103° C. The nitrile crystallises in light brown needles melting at 143° C., the amide obtained by hydrolysis with sulphuric acid, melts at 218° C., whilst the nitronaphthoic acid melts at 295° C.

With regard to the alternative method above alluded to, 1.4′-naphthylamine sulphonic acid is converted into the sodium salt, mixed with potassium ferrocyanide, and distilled in portions of 50 grms. from a glass retort. The cyanide forms light greenish-yellow needles melting at 137° C., the yield being 18—20 grms. from 100 grms. of sulphonic acid. For the saponification, the finely powdered cyanide is boiled with six times its weight of sulphuric acid ($H_2SO_4 : H_2O = 66 : 34$) until it gives a clear solution when exactly neutralised with ammonia. When this is the case, the amidonaphthoic acid is precipitated from the neutralised solution by acetic acid, and melts at 211° C. A hydrochloric acid solution of this is mixed with a *p*-nitrodiazobenzene chloride solution. The resulting diazo compound, which gives a violet-red with R-salt or with β-naphthol, is boiled with the addition of some urea. On filtering, the oxynaphthoic acid crystallises, melting at 219° C. It gives a deep red when combined with diazonaphthionic acid, which decomposes on boiling or on long standing. It also forms an ethyl ether melting at 73° C.

In a similar manner 2.2′-naphthylamine sulphonic acid gives a nitrile crystallising from toluene in light yellow needles and melting at 171° C., an amido-naphthoic acid melting at 245° C., and an oxynaphthoic acid melting at 262° C., which combines with diazonaphthionic acid to form a red, which is not decomposed on boiling. A very poor yield of 1.2′-naphthylamine nitrile, melting at 117° C., is obtained, much α-naphthylamine being formed during the dry distillation.—T. A. L.

### 1.4.2- *and* 2.3.3′-*Diamidonaphthalene Sulphonic Acid, and* 2.3.3′-*Amidonaphthol Sulphonic Acid.*

M. Oesterreich. Mitt. des tech. Gewerbe Museums in Wien, **8**, 1898, [11 and 12], 312—315.

The preparation of the first-named acid is effected by combining the ammonium salt of 1.2-naphthylamine sulphonic acid in presence of sodium acetate with diazobenzene chloride, and reducing the dyestuff obtained with stannous chloride. The acid separates as a crystalline precipitate, and is purified by dissolving in sodium acetate and bisulphite, treating with animal charcoal, and precipitating with dilute hydrochloric acid. The acid is very slightly soluble in cold water, and the aqueous solution is coloured dark green by ferric chloride, a blue precipitate appearing on standing, whilst, on warming, the solution turns cherry-red. On allowing a solution of the ammonium salt to stand in the air, it acquires an intense green fluorescence. The acid can only be diazotised with difficulty, and only one amido group appears to react, the diazo compound also being very unstable.

2.3.3′-*Diamidonaphthalene Sulphonic Acid.*—This acid is obtained by heating 2.3.3′-dihydroxynaphthalene sulphonic acid with 2—3 parts of concentrated ammonia to 200°—220° C. The free acid is very sparingly soluble in boiling water. The sodium and potassium salts crystallise from hot water, in which they dissolve with a blue fluorescence, in colourless plates. Ferric chloride gives a brown coloration in the cold, whilst a brown precipitate is formed on warming. The acid possesses all the characteristics of an *o*-diamine. It yields a very sparingly soluble condensation product with phenanthraquinone, and gives an azimido sulphonic acid with nitrous acid. It combines in a neutral or sodium acetate solution with one molecular proportion of a diazo compound. The combination with diazobenzene is cherry-red, that with tetrazodiphenyl is brownish-red, and this latter dyestuff, when diazotised on the fibre, gives a dark Bordeaux brown when developed with β-naphthol.

2.3.3′-*Amidonaphthol Sulphonic Acid.*—By moderating the action of ammonia on dihydroxynaphthalene sulphonic acid, it is possible to replace only one hydroxyl group. The acid so obtained is most probably identical with that produced by fusing β-naphthylamine disulphonic acid R with caustic soda. It was obtained by heating 25 grms. of dihydroxynaphthalene sulphonic acid R with 50 grms. of concentrated ammonia in a pressure tube for 12—14 hours to 130°—150° C. After driving off the ammonia, the acid is precipitated with hydrochloric acid, and purified by the same treatment. It forms colourless needles, sparingly soluble in hot water. The solution turns blackish-blue on the addition of ferric chloride, whilst bleaching powder yields a dark brown coloration. The diazo compound is yellow, turns bluish-violet with sodium carbonate, and gives a cherry-red with R salt. The ammonium salt is easily soluble.—T. A. L.

### *Dinitro-α-Naphthol-1′-Sulphonic Acid.* J. Karpeles. Mitt. des tech. Gewerbe Museums in Wien, 1898, **8**, [11 and 12], 309—311.

This dyestuff, which is isomeric with Naphthol Yellow S, is at the present time no longer on the market, since the shade it dyes is very much more orange than that given by the latter. It has the constitution—

$$1.2.4.1'.C_{10}H_4.OH(NO_2)_2SO_3H,$$

since it is produced by the action of nitric acid on 1.1′-naphthol sulphonic acid, or on 1.4.1′-naphthol disulphonic acid, and is obtained by stirring 250 grms. of finely powdered naphthalene into 500 grms. of sulphuric acid, warmed to 60° C., the temperature then being raised to 80° C., and maintained thereat for 1½ hours. The melt is cooled to 10° C., and nitrated, at a temperature below 20° C., with 190 grms. of concentrated nitric acid mixed with the same volume of sulphuric acid. After standing some hours it is poured on to ice, filtered from nitronaphthalene, diluted, treated with about 50 grms. of ferrous sulphate to remove nitric acid, boiled up, and reduced with iron borings. The solution decanted from the unacted-on iron, deposits the amidonaphthalene sulphonic acid on cooling. The crude product is dissolved in ammonia, and filtered into dilute sulphuric acid (1 : 4). The acid so purified is diazotised and boiled, when the naphthosultone separates as a brown precipitate. One part of this substance is warmed with three times its weight of concentrated sulphuric acid on the water-bath until soluble in water, when the disulphonic acid formed is precipitated with brine and boiled with concentrated soda-lye. After cooling, the requisite amount of nitrite is added, and the whole is boiled with concentrated nitric acid. The dyestuff begins to separate during this operation, and is completely precipitated in the cold. It crystallises in needles, deflagrates, tastes bitter, and has all the properties of a nitro-compound. It dyes wool evenly a reddish-yellow, but the presence of copper must be avoided. As already mentioned, it has no technical value, since the shade it gives can be obtained in a variety of other ways.—T. A. L.

### *2.4.1′-Naphthylaminedisulphonic Acid.* A. Fischer. Mitt. des tech. Gewerbe Museums in Wien, 1898, **8**, [11 and 12], 324—325.

This new acid is obtained by suspending 28 kilos. of finely powdered 1.4′-naphthalene disulphonic acid in 90 kilos. of concentrated sulphuric acid, and adding to the well-cooled mixture (at about 0° C.), 10 kilos. of nitric acid (90° B.) and 10 kilos. of sulphuric acid. The melt is then poured on to ice, and 40 kilos. of sodium carbonate are added, when the acid sodium salt of the β-nitronaphthalene disulphonic acid C separates out, and on reduction yields the acid sodium salt of β-naphthylamine disulphonic acid C. The salt is easily soluble in hot water, and can be precipitated by salt. The alkaline solution shows a bluish-violet fluorescence. The diazo compound forms microscopic yellowish needles, easily soluble in water, but precipitated by salt. The free acid can be obtained by decomposing the barium salt with dilute sulphuric acid. The constitution of the acid was determined by boiling its diazo compound with water, when β-naphthol disulphonic acid was formed, which, on splitting off the sulphonic acid groups, yielded β-naphthol. Moreover, on boiling the diazo compound with alcohol, it yields 1.4′-naphthalene disulphonic acid, and when the calcium salt is distilled, considerable quantities of β-naphthylamine are produced.—T. A. L.

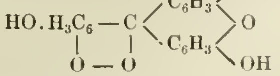
### *Paranitrotoluene Sulphonic Acid. Oxidation of.* A. G. Green and A. R. Wahl. Ber. **31**, [8], 1078—1080.

In reply to Ris and Simon (Ber. **31**, 354; this Journal, 1898, 448), the authors point out that in describing the dinitrodibenzyldisulphonic and dinitrostilbene disulphonic acids, they had naturally convinced themselves, by combustion on platinum foil, that their products left no ash, and hence were the free acids, and not the acid sodium salts. They state further that it is possible to obtain, by decomposing the sodium salt, either the free acid or the acid sodium salt, according to the conditions existing in the solution. Thus the acid sodium salt usually separates from tolerably concentrated solutions, and the acid from dilute ones. Moderately concentrated solutions, when slowly cooled, deposit the acid sodium salt, but the free acid when rapidly cooled. For example, 10 grms. of the disodium salt of dinitrostilbene disulphonic acid were dissolved in 200 c.c. of boiling water and treated with 400 c.c. of hydrochloric acid (27 per cent. HCl). One-half was allowed to cool slowly, the other being cooled quickly and the crystals filtered off immediately. The former gave a precipitate of yellowish needles; the latter, white mica-like plates, which were much more soluble than the needles. On leaving the plates of the free acid for some time in contact with the mother-liquor, they are converted into the needles of the acid sodium salt.

The suggestion that the dinitrostilbene disulphonic acid (*loc. cit.*) was in reality a nitronitroso compound, has, apart from its improbability, been disproved by a quantitative reduction method with zinc dust and sal-ammoniac, the excess of zinc being titrated with ferric sulphate. The numbers obtained prove conclusively that the dinitrostilbene disulphonic acid has the formula $C_{14}H_{10}N_2S_2O_{10}$. The method employed is as follows:—About 2 grms. of sal-ammoniac and some water are placed in a small flask provided with an india-rubber stopper and a Bunsen valve, together with a weighed quantity of zinc dust, about 4 grms. (86 per cent.), and 3—4 grms. of the nitro compound. The whole is shaken in the cold for about half an hour, then raised to the boil, and kept boiling until completely reduced. The zinc and zinc oxide are allowed to settle, washed by decantation, 10 grms. of ferric sulphate are added together with some water, when the zinc remaining dissolves, reducing part of the ferric to ferrous sulphate. After acidifying with sulphuric acid, the solution is made up to 500 c.c., and an aliquot part is titrated with decinormal permanganate, from which the amount of zinc left after the reduction can be readily calculated.—T. A. L.

### *Dyestuff from Oxalic Acid and Resorcinol.* G. v. Georgievics. Mitt. des tech. Gewerbe-Museums in Wien, 1898 **8**, [11 and 12], 365—370.

The condensation of resorcinol with oxalic acid has already been the subject of research, although previous investigators (Claus, Ber. **10** and **14**; Gukassianz, Ber. **11**) have conducted the operation under pressure at a high temperature or have employed a condensing agent. The author finds that when one part of resorcinol is heated with twice its weight of anhydrous oxalic acid for three hours to 150°—160° C., condensation readily takes place, the reaction commencing at about 140° C. This may be of interest technically, although the product so obtained has at present no value. After purification a yellow powder is obtained, soluble in concentrated sulphuric acid with an indigo-blue colour (green when impure), and in alkalis with a brown non-fluorescent colour. The dyestuff is readily soluble in cold alcohol, and very slightly soluble in water. It possesses only a slight affinity for animal fibres, giving a dull yellow shade. The formula of the substance is $C_{19}H_{12}O_6$, and, on heating with acetic anhydride, two products are obtained, which can be separated by their solubility in alcohol, the more easily soluble one melting at 182° C. On treatment with bromine an eosine-like substance is obtained, which dyes silk bluish-pink. When distilled with zinc dust, methylenediphenylene oxide is produced, whilst heating with dilute caustic soda-lye yields resorcinol, together with a brown amorphous substance having the empirical composition of a dihydroxyxanthone. The dyestuff may possibly have the constitution—



thus appearing as a triphenylmethane derivative.

In support of this view the author refers to the formation of dyestuffs by condensing dialkylated *m*-amidophenol with oxalic acid, which are not identical with, but are similar to, the pyronine derivatives obtained by Meyenburg (Ber. 29, 509), by condensing dialkylated *m*-amidophenol with phosgene, and described as triphenylmethane derivatives. By heating a dialkylated-*m*-amidophenol with twice its weight of anhydrous oxalic acid in a sealed tube to 140° C. for 2—3 hours, a product is obtained in a yield of 30 per cent. which dyes silk a Magenta shade, whilst at a higher temperature Rhodamine shades are produced.

The product obtained by the author differs distinctly from those described by Claus and Gukassianz. That of the former in an impure state dissolves in concentrated sulphuric acid with a green colour and brownish yellow when pure. The alkaline solution has a faint yellow fluorescence. It also differs from Nencki's Resaurin. The dyestuff obtained in small quantity by Gukassianz, which is soluble in concentrated sulphuric acid with a green colour, and in alkali with an intense green fluorescence, stands nearest to fluorescein of all the dyestuffs formed from resorcinol and oxalic acid. A better yield is obtained by condensing at 150° C. with zinc chloride instead of sulphuric acid, the substance also appearing as a by-product in that described by the author. When treated with hot alcohol it forms sparingly soluble clots, but when ground with cold alcohol it readily dissolves.—T. A. L.

## PATENTS.

*Aromatic Aldehydes, A New Process for Producing.* H. E. Newton, London. From Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 19,204, Aug. 19, 1897.

The process for the production of aromatic aldehydes is claimed, which consists in acting on hydrocarbons, phenol ethers, &c., with hydrochloric and hydrocyanic acids in the presence of aluminium chloride, and decomposing the compounds (aldimides) produced, by means of acids. In the reaction the mixture of hydrocyanic and hydrochloric acids acts as if it were the hypothetical imidoformyl chloride—

$$C_6H_6 + NH : CHCl = C_6H_5.CH : NH + HCl;$$
$$C_6H_5.CH : NH + H_2O = NH_3 + C_6H_5.CHO.$$

In the examples quoted, the gaseous acids are allowed to act on a mixture of the hydrocarbon and aluminium chloride for several hours at 35—40° C.—A. C. W.

*Aldehydo-naphtholsulphonic, Aldehydo-naphtholcarboxylic and Aldehydo-naphtholsulphocarboxylic Acids, and of New Blue and Green Colouring Matters or Dyes therefrom; Manufacture of.* T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 19,498, Aug. 24, 1897.

On boiling naphthol sulphonic, carboxylic, or sulphocarboxylic acids with chloroform and caustic soda (Reimer's method), an aldehyde group enters the naphthalene ring, and the resulting compound differs from the original in giving intensely red and yellow compounds with monamines and diamines in acid solution. Moreover, by condensation with mono- and dialkylated or benzylated aromatic amines and oxidation of the leuco sulphonic acids in acid or alkaline solutions, green and blue acid dyestuffs are obtained, fast to alkalis and belonging to the diphenylnaphthylmethane series. The following is an example of the formation of one of the aldehydes:—A solution of 20 kilos. of β-naphthol sodium disulphonate (R salt) of 87 per cent. in 170 litres of water, is freed from sulphuric acid by adding barium chloride. The filtered solution is boiled with 30 kilos. of caustic soda (45° B.) and 10 kilos. of chloroform in an enamelled vessel provided with a reflux condenser. When the reaction is complete, 50 litres of water are added, the solution is acidulated with hydrochloric acid, and a solution of 10 kilos. of barium chloride in 50 litres of water, is added. The barium salt of the aldehyde-β-naphtholdisulphonic acid R separates out as a yellow powder, and is filtered off, being scarcely soluble even in hot water. When boiled with a solution of sodium sulphate it forms the sodium salt which is readily soluble in water. The following is a typical example of the formation of a dyestuff:—47 kilos. of finely-ground 2.1.3.3′-oxynaphthaldehyde barium disulphonate, 40 kilos. of diethylaniline, 40 kilos. of alcohol, and 15 kilos. of concentrated sulphuric acid are heated in an enamelled vessel provided with a reflux condenser and an agitator, for 30 hours. The melt is then poured into dilute hydrochloric acid, to remove the excess of diethylaniline. The precipitate is washed with dilute hydrochloric acid and boiled with sodium carbonate solution. The filtrate from the barium carbonate is made up to 1,900 litres, and oxidised at a temperature of 10° C. with a 1 per cent. solution of potassium permanganate as long as the formation of dyestuff takes place. After filtering, the dyestuff is precipitated with salt. It forms a crystalline coppery powder, readily soluble in water, unchanged by ammonia or caustic alkalis, but turning green with acids, and dyeing wool green from an acid bath.—T. A. L.

*Dyestuffs directly Dyeing Cotton Black, Manufacture of.* O. Imray, London. From The Farbwerke vorm. Meister, Lucius, and Brüning, Hoechst a/Main, Germany. Eng. Pat. 20,126, Sept. 1, 1897.

By fusing 1.2.4- or 1.2.6-dinitraniline or 1.2.4-nitro-*o*-phenylene diamine, with sodium sulphide and sulphur, a product is obtained which dissolves in water with a dirty green colour, and dyes cotton black from a cold or boiling bath with sodium sulphide and with salt and soda. A mixture of 18 kilos. of sodium sulphide and 7 kilos. of sulphur is heated to 150° C. in an iron vessel, when 6 kilos. of 1.2.4-dinitraniline are gradually added with constant agitation, the temperature being kept for four hours at about 180°—190° C. The melt is then taken out, broken up, and roasted in a flat pan at 230°—240° C. for four hours, when a black porous metallic mass remains, which has the properties mentioned above.—T. A. L.

*Alkyl-hydroxy-p-amidobenzenes, Production of New Derivatives of.* W. H. Claus, A. Rée, and L. Marchlewski, Manchester. Eng. Pat. 20,333, Sept. 4, 1897.

The carbohydrates which contain an aldehyde group, unite in alcoholic solution with *p*-phenetidine and *p*-anisidine on heating on the water-bath for several hours. The process is claimed, and the following compounds as new products:—Gluco-*p*-phenetidide, gluco-*p*-anisidide, galacto-*p*-phenetidide, galacto-*p*-anisidide.—A. C. W.

*Colouring Matters [Blue] derived from Anthraquinone, The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 20,649, Sept. 8, 1897.

In a previous specification (Eng. Pat. 19,622 of 1897; this Journal, 1898, 755) the patentees mention that the reduction of dinitro-anthrarufin and of dinitro-chrysazin disulphonic acids takes place in two stages, and in the present specification details are given for the separation of the intermediate products, which are hydroxylamine compounds, since on further reduction they are converted into the diamido compounds previously described, whilst on oxidation they yield the original nitro products. To a solution of 5 kilos. of sodium *p*-dinitro-anthrarufin disulphonate in 250 litres of water is added a mixture of 8 kilos. of stannous chloride, 30 litres of water, and 30 litres of concentrated hydrochloric acid. The resulting mixture turns blue, and is heated to 50°—60° C., an excess of a concentrated solution of ammonium chloride being subsequently added. On cooling, the ammonium salt of *p*-dihydroxylamine-anthrarufin disulphonic acid separates in small needles, and is filtered off, dried, and powdered. It dissolves in water with a blue colour, changing to bluish-green on addition of ammonia. Concentrated sulphuric acid dissolves it with a yellow colour. The dyestuff gives reddish-blue shades on unmordanted wool from an acid bath and bluish-green shades on a chromium mordant. In a similar manner, ammonium *p*-dihydroxylamine-chrysazin disulphonate dissolves to a violet solution in water, turning green on the addition of soda-lye. This product dyes unmordanted wool blue shades from an acid bath and gives bluish-green shades on a chromium mordant.—T. A. L.

# V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Cotton having a Silky Gloss, Appearance under the Microscope of.* H. Lange. Färber-Zeit. 1898, **9**, [13], 197—199.

The author has already described the production of a silky gloss on cotton, by mercerisation with or without tension (this Journal, 1896, 898).

The appearance under the microscope has now been examined, and the following photographic figures show some of the peculiarities:—

Figs. 1 and 2 show sections of ordinary unmercerised cotton. In longitudinal section it has the appearance of spirally twisted bands, with slightly thickened or rounded edges. In cross section it presents ear-like forms with small slits in the centre.

Fig. 1.

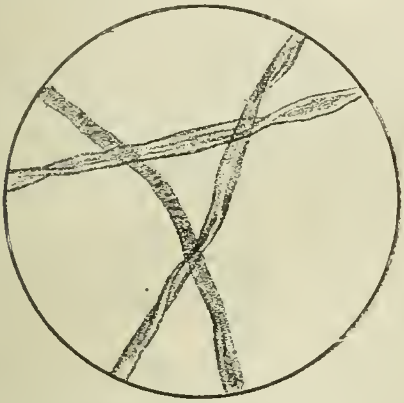


Fig. 2.

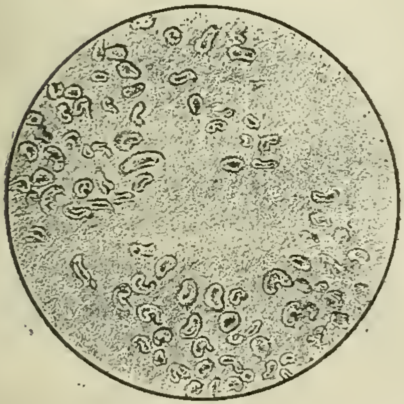
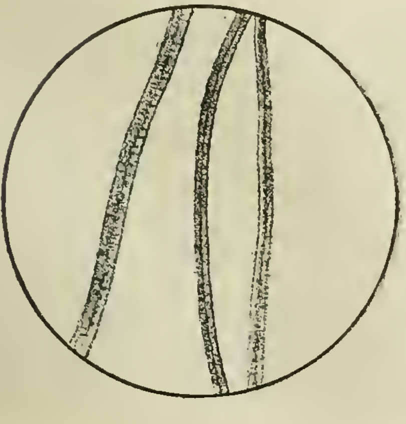
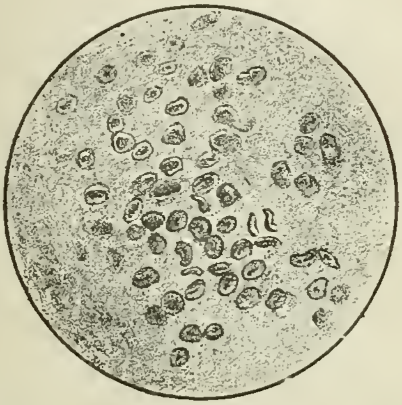


Fig. 3.



Figs. 3 and 4 show sections of cotton mercerised without tension. The cross section shows very thick cell-walls and faint radial lines from the central opening.

Fig. 4.



Figs. 5 and 6 show sections after mercerisation with tension. The fibre has now a smooth rod-like appearance; in cross section it appears round with a more or less distinct opening in the centre. These slight changes in structure of the mercerised cotton probably enhance its appearance and dyeing properties (see also this Journal, 1898, 43, 344, 573, and 658).—C. M.

Fig. 5.

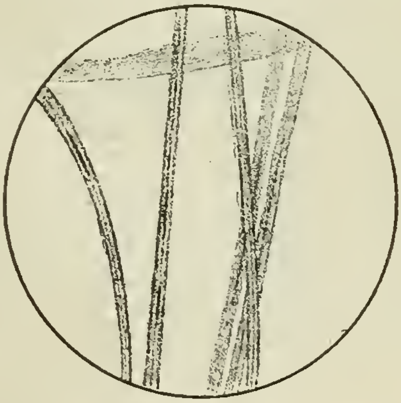
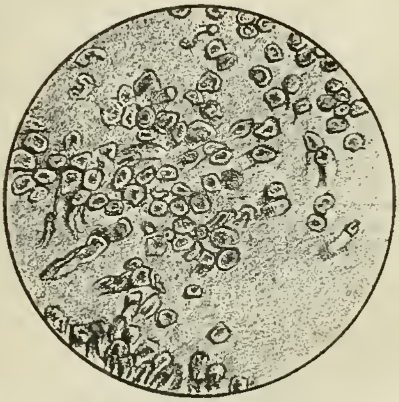


Fig. 6.



*Silk Lustre on Cotton.* A. Fraenkel and P. Friedlaender. Mitt. des tech. Gewerbe-Museums in Wien, 1898, **8**, [11 and 12], 326—333.

According to our present knowledge, when cotton is mercerised, combination takes place between the cellulose and the alkali in the proportion of $C_{12}H_{20}O_{10}$ : 2NaOH, together with water. On washing with water the alkali is split off and the cellulose appears as the hydrate $C_{12}H_{20}O_{10}$, $H_2O$. This effect is only produced with strong lye and in the cold; in fact, Mercer states that boiling soda lye does not produce a similar result. The physical change which takes place in the fibre, converting it from a flat shape into a straight cylindrical form, is probably due to the change in volume which accompanies the conversion of the cellulose into its sodium compound. This, however, does not in any way explain Thomas and Prevost's observation that cotton when mercerised under tension, has a distinctly different appearance. The authors consider that the silky lustre produced is due to the removal of the cuticle from the fibre, and that this is partly a physical and partly a chemical process. They conclude,—and here they have the support of the botanists,—that the cuticle is a cork-like substance, although they could not confirm Flückiger and Gilson's work with regard to certain colour reactions (J. Chem. Soc. 1891, A. 465). This investigator isolated from cork, phellonic acid, glycerin, phloionic acid, and suberic acid, by treating finely-rasped cork first with sodium carbonate, and subsequently with 3 per cent. potash lye at the boil for 45 minutes, this solution on cooling depositing a precipitate of potassium phellonate. When fine cotton yarn is treated in the same way, a similar result is produced, but the colour reactions which Gilson stated were given by phellonic acid were not obtained; and this was also true of purified phellonic acid procured from another source. Practically, it would appear that the cuticle is partially dissolved by the alkali lye, but that the greater part is split off by the swelling up of the fibre, and that this is assisted by simultaneously or subsequently stretching it. Experiments are also given as to the action of alcoholic soda on cotton yarn by steeping the yarn in a 10 per cent. solution of caustic soda in 96 per cent. alcohol. No mercerisation takes place, even after some time, but when the yarn is removed from the solution and allowed to hang in the air, it contracts considerably, and appears to be mercerised. If the yarn, after removal from the alcoholic solution, be stretched, and washed in this condition, it acquires a silky appearance not inferior to that produced by aqueous lye. The following table shows that mercerisation only takes place in presence of water, which is most probably necessary in order to form the sodium compound of the cellulose, and this, on further washing with water, forms hydrocellulose :—

| Immersed in | Length of Yarn. | | |
|---|---|---|---|
| | Original. | Before Washing. | After Washing. |
| | Centim. | Centim. | Centim. |
| 1. Aqueous lye, 17° B., for 10 minutes. | 67 | 58 | 62·5 |
| 2. Alcoholic aqueous lye, 11·6 per cent., for 10 minutes. | 67 | 58 | 60 |
| 3. Solution of 6·7 grms. of sodium in 100 c.c. of alcohol. | 67 | 64·5 | 66·5 (washed in alcohol). |

| Nature of the Yarn. | Experiment. | Strength. | Extension. |
|---|---|---|---|
| | | Grms. | Mm. |
| Unmercerised yarn .. | 1 | 360 | 20 |
| | 2 | 356 | 20 |
| | 3 | 360 | 22 |
| Mercerised with— | | | |
| Aqueous lye, 35° B. | 1 | 530 | 44 |
| | 2 | 570 | 40 |
| | 3 | 559 | 35 |
| Cold alcoholic lye.. | 1 | 645 | 24 |
| | 2 | 600 | 27 |
| | 3 | 610 | 33 |
| Warm alcoholic lye | 1 | 740 | 33 |
| | 2 | 730 | 38 |
| | 3 | 680 | 38 |

The fact that mercerising increases the strength of the fibre is shown in the above table, from which it appears that the strength, in the case of alcoholic lye, is much higher, whilst the extension, and consequently the elasticity also, of the fibre is much smaller than with aqueous lye.

—T. A. L.

*Cotton, A Process for the Mercerisation of, not accompanied by Shrinkage of the Thread.* Ducat. Bull. Soc. Chim. 1898, **19**, [15], 718.

The shrinkage during mercerisation is totally obviated by an addition of glucose to the bath. This is composed of 50 litres of caustic soda of 40° B., 30 kilos of glucose (solid), and 20—25 litres of water. The cotton is immersed for 5—6 minutes.

The fabric may be simultaneously dyed, especially in light shades, by the substantive dyestuffs.—A. C. W.

*Silk, Weighting of.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [8], 359—360.

The adulteration of silk by weighting, has for its object the increase in volume or weight, or both, of the material. Organic substances are usually employed to swell the fibre, whilst metallic salts are used to increase its weight, but unless carefully applied, weighting agents seriously decrease the lustre, strength, and elasticity of the fibre.

All classes of silk are now weighted. Raw silk is usually weighted with sugar (75 per cent.); white ungummed silk with stannous chloride ("tin crystals" or "tin salt") and sugar; souple silk with sugar, with stannous chloride, or better, with tannin.

For light shades magnesium sulphate, barium or lead sulphate, tannin, and stannous chloride are used. Darker shades with stannous chloride, for blue, green, or brown, salts of iron and potassium ferrocyanide, or with tannin in boiling solution. For black with iron and tin salt, after mordanting the silk in tannin.

The weighting may take place before (with stannic chloride and tannin, or iron phosphates, or Prussian blue), during (with tannin), or after dyeing (with magnesium or lead sulphates, sugar, or white of egg), as the case may be.

*Weighting with Sugar.*—The silk is worked several times in a strong syrup of sugar, when it takes up 15—20 per cent. of its weight. This weighting agent gives a deliquescent sticky silk, which has the additional disadvantage of attracting flies unless treated with a concentrated solution of *quassia amara* or of magnesium sulphate.

*Weighting with Sulphates of Lead and Barium.*—The silk is treated with basic lead acetate or barium chloride; then in a second bath with sulphuric acid or sodium sulphate, washed, soaped, and finished as usual. In this way silk may be made to take up 350 per cent. of its weight, but as the fibre suffers greatly in lustre, &c., the method is not now used.

*Weighting with Tannin.*—Silk weighted in this way retains its strength and elasticity, but the lustre suffers in proportion to the quantity of tannin employed. In the cold, 10—15 per cent. is taken up, whilst from warm solutions, 25 per cent. may be gained. After treatment the silk is not soaped, but is brightened by means of acid.

The tannin matters usually employed are galls, myrobolans, sumach, chestnut extract, bablah, divi-divi, and catechu.

An extract of galls made in the cold is employed for light shades, catechu is applied hot, or fixed, after standing in the cold overnight, with bichrome or tin salt.

With gall-nut extract the silk is treated at 50°—70° C. for 1—2 hours in an extract of 25°—30° B. and then left in the bath until cold before washing.

Silbermann recommends the following treatment for souple silk, using sumach or gall-nut extract :—A bath is prepared containing 100 litres of water and 12—15 kilos. of the extract. It is then heated to 60° C. and sulphuric acid added until distinctly acid to litmus. The silk is introduced, the temperature raised to 90° C., and a gelatin solution, 10 per cent. of the weight of the silk, added. After working for 1—2 hours the silk is removed to another tannin bath. In this way 102 per cent. in weight is gained. The gelatin solution is prepared by mixing 10 kilos. of gelatin with 1,660 litres of nitric acid of 36° B., and heating to 60° C. for one hour. When the evolution of nitrous fumes has ceased, 100 kilos. of soda are added and 20 litres of water. (This Journal, 1897, 297—303.)—H. I.

*Textiles, Absorption of Liquids by.* L. Vignon. Comptes Rend. **127**, [1], 73—75.

The surface of textiles is so great relatively to their volume that they behave similarly to porous substances. The author has experimented with fibres of silk, wool and cotton, dipping quantities of each, varying in weight from about 0·5 grm. to 3 grms., into various liquids, allowing them to drain by gravity without pressure, and determining the increase in weight. For each liquid the amount absorbed per gram of each textile, was constant, silk being always the most absorbent, wool very near it, and cotton absorbing least. With water and solutions of common salt, the weights absorbed are closely proportional to the densities of the solutions, but there is no *general* relation of the kind. The author's chief results are given in the following table:—

| Liquid. | Weight Absorbed by 100 Parts by wt. of the Textile. | | |
|---|---|---|---|
| | Silk. | Wool. | Cotton. |
| Water | 574 | 559 | 497 |
| Alcohol (93 per cent.) | 683 | 675 | 514 |
| Benzene | 592 | 571 | 506 |
| Water + 10 per cent. HCl of sp. gr. 1·18. | 575 | 592 | 482 |
| Water + 10 per cent. ammonia of sp. gr. 0·928. | 586 | 588 | 509 |
| Salt solution, sp. gr. 1·066 | 613 | 597 | 522 |
| " " 1·130 | 648 | 630 | 551 |
| " " 1·200 | 680 | 660 | 580 |
| Aniline | 781—793 | .. | .. |
| Nitrobenzene | 810—821 | .. | .. |
| Olive oil | 1195 | .. | .. |
| Glycerin | 1611 | .. | .. |

A coarse sponge will absorb 615—638 grms. of water per 100 grms.—J. T. D.

## PATENTS.

*Artificial Silk, Impts. in the Manufacture of, from suitable Forms of Cellulose.* W. P. Dreaper, Braintree, and H. K. Tompkins, West Dulwich. Eng. Pat. 17,901, July 30, 1897.

Cellulose is dissolved in zinc chloride, and fine jets of this solution heated to a suitable temperature are squirted into alcohol or acetone, which precipitates the cellulose and dissolves the zinc chloride. The fibres thus formed are washed and dried in a stretched condition. Dyeing may take place after washing, or colouring matters may be added to the cellulose solution. Waterproofing may be effected by adding substances to the dissolved cellulose, which will become insoluble on drying, or which may be rendered so by suitable treatment, *e.g.*, gelatin. The alcohol or acetone, and the metallic solvent, are recovered by distillation and evaporation.—R. B. B.

*Cloth and other Materials, Woollen or Cotton; Impts. in Waterproofing.* E. Stephens, Totnes, Devon. Eng. Pat. 24,427, Oct. 21, 1897.

Cloth is treated either by hand, or by passing between hot rollers or plates, or by treatment with a hot iron with a mixture of paraffin wax or vaselin, or other hydrocarbon. This mixture may be dissolved in benzoline and applied, or merely painted on. As an example of the method:—3 ounces of a mixture of 4 parts of paraffin wax and 1 part of vaselin, are forced by a hot iron into each yard of cloth, having a width of about 54 ins.

The quantity per yard varies, of course, with the thickness of the cloth.—C. M.

*Bleaching, Scouring, and Stripping; Alkali Phenate Compound for.* E. M. Taylor, Prestwich, Lancashire. Eng. Pat. 25,042, Oct. 29, 1897.

The compound described, is prepared from an alkaline carbonate and phenol, or the phenol may be replaced by a mixture of cresol, phenol, and creosote. When a strongly alkaline compound is required, an addition of caustic alkali is made, and in certain cases rosin is also added. It is claimed that the antiseptic properties of this bleaching and scouring agent preserve the materials from mildew and fermentation, after sizing and finishing.—R. B. B.

*Printing or Reproduction of Words, Figures, Designs, or the like, upon Paper, Textile Fabrics, or other Surfaces; Impts. in, and in the Means or Apparatus employed therein.* W. Friese-Greene, London. Eng. Pat. 27,243, Nov. 20, 1897.

The paper or fabric is prepared by coating or impregnating with a suitable substance, *e.g.*, lead acetate, ferrous sulphate, "amidol," "metol," or other photographic developer. The material travels over a conducting surface of carbon or zinc with an interposed layer of moist cloth or blotting paper, and is connected with one pole of a continuous electric current. The other pole is connected with the words or designs to be reproduced, these being in contact with the prepared material. When the connections are made the current will pass through the paper or fabric, and reproduce a fac-simile of the words or designs. Various colours can be produced, according to the substance in the paper and the material constituting the face of the designs. —R. B. B.

*Waterproofing Compositions, An Improved Method of Applying.* E. Stephens, Totnes, Devonshire. Eng. Pat. 5085, March 2, 1898.

An endless web of canvas is made to travel through a bath in which it is saturated with the waterproofing composition (*e.g.*, "2 parts of paraffin wax and 1 of hydrocarbon oil"), at a temperature of about 125° or 135° C., drained of all but the required quantity (2 to 5 oz. per linear yard of 54 ins. wide), and then passed between steam-heated rollers, &c., in contact with the material which it is desired to waterproof, in such a manner that the latter takes up a sufficiency of the composition, whilst the web returns to the bath to be coated afresh.—F. H. L.

# VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Albumin, the Coagulation of.* G. Halphen. J. Pharm. Chim. 1898, **8**, [4], 173—175.

The addition of ammonium salts (citrate or chloride) favours coagulation by heat. Such coagulation is complete, even in dilute solutions; the liquids filter well and quickly through a double filter. To give one experiment: 1 c.c. of egg albumin solution, 150 c.c. of water, 5 c.c. of 10 per cent. citrate solution, gave a precipitate weighing 0·029 grm.; filtering and washing took five minutes. The explanation of the reaction is that the soda liberated in coagulation displaces ammonia, and thus leaves the medium still favourable for coagulation. The same process has been employed to coagulate the albumin in pepsin and urine. —A. C. W.

*Turkey Red, Alkaline Discharge Colour Printing on.* N. Diakonoff. Färber-Zeit. 1898, **9**, [13], 199—201.

Caustic soda printed on Turkey red and slightly steamed, destroys the alizarin lake, and at the same time forms a soluble compound with it. This soluble compound is best removed by passing the cloth through a solution of silicate of soda, when a compound of the alumina mordant with the silicate is formed, easily soluble in excess of sodium silicate.

The caustic soda is best thickened either with dextrin or British gum. Several formulæ are given for various printing-colours, of which the following is an example:—

For white and yellow, use the same thickening: take 12 kilos. of dextrin and 40 kilos. of caustic soda of 48° B., and pass the clear hot solution through a sieve.

For white, take 36 kilos. of thickening, mix with 36 kilos. of caustic soda of 48° B. Place the vessel in ice-cold water, and then dissolve 5·4 kilos. of zinc chloride in 5 kilos. of water. Add this solution in small quantities at a time, to the well-cooled mixture, with constant stirring, keeping the temperature below 30° C. The addition of the zinc salt gives rise to the production of a very pure white. After printing, the material is passed through a Mather-Platt steaming-chamber, and finally through the sodium silicate bath, the arrangement of which is described. The temperature of the sodium silicate is very important in obtaining pure shades; it must not fall below 70° C.—C. M.

*Fastness of a Dyestuff, Testing the.* J. Herzfield. Textile Record (through J. Soc. Dyers and Colourists, **14**, [8], 176—178.)

In ascertaining the fastness of a colour, attention should be paid to the material of which the fabric is composed. Thus, shoddy goods do not require expensive permanent colours that will last longer than the cloth itself.

Dyes for military cloths must of course stand different tests, as regards fastness, from those applied to goods like valuable silks, which are seldom worn and then only in an artificial light. The common error, that the same dye is equally permanent on all fibres, is to be guarded against. Another circumstance of frequent occurrence is, that a fast colour, when used in a diluted condition for dyeing light shades, is less permanent than when used for dark shades; a good example of this is afforded by the alizarin colours. The tests for fastness of dyes are applied as follows:—

(*a.*) *Fastness to Washing.*—The dyed yarn is plaited with white yarn, or a cutting of the fabric under examination is taken, and rubbed for two or three minutes in a soap solution (5 grms. per litre) at 105° F., then left for 20 minutes in the solution, rinsed, and left for another 20 minutes in the rinsing water; it is finally wrung out and dried. If the colour ought to be particularly fast, the soap solution is heated to 130° F., and the treatment repeated several times. If, under these conditions, the colour remain almost or quite unaltered, and do not stain other coloured or white fabrics washed in contact with it, it is said to be fast to washing.

The test is applicable to fabrics, whether composed of wool, cotton, or a mixture of both.

(*b.*) *Fastness to Rubbing.*—Rub the material on white paper (not too smooth), or better still, on white, unstarched calico. In order to obtain reliable, comparable results, the rubbing must be equal in all cases, and friction surfaces of as nearly as possible the same constitution should be employed. Colours on stockings, hosiery yarns, corset stuffs, and other fabrics intended to be worn next to the skin, must be fast to rubbing.

(*c.*) *Fastness to Perspiration.*—The sample is dipped into a bath of dilute acetic acid (6 c.c. of 30 per cent. acid per litre) at 100° F., rubbed vigorously, and, without rinsing, dried at 68°—77° F. between parchment paper. The more frequently this operation is repeated, the nearer will the test approximate to actual conditions of wear. Another test is to place a sample on the back of a horse, beneath the saddle, and examine the effect of a brisk ride.

(*d.*) *Fastness to Rain.*—This quality is generally required in silk materials for umbrellas. Skeins of dyed silk are plaited with undyed yarns, and left to stand all night in cold distilled water. In the case of woollen yarns, they are generally boiled ten minutes in water. In both cases the white yarn should not be stained, nor should the water be more than slightly discoloured.

(*e.*) *Fastness to Street Mud and Dust.*—This quality is specially demanded in the case of ladies' dress goods, and is tested as follows:—

(1.) Sprinkle the moistened sample with lime and water, dry and brush.

(2.) Sprinkle with 10 per cent. solution of soda, dry, brush, and note any change of colour.

(3.) Immerse in strong ammonia for three minutes, and observe the colour, both in the damp and in the dry state.

(4.) Dissolve 10 grms. of soda in 1 litre of water, and mix with 10 grms. of lime, previously slaked, and reduced to milk of lime by the addition of water, and 12 c.c. of ammonia. Stir the whole well, settle, pour off supernatant liquid, and steep the sample for five to ten minutes in the residue, dry without rinsing, brush, and note any change of colour.

(*f.*) *Fastness to Weather, Light, and Air.*—Cover half the sample with a closely surrounding, but readily removable wrapper of paper, and expose to the sun's rays, but sheltered from rain. At the same time, normal check tests are made with Turkey-red, or a medium indigo-blue on cotton. The samples must be examined daily, so as to ascertain the exact time when alteration begins. In the case of Turkey-red, this will be on the twenty-fifth to thirtieth day, and between the twelfth and fifteenth days for indigo in summer, or double these periods in winter.

The fastness of other colours can then be estimated in comparison with these. An apparatus for quick determinations has been devised by Kallab. The samples to be tested are suspended vertically in the apparatus, and continuously exposed to the sun's rays, the position of the apparatus being changed in conformity with the apparent movement of the solar orb. The action of the rays is strengthened by concentration on a small surface, by the aid of a lens 8 ins. in diameter, and with a focal length of 16½ ins.

Perger proposes a testing apparatus consisting of a plano-convex and a bi-convex lens, the former with its flat surface turned toward the light, serving to parallelise the rays of an arc lamp, situated at the focal length of the lens, which rays encounter the second lens placed in their path at a suitable distance away. A metal disc, placed at a point between the second lens and its focus, receives the sample to be tested.

In estimating the capability of a dye to withstand weather, the country where the material is to be worn must be taken into consideration. Thus, a colour is not so durable in southern countries as in northern climes, and is more strongly affected on the sea coast than in inland districts.

(*g.*) *Fastness to Ironing and Steaming.*—Stuffs to withstand ironing, should not lose their colour when hot-ironed or dried on a hot metal plate.

The capability to resist steaming is determined by steaming a sample laid between the folds of a larger piece of cloth, during which operation the colours should remain unaltered.—J. E. H.

*Chroming of Wool, Note on the.* W. M. Gardner and T. Carter. J. Soc. Dyers and Colourists. **14**, [8], 167—169.

It is well known that old chrome baths give better results than freshly-prepared baths. Whether this is due to the influence of the gelatinous matter ("wool-gelatin"), which is known to dissolve out from the wool when the fibre is boiled in water, is a question which the authors have put to the test of experiment.

To determine the amount of "wool-gelatin" which might come into play during a mordanting or dyeing operation, a quantity of wool was steeped several times in cold water, and then boiled for two hours in water; the solution being subsequently filtered and evaporated to dryness. The amount of "wool-gelatin" extracted was found to be 1·65 per cent. of the weight of the wool. (The wool contained 10·3 per cent. of moisture.) This shows that the action of the "wool-gelatin" must be taken into account in any complete explanation of mordanting and dyeing processes.

Experiments on the large scale and on the small scale showed, however, that the amount of "wool-gelatin" in solution in waste chrome liquors was very much less than would have been present if the wool had been boiled in water instead of in the solution of the mordant. This is probably due to the insolubility of the "wool-gelatin" in the boiling chrome liquors.

"Wool-gelatin" is distinctly alkaline. It has only very feeble reducing properties. The effect of varying increasing amounts of "wool-gelatin" when mordanting with potassium bichromate was to increase the amount of chrome fixed, but to retard the reduction of the mordant. It does not appear, therefore, to be this constituent of the wool which induces the partial reduction of the $CrO_3$ in the mordant bath.

Commercial gelatin and glue, the latter to a greater extent, behaves similarly to the "wool-gelatin."

The chief conclusion drawn by the authors from their experiments, is that the "wool-gelatin" is not the principal cause of the better effect obtained in chrome mordanting baths, after they have been used once or twice.

The function of the "wool-gelatin" in mordanting and dyeing processes, other than the mordanting with potassium bichromate, has not been as yet investigated.—J. E. H.

*Lubricating Oils used for Machinery in Cotton-Spinning and Weaving, &c., the Cause of Injury to Cotton Yarns and Fabrics.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, 334—335.

The formation of "flecks" on cotton goods often results from the oils used in lubricating the machinery. All oils, if they come in contact with the fabrics or yarns, will stain more or less, according to whether they be dark or light coloured. Hence, the preference is usually given to the latter. If the machines be not kept clean, the lubricating oils become quickly charged with metallic particles from the bearings, &c., and on coming in contact with the cotton, they give rise to stains (iron-moulds, &c.). These especially become apparent after the bleaching operations. During spinning and weaving processes, contaminations with the lubricants are almost impossible to avoid, but these are usually removed during the subsequent scouring and bleaching.

Oils differ very greatly in the ease with which they may be removed by scouring. Animal and vegetable oils, such as sperm, olive, cocoa-nut, tallow, lard, and rape, are easily saponified, whilst mineral oils, now very generally used as spindle oils, &c., are removed with great difficulty during scouring and bleaching. The chlorine in the latter operation acts on the remaining oil, converting it into products which rapidly acquire a deep yellow colour, and hence goods showing no traces of flecks may develop them during bleaching. It is interesting to note that Hertel, some years ago, recommended the use of certain oils for the direct production of a fast chamois yellow on cotton, by bleaching.

By the addition of from 15 to 25 per cent. of a fatty oil, such as olive or lard, to the lubricant, the removal of the mineral oil by scouring may be effected, and "flecking" from this cause be avoided.—H. I.

*Dyeing Machinery, Glass and Earthenware Rollers for.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [8], 338.

The great difficulty experienced in cleaning the rollers of dyeing machinery, and the consequent existing liability to stain the goods, has led an English inventor to construct rollers made of glass or glazed earthenware, either massive or hollow, which are easily cleaned.

The hollow cylinders are fixed to a shaft or spindle, running in bearings, either by screws or wedges, or they are cemented to them by means of molten lead or sulphur. Such rollers are stated to be sufficiently strong for ordinary purposes.—H. I.

*Albumin, Testing of Commercial.* Ann. Chim. Anal. Appliq. 1897, 241, through Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [7], 289.

*See under* XXIII., *page* 877.

*Tannic Acid, &c., Absorption of, by Cotton.* W. M. Gardner and T. Carter. J. Soc. Dyers and Colourists, **14**, [7], 143—145.

It is well known that an affinity exists between cellulose and tannic acid, whilst between the former and gallic acid there appears to be no affinity. No satisfactory elucidation of these facts has as yet been put forward. In the authors' opinion, a complete explanation will only be arrived at when the constitution of cellulose is absolutely known. They have endeavoured to find some relation between the degree of absorption and the chemical constitution of the absorbed substance. The substances used were various aromatic phenols of known constitution. In all cases, 1 litre of distilled water, 10 grms. of purified cotton, and 1 grm. of substance, were the amounts employed.

The cotton was steeped for three hours in the cold solution, and the amount of substance was determined in the residual solution. The percentages of absorption are given below—

Table I.

| | |
|---|---|
| Resorcinol .............................. | 45—50 |
| Tannic and catechutannic acids ........... | 30—32 |
| „ phloroglucinol ................ | 24—26 |
| „ mandelic acid .................. | 7—8 |
| „ pyrogallol ..................... | 4—5 |
| Gallic, protocatechuic, and salicylic acids .. | Nil |
| „ pyrocatechol and guaiacol .......... | Nil |

The differences of absorption between the isomers resorcinol and pyrocatechin, also between phloroglucinol and pyrogallol, are very striking.

The absorption of tannic acid by cotton is increased to from 48 to 50 per cent. by the addition of lower fatty acids, like formic, acetic, and propionic acids in equivalent molecular amounts, *i.e.*, equivalent to 4·5 grms. of acetic acid per litre.

Other organic acids, and also mineral acids, were added to the tannic acid solution, the amounts employed in each case being equivalent to the amount of acetic acid used above. The results are shown below, the figures representing the percentages of absorption.

Table II.

| | |
|---|---|
| Tannic acid alone ........................ | 30—32 |
| „ + acetic acid.................. | 48—50 |
| „ + oxalic „ ............ | No definite result on account of secondary reactions. |
| „ + citric „ .................. | 19—21 |
| „ + tartaric acid................ | 20—22 |
| „ + sulphuric acid............... | 18—20 |
| „ + hydrochloric acid........... | 30—32 |
| „ + water saturated with $CO_2$... | 26—28 |
| „ + sodium acetate ............. | 16—18 |

The difference between the action of acetic acid and citric and tartaric acids is noteworthy, and the fact that hydrochloric acid has little or no effect on the amount absorbed, whilst sulphuric acid proves detrimental, may be utilised in the neutralisation of hard waters, &c.

Experiments to determine the best amount of acetic acid to use along with tannic acid, showed that the greatest absorption occurred when 5 grms. of acetic acid per litre was added. This amount of acetic acid is the most effectual in the case of the absorption of gallic acid. Formic and propionic acids also induce the absorption of a small amount of gallic acid, but as with tannic acid, other organic acids, and also mineral acids, have no such effect.

Cotton absorbs only a small amount of gallic acid even from strong solutions (*e.g.*, 10 grms. per litre), and in the presence of acetic acid.

The addition of acetic acid to a tannin bath thus renders about one-twelfth of the gallic acid useful, in addition to increasing the amount of tannic acid fixed, by about 50 per cent.

Since the effect of acetic acid is to increase the amounts of tannic and gallic acids absorbed by cellulose, the authors tried each of the substances enumerated in Table I. with the addition of acetic acid, but in every case the effect of this addition was very slight indeed.—J. E. H.

*Felt Hats, Defects in the Dyeing of.* C. A. Otto. Färber Zeit. **9**, [12], 181—183.

Defects may arise from the raw material used in the manufacture of the hats. Some wools contain tar and red lead, which are used in the marking of the sheep. Such wools are very difficult to get clean, small spots always remaining, which give rise to yellowish spots on steaming and ironing. Hence such wools must be used only for dark-coloured hats. Again, seeds are found in the wool which produce dark spots about the size of peas, these spots being particularly prominent when the dyeing is performed with natural dyestuffs. Where mineral oils are used in spinning the wool, cloudy and spotted goods are obtained on dyeing, since mineral oils are only partially saponified, or not at all, during milling, especially if the milling be performed with sulphuric acid. In the process of felting, defects due to the source of the steam may arise. To avoid these, the feed-water of the boiler must be as pure as possible.

Any oil spots present on the felts are fixed during steaming, and after being milled and then "spunged" (that is, shrunk) appear as yellowish spots. Such hat bodies cannot be employed in the dyeing of light shades.

The felting boards, which are made from mahogany, alder, and other woods, must be well steamed or boiled in water before use; when used for mixed and shoddy felts, they must be well cleansed from dyestuff and dirt, before employing them for white felts.

Too large a stock of felts should not be prepared, because, when the felts lie for a long time, light spots appear, particularly in summer.

After long periods of rest, the water in the pipes must be drawn off before commencing work again, because long standing there causes rust or green scale to be formed. Defects due to this cause may be removed by milling with acids, but not with soap. In the milling process, which follows the felting process, cleanliness is of paramount importance, especially when the felts are worked with soap and acids. Both these latter yield together a grease which is not very easily removed. In the case of half-milled felts which have to be carbonised, this grease must be completely removed, otherwise it would be impossible to dye light shades. The fuller himself must be well experienced in the behaviour of wools. One wool may mill more quickly than another. In this case one portion of the felt contracts more rapidly than another, and thus the fuller has to shorten the milling process, so as to keep the felts evenly contracted. This tends to a superficial cleansing of the felts, which gives rise to a defect in dyeing. Before adding alkalis in the milling process, these must be sufficiently diluted, otherwise they will produce large spots and stripes; also, the appearance of "milling folds" must be avoided. Both these defects can only be overcome by dyeing the felts black. During the shaping of the hats, they must not remain too long on the wooden blocks. The so-called "saddle-pin" must be very clean, so that no rust spots may be produced. In drying the hats, the temperature must not be too high, and there must be good ventilation to carry off the moisture. Much attention and practice is needed for the successful polishing of hats, which ought to be perfectly dry, because the moisture and the heat which is given off form, together with the dust, a more or less scaly coating; this cannot be removed by boiling the hats in water, nor by beating and brushing; such a coating takes up very little colour on dyeing.

If the hat bodies be not quite free from sulphuric acid or alkalis (used in the milling process), the small hairs lying on their surface assume a yellowish tone when ironed, and, after dyeing, the bodies seem as if they are not dyed through and their surfaces appear soiled. In connection with the shrinking process everything must be absolutely clean. Dry steam and a good "shrinking-kettle" are indispensable. The iron blocks and saddles must be well covered with zinc. If the blocks be worn out or the saddle pins damaged, or the steam be damp or the shrinking kettle be out of order, the much-dreaded "spunging" spots appear, in which case the bodies must be dyed black as the only alternative. Where workmen, given to perspiring, handle the bodies, yellowish spots are prominent in certain places, which cannot be got rid of. White and coloured hats must never be shrunk together. The hat bodies have to be completely dry, and any droppings of water must be avoided, if it is desired to dye light shades. With regard to dyeing, many defects may arise in consequence of badly cleaned kettles, careless management, and even during the addition of the dyestuff. The dye kettles, which are made of copper with double bottoms, must be cleaned, more frequently for fancy shades. This is done with acids or sand, after which they are well rinsed and filled up with water to prevent the oxidation of the copper, which would cause yellowish spots on dyeing the hats, which cannot be removed. During the dyeing, especially with alizarin dyestuffs, the hat bodies must be well turned; if this be not done, so-called "kettle-spots" are produced, and cracks begin to appear in the hats if allowed to lie too long in one position. Of course, with wooden vats, the "kettle-spots" and spots arising from the cleaning of the vats do not make their appearance, but the steam must be turned on gradually, else the dyestuff "goes on" too rapidly, and the bodies are dyed unevenly. The dyestuffs must be well dissolved before adding them to the dye-bath. Alizarin is added carefully through a hair-sieve, or, better, thick cloth. The dyestuff solutions are introduced into the dye-bath along with the hat bodies, at a temperature which is not too high.

Alizarin powder should be boiled previously in a small kettle and then added to the dye-bath, through a cloth.

If the hat bodies have been milled with sulphuric acid, it is best to boil them with bran before mordanting. This is a cheap and effective method of obtaining level dyeings, especially with Alizarin Blue S powder and Anthracene Brown. Ammonia or carbonate of soda cannot be used to neutralise the sulphuric acid, since they weaken the material too much.—J. E. H.

### *Calico Printing, Some New Effects in.* Leipziger Färber- u. Zeugdr. Zeit. 1898, **47**, [7], 315—316.

Alizarin Yellow F.S. (Durand) gives on cotton mordanted with chromium a bronze-yellow shade, which is decomposed and reduced to original magenta base by means of stannous chloride, thus producing a change in colour from yellow to puce or violet, a discovery recently made by Brandt (this Journal, 1898, 759). Further, this dyestuff is unaffected by oxidising agents, such as potassium ferrocyanide and sodium chlorate. On the other hand, the dyestuffs of the Phenocyanine group are destroyed both by the latter and by stannous chloride.

Advantage is taken of these different properties of Alizarin Yellow S. and Phenocyanine for printing patterns in green, puce or violet, white, and yellow upon cotton. The process is as follows:—Pad the material in chromium acetate, discharge with sodium citrate, and dye in a mixture of Alizarin Yellow S. and Phenocyanine R. In this way a white pattern is obtained on a green ground. Then dry, and print with a discharge containing potassium ferrocyanide and sodium chlorate, thus producing the yellow pattern. Again, print on stannous chloride, which gives the puce or violet.

The entire effect is said to be very good. Light yellow patterns on a red ground may be obtained by dyeing the cotton in a boiling bath consisting of 3 per cent. of Titan Red, 10 per cent. of Glaubers' salt, and 10 per cent. of common salt. Dry and print with a discharge containing stannous chloride and either Auramine or Canary Yellow. Steam, wash, and dry.

To obtain yellow patterns on a brown ground, dye the cotton in a bath containing 3 per cent. of Paramine Brown C. and 20 per cent. of Glaubers' salt. Dry and print with a discharge paste composed of stannous chloride and either Persian Berry extract, or Auramine, or Thioflavin T and tannic acid. If the latter be employed, fix with tartar emetic as usual.—J. E. H.

### *Waste Liquor from Sulphite Wood Pulp Manufacture (Lignorosin), Utilisation of.* V. H. Seidel and L. Hanak. Mitt. des tech. Gewerbe Museums in Wien, **8**, 1898, [11 and 12], 348—361.

According to the Badische Anilin und Soda Fabrik, violet-black to black shades are produced on vegetable fibres by the products obtained by reducing 1·1′-dinitronaphthalene with glucose in an alkaline solution (Eng. Pat. 20,250 of 1896; this Journal, 1897, 582). By carrying out the reduction in presence of alkaline sulphites or bisulphites, a water-soluble dyestuff is obtained, which dyes wool violet from an acid bath. By substituting sulphite liquor for the glucose, the authors have obtained lilac shades on cotton, and dark violet on wool. For example, 10 grms. of 1·1′-dinitronaphthalene, 800 c.c. of water, 200 c.c. of sulphite-lye, 20 grms. of sodium bisulphite (38° B.), and 20 grms. of soda-lye (30° B.) are heated on the water-bath until completely dissolved. The greenish-blue solution is made acid with hydrochloric acid and boiled until free from sulphurous acid. The dyestuff can be precipitated with alcohol. It dissolves in ammonia with a bluish-violet colour, and dyes wool dark violet and silk violet-grey from an acid bath. In a like manner, 1·4′-dinitronaphthalene gives a brown dyestuff. With regard to the employment of waste sulphite liquor in the indigo vat, experiments have been carried out on a manufacturing scale, employing the following quantities:—2,283 kilos. of indigo (Java, 80 per cent.) (ground with soda-lye, 1 part indigo, 3 parts water), 3·75 kilos. of lime (slaked), 2·5 kilos. of calcined soda, and 10 kilos. of sulphite liquor (28° B.) are brought up to 62·5 litres and heated with direct steam until a reaction suddenly commences at 75° C. The vat is then made up to 500 litres, the diluting water being treated with 10 kilos. of sulphite liquor, 3·75 kilos. of lime, and 2·5 kilos. of calcined soda. Samples of the shades obtained, are given in the paper.

*Utilisation of Sulphite Liquor as a Wool Mordant.*—The most important wool mordant is, without doubt, potassium bichromate, which requires, as a secondary mordant, tartaric acid. The employment of this latter has certain drawbacks, *viz.*, its cost, and the fact that the whole of the bichromate is not utilised but partly runs to waste. The latter is remedied by using a mixture of lactic and sulphuric in place of the tartaric acid. Bearing in mind that tartaric and lactic acids act as reducing agents on potassium bichromate, the authors have employed sulphite liquor with complete success as a mordant, together with potassium bichromate. The liquor is exactly neutralised and evaporated in a vacuum to 30° B. It is brought on to the market under the name of "Lignorosin," and forms a dark brown syrup of 30° B., miscible with water, and containing 50 per cent. of solid matter, principally calcium lignin sulphonate, 6·2 per cent. of ash, 4 per cent. of organic sulphur, and only traces of sulphurous acid. When used as a mordant, it is necessary to add sulphuric acid in such quantity as is equivalent to the potassium of the bichromate and the calcium of the lignorosin. On the large scale it has been found that in presence of sulphuric acid, 1 kilo. of lignorosin will replace the same weight of lactic acid. Tables are given showing comparative tests with lignorosin and dyed patterns of some of the shades produced, compared with those obtained with tartaric and lactic acids.
—T. A. L.

*Tannin, Absorption by Cotton of.* G. v. Georgievics. Mitt. des tech. Gewerbe Museums in Wien, **8**, 1898 [11 and 12], 362—364.

The following facts are known with regard to the absorption of tannin by cotton. 1. A tannin vat is never exhausted, some dissolved tannin always remaining unabsorbed. 2. Cotton, after having been treated in a concentrated solution of tannin, when immersed in a dilute solution does not absorb a further amount, but gives up some of that already taken up, in fact tannined cotton by successive washings with water parts practically with the whole of the astringent. 3. The absorption depends less upon the actual amount of tannin than upon the concentration, and in practice as concentrated a bath as possible is employed. 4. After a certain time the absorption, which is always slow, ceases entirely. From the above it follows that the absorption of tannin follows some regularity and is a reversible process, but observations made by Ed. Knecht, who employed Löwenthal's method of titrating tannin with potassium permanganate and indigo carmine, were not sufficiently concordant to enable a law to be formulated. It is found actually in titrating two tannin solutions, one twice the strength of the other, that in order to obtain exact results they must be brought to the same degree of concentration. In order to determine with as much accuracy as possible in what proportion tannin divides itself between the water and cotton, the author has carried out a series of experiments, details of which are given in the table which follows. The experiments were carried out at the ordinary temperature and lasted about 37 hours.

| — | Tannin. | Cotton. | Water. | Tannin. | | Co-efficient of Distribution | $\frac{\sqrt{C \text{ Vat}}}{C \text{ Fibre}}$. |
|---|---|---|---|---|---|---|---|
| | | | | Absorbed. | Unabsorbed. | $\frac{C \text{ Fibre}}{C \text{ Vat}}$. | |
| | Grms. | Grms. | C.c. | Grms. | Grms. | | |
| I. | 0·5 | 3 | 100 | 0·357 | 0·143 | 13·4 | 0·12 |
| II. | ,, | ,, | 200 | 0·394 | 0·106 | 17·9 | 0·12 |
| III. | ,, | ,, | 300 | 0·404 | 0·096 | 23·8 | 0·11 |
| IV. | ,, | ,, | 400 | 0·4097 | 0·0903 | 29·3 | 0·11 |
| V. | ,, | ,, | 500 | 0·4156 | 0·0844 | 35·9 | 0·10 |
| VI. | ,, | ,, | 600 | 0·4251 | 0·0749 | 35·2 | 0·11 |

In the above table C Fibre denotes the amount of tannin absorbed by the cotton calculated on 100 grms. of cotton whilst C Vat refers to the amount of tannin remaining in the water after the operation per 100 c.c.; thus in II., C Fibre = 3·53, whilst C Vat = 0·197. The formula—

$$\frac{\sqrt{C \text{ Vat}}}{C \text{ Fibre}}$$

is thus a constant, and assuming that in cold aqueous tannin solutions a double molecule of tannin is contained, it follows that a single molecule of tannin is absorbed by cotton from such solutions. An example illustrates practically the working of this rule. From experiment V. 3 grms. of cotton abstract 0·0844 grm. of tannin from a solution containing 0·5 grm. in 500 c.c. In order to double this quantity, previous experience would show that about 1 grm. of tannin is necessary whilst it follows from the formula that in round numbers 2 grms. of tannin must be employed. The amount of tannin absorbed by cotton depends much more upon the quantity of water than has hitherto been assumed, but with the help of the above formula the quantity of tannin necessary in a particular operation can be readily determined and it becomes a definite and precise operation in a cotton dyeworks in the hands of a duly qualified dyer.—T. A. L.

## PATENTS.

*Bleaching Fabrics, An Improved Process of.* E. Cabiati, Milan, Italy. Eng. Pat. 22,450, Sept. 30, 1897.

Vegetable fabrics are bleached in the open width by the evaporation of the lyes employed. The process consists in alternately sprinkling the goods with alkaline solutions, and causing them to pass over heated metallic surfaces. The alternate sprinkling and evaporation are repeated as often as may be necessary to effect saponification, and the goods are finally washed.—R. B. B.

*Dyeing Vegetable Materials, Impts. in.* W. P. Thompson, London, &c. From L. Gevaert, Bevere-Audemarde, Belgium. Eng. Pat. 18,180, Aug. 4, 1897.

To dye a blue colour, Diaminogene Blues are employed together with Alizarine Red or Brown, with or without Alizarine Blue, on chromium or iron mordants.

For the deepest shades, the cotton, &c. should be oiled before mordanting, then mordanted in the ordinary manner, and dyed with the colouring matters mentioned above, after which the colour is diazotised, and developed with $\beta$-naphthol. It is claimed that very fast blues can be cheaply produced by this process.—R. B. B.

*Coloured Designs on Woven Fabrics, Impts. in the Manufacture and in the Production of.* G. B. Ellis, London. From J. Heilmann and Co., Mulhouse, Alsace. Eng. Pat. 18,487, Aug. 9, 1897.

The object of this invention is to produce woven fabrics having raised embroidery-like designs, the fabrics being coloured in the raised portions. According to one method, a resist is placed on the back of the fabric, which is then dried, and a colour is printed on the other side and fixed, *e.g.*, by steaming. The resist prevents the colour from being fixed in the ground, and only the raised parts of the fabric become coloured; when the resist and unfixed colour are washed off, a raised coloured design remains on a white ground.

As an alternative process, the whole fabric is dyed, and the ground colour subsequently discharged by padding the back of the fabric with some substance, which, when passed into a suitable solution, effects a discharge.—R. B. B.

*Colouring Yarns, Impts. in Method of and Apparatus for.* W. Shaw, Brooklyn, U.S.A. Eng. Pat. 12,483, June 3, 1898.

A method of rubbing the bands or stripes of colour printed on yarns, is described, in order to prevent the spreading of colour from one band into adjoining bands of different colours. The apparatus consists of a drum carrying the yarn, a colour wheel, and a spreading device comprising two rubbing portions mounted on the same spring-supported base, the spring pressure upon one portion being lighter than that upon the other.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Nitric Acid, The Reaction of Free Hydrogen on.* Berthelot. Comptes Rend. **127**, [1], 27—29.

Parallel experiments, in which nitric acid was kept in tubes, vacuous or filled with hydrogen, showed that whether in light or darkness, at ordinary temperatures or at 100° C., hydrogen exerts no decomposing action, nor does it interfere with the evolution of oxygen by the nitric acid, which takes place in sunlight or at 100° C. The author contrasts this inertness on the part of hydrogen with the behaviour of metals towards nitric acid, and points out a striking difference in the case of sulphurous acid. The reducing action of metals on these acids, usually ascribed to the influence of "nascent hydrogen," is really due to auxiliary effects in which a "determining" quantity of heat is evolved.—J. T. D.

*Nitric Acid, A New Method for the Estimation of.* W. Ackermann. Chem. Zeit. **22**, [68], 690.

*See under* XXIII., *page* 871.

*Boric Acid, Determination of.* Vadam. J. Pharm. Chim. 1898, **8**, [6], 109—111.

*See under* XXIII., *page* 871.

*Chromous Salts, Decomposition of Water by, and their Use as Absorbers of Oxygen.* Berthelot. Comptes Rend **127**, [1], 21.

*See under* XXIV., *page* 881.

### PATENTS.

*Sea-weed (Alginic Acid], An Improved System or Apparatus for Treating for the Manufacture of Products therefrom.* A. Krefting, Christiania, Norway. Eng. Pat. 12,116, June 2, 1898.

In obtaining the solution of sea-weed described in Eng. Pat. 11,538, 1896 (this Journal, 1896, 720), the inventor found the filtration difficult. To facilitate this operation, he now runs the solution to be filtered on to a horizontal net which is kept moving by suspension between rotating rollers. The portion of the solution which goes through the meshes, is received in a shallow trough, whence it flows by a pipe on to another net below, similarly moving, and mounted, but having smaller meshes, and so on through a series. The coarse particles which remain on the net are washed off by a jet of water, when they arrive underneath and past the end roller, and are caught in a receptacle, whence they pass to the upper side of the first of another similar series of moving nets, the meshes of which diminish in size successively. A modified apparatus is shown with circular moving meshes. (See also this Journal, 1894, 698—699.)—E. S.

*Sea-Weed, Impts. in the Manufacture of Organic Products from.* A. Krefting, Christiania, Norway. Eng. Pat. 13,151, June 11, 1898.

Sea-weed solution, obtained by treating sea-weed with suitable alkalis, was, according to Eng. Pat. 11,538, 1896 (this Journal, 1896, 720), precipitated by sulphuric acid. The inventor now substitutes for the acid treatment the action of an electric current, whereby it is stated that "the alkaline lye, which is necessary for dissolving the sea-weed or the tang-acid, can be regenerated." Certain salts (sodium sulphate, for instance) are added to the solution to make it more conductive. When chlorides are added, chlorine is formed, which bleaches the organic matter.

—E. S.

*Sea-Weed, Impts. in the Manufacture of Organic Products from.* A. Krefting, Christiania, Norway. Eng. Pat. 13,289, June 14, 1898.

Sea-weed is methodically washed in communicating tanks with water in which materials are dissolved to prevent the absorption of water, lime being specified. The sea-weed is thus stated to be deprived of its salts, and is then, after a final wash with water, "brought into the cold, thin solution of alkali or alkali carbonate," in which it dissolves. Antiseptic materials, such as hypochlorites, are added to prevent putrefaction. The mass is then ready for filtration and further treatment, according to Eng. Pat. 11,538, 1896 (this Journal, 1896, 720); but under the present invention the acid treatment there prescribed is omitted.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Porcelain, Hard; Use of Pyroscopes in Firing.* Peyrusson. La Céramique. (Series II.) **1**, 8—9.

In view of inaccuracies and irregularities in the record of temperature by the Seger cones, the author recommends the employment of home-made fusible pyroscopes, consisting of the ordinary hard-porcelain body mixed with definite proportions (5 per cent. rising by units to 14 per cent.) of a cheap flux of known composition—preferably precipitated calcium carbonate. The mixture is made up in cones or pyramids 15 millimetres in diameter and 6—7 centimetres high, which are placed in saggars in the kiln, their change of form (curvature) under the influence of heat is observed through windows of mica. Another shape, which facilitates the detection of curvature, is that of a cylinder, turned over at the top so as to form a horizontal tongue 2 c.m. in length. This forming a more sensitive indicator, the body must be adjusted to finer gradations of difference and may therefore be simply mixed with 5, 10, 15, and 20 per cent. of glaze to form a pyroscope series. It is considered that these will answer for practical purposes.

—C. S.

*Porcelain, Influence of the Atmosphere of the Kiln on the Colours of.* E. Damour. Monit. Céram., **29**, [8], 88.

The apparatus employed by the author, consists of a tube heated to about 1,100° C., and charged with the test articles to be fired, an oxidising or reducing atmosphere being produced at will within the tube, by introducing a current of carbon dioxide from a generator and mixing the same with varying proportions of carbon monoxide, or employing the latter alone.

The results obtained were as follows:—

*Cobalt*.—The blue coloration varies in brilliancy according as an oxidising or reducing flame is used.

*Iron.*—In glazes, the range of colour shade (blues and greens) is limited. Pale Chinese green is obtained with Sèvres glaze + 2 per cent. of iron, in an atmosphere containing 10 per cent. of carbon monoxide, and is persistent in highly reducing atmospheres. Other glazes do not readily yield this shade and are easily decolorised by reducing atmospheres. A variety of fine shades are obtainable with biscuit; and porcelain containing up to 2 per cent. of iron can be entirely decolorised by an atmosphere containing 20 per cent. of carbon monoxide.—C. S.

*Blue Glasses, Chromium as a Base for.* A. Duboin. Comptes Rend. **127**, [1], 52—54.

By fusing for five hours in a brasqué crucible in a wind furnace the materials for forming a glass, along with small quantities of potassium chromate or chromic oxide, fine blue glasses were obtained from melts of the following compositions:—

1. $4\frac{1}{2}SiO_2, Al_2O_3, 3BaO$;
2. $4\frac{1}{2}SiO_2, Al_2O_3, 1\frac{1}{2}CaO, 1\frac{1}{2}BaO$;
3. $28SiO_2, 9B_2O_3, 16BaO, 3Al_2O_3$.

Mixtures for ordinary glasses (Bohemian glass, for example) do not give good blue glasses with chromium.—J. T. D.

### PATENT.

*Glass, Marble, Metal, and Similar Material; Impts. in the Method of and Means for Producing Indelible Designs upon the Surface of.* W. S. Hadley, F. W. Sephton, and R. Mills, Manchester. Eng. Pat. 23,080, Oct. 8, 1897.

The pattern plate is covered with an acid-resisting medium, on which the design is worked, and "bitten-in" with hydrofluoric acid. Ammonium fluoride solution is now allowed to flow over the surface; or the plate is sand-blasted, and the "resist" material is removed. To obtain a transfer, the surface of the etched plate is sponged with glycerin, water or oil, and is then covered with an ink, consisting largely of fatty matter. The ink is scraped off, so as to leave it only in the bitten-in parts. Waxed paper is next pressed upon the plate, the design of which may be transferred to the surface of the glass, marble, metal, or pottery ware to be treated. The design is finally etched by a "suitable acid, sand-blasted, silvered, or fired."
—E. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Fire-Clay Goods from the Under-Clays of Thin Coal-Seams, Manufacture of.* P. Kirkup. Trans. North of England Inst. Mining and Mech. Eng. **47**, 177—198.

The object of this paper is to show how the under-clays of thin coal-seams in the Durham district may be utilised for the manufacture of fire-clay goods, and thus assist the working of seams which by themselves could not be worked at a profit.

The clays underlying many of the thin coal-seams in the western part of the county are well adapted for manufacturing purposes, and may be divided into two classes, *viz.*, the strong or highly siliceous clays, suitable for the manufacture of fire-bricks, and the mild or tender clays, with less silica, specially adapted for making sanitary goods.

Some clays, particularly those rich in silica and containing an undesirable quantity of iron pyrites, are improved by weathering, whilst others deteriorate on exposure.

Rich clays, which vitrify at a low temperature, may be rendered more refractory by the addition of a small quantity of good clean sand, the proportion being based upon the relative quantities of silica and alumina in the initial clay. If, however, the clay does not contain more than 10 per cent. of alumina, the addition of sand will not increase its refractory character.

There are many clays in the northern coal-field which, owing to the presence of from 4 to 5 per cent. of potash or soda, are too fusible for the manufacture of refractory articles.

There are others, however, which are not objected to on account of the alkalis which they contain, but from the too near balance of the silica and alumina, and these could be utilised if properly manipulated. A clay of this description should be mixed with from 7 to 10 per cent. of good sharp river sand, and a small quantity of ground brick.

The clay should be carefully selected and sent to the surface in large pieces, those portions being rejected which contain nodules of ironstone or other impurities, or show a want of uniformity in composition. As the clay arrives at the surface, it should be sorted over, coal-pipes, nodules of iron and other rubbish picked out, the strong clay separated from the mild, and each tipped to the mill-house to be ground and passed through riddles of varying sizes of mesh to suit the special purposes for which the goods are required.

The position of the works should be as near as possible to the pit, with sufficient space for kilns, drying-sheds, and ground to store the finished goods.

The following table shows the saving that may be effected on the coal by working the clay out of the narrow working-places as an economical product:—

*Comparative Statement showing the Cost of Working an 18-in. Coal-Seam by the Single-Stall System: (1) When the Product of Bottom-Cutting is Dealt with as Rubbish in the Pit, and (2) When the Product of Bottom-Cutting is Sent to Bank in the Proportion of 1 of Fire-Clay to 5 of Coal.*

| — | Total Underground Cost. | |
|---|---|---|
| | Amount. | Cost per Ton of Coal. |
| | £ s. d. | s. d. |
| Output per pay of 10 days: 1,000 tons of coal and no clay, when product of bottom-cutting is dealt with as rubbish. | 213 8 4 | 4 3·22 |
| Output per pay of 10 days: 1,000 tons of coal and 200 tons of clay, when product of bottom-cutting is sent to bank in the proportion of 1 of clay to 5 of coal. | 194 0 10 | 3 10·57 |

The 200 tons of clay referred to are delivered at the brick-works at a cost of 2*s.* 2¼*d.* per ton for labour.—H. H. B. S.

*Portland Cement, The Finer Grinding of, and the Comparative Value of the Coarser Particles.* D. B. Butler. Proc. Inst. Civil Eng., Vol. **132**, part 2, 1—14.

The experiments described by the author were instituted primarily with the object of testing the generally accepted theory as to the inertness of the coarser particles of Portland cement. The results, however, led the author to undertake a further series of experiments on the effects of fine grinding. The cements used, comprised samples from the four chief centres of the English industry, *viz.*, the lias districts of Warwickshire, the Northfleet and Grays shores of the Thames and the Medway. Experiments were first made by separating the coarser particles into three degrees, *viz.*, those retained on a 50 mesh sieve, those passing a 50 mesh sieve, but retained on a 70 mesh, and those passing a 70 mesh sieve, but retained on a 120 mesh. The grit, after washing to remove adhering fine dust, was made into briquettes, which were tested for tensile strain, at the end of 6 and 12 months respectively. The results of these experiments show that the coarser particles have a certain cementitious value varying approximately in inverse ratio to their diameter. The experiments upon very fine grinding show that in some instances it may render the cement too quick setting, unless means are taken to correct this tendency, either by thorough aeration, or by the addition of gypsum. On the other hand, finer grinding, though decreasing the cohesive power, largely increases the adhesive power of a cement, and for this reason, samples in addition to being tested neat, should always be tested with a certain proportion of sand. With regard to the addition of gypsum, the author is of opinion that the ultimate effect produced has not been sufficiently studied to warrant its acceptance without reserve, inasmuch as it is somewhat easy to overstep the mark, a 5 per cent. addition causing decided disintegration. Fine grinding corrects a tendency to unsoundness. A cement that would be unfit for use on this account when coarsely ground, would be perfectly reliable if ground extremely fine, owing to the water being able to attack the free lime quicker than it could do if the latter were confined within the coarser particles. The author remarks with reference to the heat evolved on setting, that as the setting is a process of crystallisation, heat necessarily accompanies it, and that the quicker the setting, the greater the rise in temperature. Little or no rise in temperature indicates that the cement is an extremely slow setting one, but it affords no criterion as to its soundness.—H. H. B. S.

*Proportioning Cement in Concrete.* Eng. and Mining J., Aug. 27, 1898, 244.

The quantity of cement mixed with pebbles or broken rock to form concrete, and of lime mixed with sand to form mortar, should be just so much as will fill up the interstices of the silicious substances. With a deficiency of lime or cement, the mortar or concrete will be too poor, so that its homogeneity will not be insured; and too large a proportion of the binding substance will only occasion unnecessary expense without increasing the strength of the mortar or concrete. The above is a summary of some observations in the "Semaine des Constructeurs," by M. Paul Mesnard, who gives the following directions for properly apportioning lime or cement. The volume of the spaces in the silicious matter employed must first be measured, which is easily performed by pouring upon them in a vessel just sufficient water to cover their surface, and then measuring the volume of water poured in.—A. S.

*Lime and Cement, Slaking and Storing.* H. Le Chatelier. Monit. Céramique **29**, [8], 87, 88; 99, 100; [10], 112, [11], 124.

The rate of slaking in lime varies with the degree of compactness of the product, the porous lime obtained by the calcination of calcium carbonate at a comparatively low temperature (900° C.), becoming at once completely hydrated when brought into contact with water, whilst the more compact form, prepared by decomposing a fusible lime salt, *e.g.*, the nitrate, requires from 24 to 48 hours. The presence of fusible substances, *e.g.*, silicates, aluminates, &c., retards the operation; as does also magnesia, but this influence is counteracted by increasing the temperature of the water employed for hydration, or by the admixture therewith of about 2 per cent. of calcium chloride.

In the case of hydraulic lime, the proportion of free lime should be reduced to a minimum, since when hydrated it contributes nothing to the hardening of the mass, and when unslaked, tends to reduce the setting power. In practice, the former evil is obviated by a proper selection of the materials—limestone and fuel—and the latter by slaking at a high temperature, *i.e.*, arranging the heap so as to preserve the heat evolved during hydration. In the laboratory, hydraulic lime is best slaked by immersing the vessel in a boiling saturated solution of calcium chloride.

The process of slaking is divisible into four stages in practice, the first being one of simple absorption of water. The mixture being then shovelled up against the face of the main heap, is warmed in contact, and also evolves heat by the chemical action going on; thus a portion of the added water is evaporated. In the third stage the mass cools, and moisture is fixed by the silicates, although some of the free lime still remains unslaked. That this is so may be proved by quickly heating a sample to 150° C. in a tube, whereupon moisture will be deposited at the upper end, though the temperature is too low to eliminate water from calcium hydrate.

In the final stage the unslaked lime removes this water from the silicates, and becomes completely hydrated without any access of external moisture. The operation is simple, the interstitial atmosphere of the heap containing aqueous vapour at a tension equal to that of the dissociation of the hydrated silicates present; the lime continually absorbs this moisture, which is replaced by a fresh quantity released by the silicates, the speed of the operation varying directly with the temperature.

These necessary stages are frequently curtailed in practice, to the detriment of the product, a sufficient time being essential to enable hydraulic lime of good composition to slake properly, whilst that which becomes quickly and thoroughly hydrated is sure to be of poor quality.

To enable cement to attain the requisite degree of hydration of the free lime, storage—preferably in silos—is indispensable. The changes it then undergoes correspond to the fourth stage in the slaking of hydraulic lime, though they are accomplished more gradually by reason of the lower temperature prevailing. The absorption of moisture from the atmosphere being precluded, the addition of a sufficiency of water is necessary to produce the maximum benefit from the process.

The successive crushings employed in grinding hydraulic cements, result in the pulverising of the more completely hydrated portions first, and thereby give rise to differences in the quality of the product. This defect is avoided by a certain addition of water previous to crushing.

By maintaining the stored cement at higher than the ordinary temperatures, say 100° C., the time of storage could be reduced to about 14 days. Such a course would facilitate the manufacture of natural cements—free from aluminates, and therefore less fusible and more easy to kiln. Such cements, although in the absence of aluminates the combination of silica and lime does not ensue, may, when devoid of free lime, have greater powers of resistance than Portland cement. Consequently, proper storage, by tending to eliminate this element of weakness, affords a means of reviving the said industry.

The presence or absence of free lime may be determined as follows:—The sample is made up into a strong paste with water, and left to set, being moistened to prevent desiccation. Then, within 24 hours, the block is placed in water and heated to boiling point in less than four hours, the distension produced, being measured by the aid of a divided mould or other suitable means.—C. S.

*Normal Sand [for Cement Testing].* M. Gary. Mitth. Königl. techn. Versuchsanst. **16**, [3], 121—143.

The samples of sand submitted to examination were eight in number, *viz.*, Prussian normal sand (Freienwalde); Rhine sand; Austrian normal sand (Lemberg); Swiss normal sand (Solothurn); Russian normal sand; Norwegian normal sand; French normal sands (Cherbourg and Leucate); English sand (Leighton Buzzard); and American normal sand (Massachusetts).

As regards composition, all were found to be free from sulphuric acid, and contained only a small proportion (maximum 0·14 per cent.) of matter separable by elutriation. The German, English, American, and Austrian sands contained the smallest proportion soluble in hydrochloric acid.

The test samples were prepared by mixing the sand with Portland cement, in two proportions, 3:1 and 5:1, by the Böhme percussion apparatus, those for the tensile tests being taken out of the moulds at once, and those for the compression tests after 24 hours. The blocks were all stored in water and tested on the 7th, 28th, and 90th day.

From the tabulated results it appears that the resistance to tension and compression differed, according to the size and shape of the grains, the American (3:1), for example, having the greatest tensile strength and the least resistance to compression, the same peculiarity being shared by the other sharp-edged sands (Swiss and Cherbourg), though in a reduced degree in the 5:1 mixtures.

The Russian and Norwegian sands gave very low tensile values, both initially and on maturing, and in point of resistance to compression, they were inferior to all others except the American.

Taking the figures given by the German normal sand (3:1) as 100, the values yielded by the others are as follows:—Rhine sand, tensile strength, 104 (compression, 114); Leucate, 106 (111); Austrian, 108 (104); English, 98 (109); Swiss, 111 (81); Cherbourg, 110 (81); American, 115 (66); Norwegian, 90 (80); Prussian, 72 (85).
—C. S.

## PATENTS.

*Potters' Clay and other Substances Impts. in or applicable to Filter Presses for Treating.* W. Boulton, Burslem, Staffordshire. Eng. Pat. 20,399, Sept. 4, 1897.

The grooved iron filter-press plates used when dealing with potters' clay and similar substances are made by the improved plan of moulding between opposite truly-turned cast-iron rings, whereby the plates are cast true on each side with their grooved faces, and with chilled and smooth peripheral faces of the required breadth, so that, when applied to the frame and screwed up, they face together truly without having to be turned up.

A further claim relates to overhead lifting and traversing gear, enabling the plates to be lifted for removal of the clay and replaced again by one operator, instead of two having to be employed as at present.—H. H. B. S.

### *Kilns for Baking Bricks, Pottery, Cement, and the like; Impts. in and relating to.* H. Beyer and F. Recke, both of Berlin. Eng. Pat. 1087, Jan. 14, 1898.

This is a round kiln, so arranged that the products of combustion, instead of ascending vertically through a short straight passage through the stack of bricks, are led in a spiral course from the base of the kiln to the flue. In the centre of the kiln is a massive column forming the chimney, and at the inner wall of the kiln is a narrow pillar, and both column and pillar have obliquely ascending passages in them. The materials to be baked are arranged in layers in a spiral manner round the central column, spaces being left for the heated gases to pass through. The gases ascend from one layer to another through the oblique passages in the column and pillar alternately, thus traversing the kiln in a spiral course until they reach the flue and escape. It is recommended that several of such kilns be used in conjunction, in order to utilise the heat to the maximum extent.—H. H. B. S.

### *Artificial Stone, Impts. in and in the Manufacture of, and in Furnaces for Burning the same.* C. Schoenfelder, Vienna. Eng. Pat. 4543, Feb. 23, 1898.

The following ingredients are ground and mixed together:—416—446 parts of silica, 600—630 parts of kaolin powder, 984—1,014 parts of sulphate of lime, 100—140 parts of glass, and 45—100 parts of fluorite. The mixture is then formed into the articles desired, either by pressing or casting. In the former case, the mass is moulded under high hydraulic pressure, and afterwards burnt in a furnace. In the latter case, the mixture is roasted in a current of air heated to 110° C. until 75 per cent. of the water of crystallisation is driven off, and the resulting mass mixed with 1 to 8 per cent. of solution of alum and poured into the moulds. The articles so formed are then dried and burnt.

An improved furnace for burning the moulded articles is also claimed. This is constructed in such a way that a mixture of air and oxygen is conveyed into the burning chamber, so that a perfectly clear flame is produced. The furnace may also be quickly converted into an open fire or into a muffle furnace, without making any alteration in the furnace itself. The finished articles have the appearance of marble, and may be decorated with muffle or stove colours, or by the use of "sgraffitto." They may also be ground and polished.—H. H. B. S.

### *Hard Stones, especially Lithographic Stones; New Method for the Production of.* P. A. Winkler, Leipzig, Germany. Eng. Pat. 5351, March 4, 1898.

Slaked lime is pressed by hydraulic power into metal moulds, then dried at 25° C., and subjected to the action of carbonic acid. The result is an artificial limestone, free from the flaws or defects commonly found in natural stone, and particularly adapted to lithographic purposes.

—H. H. B. S.

## X.—METALLURGY.

### *Gold, Extraction of, and the Cyanide Process.* T. K. Rose. Science Progress, 1898, 7, 307—318.

In a *résumé* of the development of the cyanide process the author emphasises the fact that solution of the gold does not take place in the absence of oxygen or an oxidising agent. It is doubtful whether the efficiency of ferric chloride and the halogens is due to their oxidising power or to their direct affinity for potassium. In most cases with tailings poor in gold and free from sulphides, the air entangled in the ore is sufficient, whilst with ordinary pyritic tailings oxygen is generally supplied by "double treatment," aeration taking place during the draining off of the first solution. The most difficult materials to treat are slimes containing ferrous sulphide, which results from the decomposition of pyrites during the accumulation of the slimes. These slimes must be thoroughly oxidised by mechanical aeration, and, if necessary, by permanganate of potash. On the other hand, an abundance of oxygen means a large destruction of cyanide, and judgment must be used to obtain the most economical result.

The author discusses the subject of dilution, pointing out that a dilute solution is just as efficient as a strong one if the supply of oxygen be not increased as well as the concentration. With slimy ores, which, owing to mechanical difficulties, require prolonged treatment, there is no necessity to hasten the solution of the gold, and it is better to use a dilute cyanide solution, which suffers less waste than a strong one. The objection formerly raised, that the gold could not be entirely recovered from a dilute cyanide solution by means of zinc, has been overcome by using the zinc in threads with a very large surface and by prolonged contact. The electrical processes for the precipitation of the gold have been of immense benefit in the treatment of very dilute solutions, but the electrodes must be large and very close together to overcome the great resistance of the dilute liquid. Anodes of iron or lead peroxide and cathodes of sheet lead are used, and it is best to have a slight excess of potassium cyanide present, to prevent the precipitation of gold cyanide along with the Prussian blue produced.

Very clayey slimes have recently been successfully treated by circulation as a pulp with very dilute cyanide solution through a centrifugal pump with admission of air, and subsequent settling in large vats, and filtration of the decanted liquid through sand. The solution must be absolutely clear, and a low-current density of not more than 0·04 ampère per square foot should be used.—J. F. B.

### *Amalgamation and Cyanide Process, A Combined.* Eng. and Mining J. 1898, 66, [4], 94.

A description is given of a combined cyanide and amalgamation process, for which the U.S. Pat. 601,201, of March 22, 1898, has been granted to the inventors, S. Newhouse, A. J. Bettles, and T. Weir. This process is stated to possess advantages over any other process in the treatment of ores containing coarse and fine gold, which are partly amenable to cyaniding and partly to amalgamation, and also in the case of ores in which the gold is sufficiently fine to be suitable for cyanide treatment, but which contain clayey streaks, which prevent percolation. In the case of an ore like the first-named, the process is stated to cost about the same as pan-amalgamation, but to effect a higher percentage of extraction.

The method is as follows:—The ore is treated with weak cyanide solution in a pan, the gold precipitated from the solution, and the precipitate amalgamated together with any gold which was too coarse to be dissolved. It is stated that there is no difficulty in making the precipitation in the pan and amalgamating the same. The treatment of ores containing much copper is not claimed, but good results have been obtained, nevertheless, with several ores in which the content of copper was considered to be too high to admit of cyaniding, although a slight variation in treatment had to be made.

The essential points to be noted in the process are the strength of the solution, the amount of agitation, the time of precipitation, the condition of the mercury, and the quantity of mercury.—A. S.

### *Tailings in the Witwatersrand, Treatment of.* E. P. Chester. Eng. and Mining J. 1898, 66, [1], 5.

The tailings, as they come from the copper plates of the mill, contain 3 or 4 per cent. of sulphides (chiefly of iron). These tailings assay from 3 to 6 oz. of gold per ton. It is necessary to separate these sulphides prior to the general cyanide treatment, since if the gold should be extracted by cyanide only, they would have to be under solution for nearly a month thus reducing largely the capacity of a plant. When the ore is worth more than 0·6 oz., the separation is effected by means of concentrators—usually the Frue vanner—and the resulting concentrates are either roasted and chlorinated, or the gold extracted by cyanide without previous roasting. About 80 per cent. of the gold is extracted by cyanide, and about 97 per cent. by chlorination, but the resultant profit is about the same in each case, owing to the higher cost of the latter process. When the ores are worth less than 0·6 oz., the tailings are passed through a spitzluten or hydraulic separator, where the greater part of the sulphides is separated from the pulp; but a far larger quantity of sand is collected than when a Frue vanner is employed. The concentrates and sand thus obtained then undergo a double cyanide treatment for a time averaging from 21 to 28 days. The tailings, after leaving the spitzluten, pass into a separator box which divides the slimes from the coarser sands. These latter go through a double treatment, the cyanide tanks being situated one above the other. The slimes that leave the separator box go into another set of tanks, and also under a double treatment in some cases. The first portion—concentrates and sand—is run into the top tank and the water allowed to drain off. A 0·05 per cent. cyanide solution is then pumped through the material for five days, the solution drained off, the tailings discharged into a lower tank, and treated in a similar manner for 10 or 20 days with a 0·02 per cent. solution. After again draining, the concentrates, &c. are finally washed with a very weak cyanide solution.

The tailings from the spitzluten are run into another set of tanks, and the water drained off. They are washed, first with an alkaline solution, and then with a 0·05 per cent. cyanide solution, which is pumped through the tailings for a period of three or four days. After this has drained off, the material is treated for two days with a 0·25 per cent. cyanide solution, allowed to drain after standing three hours, then discharged into the lower tanks, and finally washed with the weakest cyanide solution available. From 75 to 90 per cent. of the gold is extracted from this portion of the tailings.

The slimes coming from the separator box are run into a special set of tanks, one situated above the other, until one-fifth of the upper tank is filled with solid slimes. The water is decanted off, and the settled slimes treated, by means of a centrifugal pump, with a 0·01 per cent. cyanide solution, after which they are washed through into the lower tanks. After settling, the cyanide solution is decanted off, and in the case of very rich slimes a third treatment is carried out, the slimes being finally discharged into a third tank, and thence, after removal of the cyanide, to the waste dump.—A. S.

*Permanganate, Use of, in Gold Mining.* Dingler's Polyt. Journ. **308**, [13], 269.

Potassium permanganate is now successfully used in New Zealand for gold extraction. The finely crushed ore is ground with common salt and sulphuric acid, and the permanganate added to the mixture. Hydrochloric acid is first formed; this with the permanganate yields chlorine, which at the moment of its liberation dissolves the gold. It is claimed that the process, besides having the advantage over the cyanide process of using non-poisonous materials, extracts the gold more completely. It can, moreover, be used with copper-containing ores.—J. T. D.

*Proof Gold and Silver, Improved Method of Preparing.* J. W. Pack. Eng. and Mining J. 1898, **66**, [2], 36.

The gold solution is prepared in the ordinary way, but instead of precipitating the metal by means of oxalic acid or iron sulphate, the solution is copiously diluted with distilled water, and then allowed to flow slowly into a beaker containing a piece of pure aluminium. A perfect precipitation of the gold immediately takes place. The aluminium is removed, the gold well washed in distilled water, dried, and melted, resulting in metal 1,000 fine.

For the preparation of proof silver, fine silver, as free from gold as possible, is dissolved in nitric acid, and the solution diluted copiously with distilled water, and allowed to stand several days in order that any trace of gold present may settle. The solution is carefully decanted and filtered, and the silver precipitated with hydrochloric acid, stirring rapidly during precipitation, so that the silver chloride may be well broken up. Wash with distilled water, place a piece of commercial aluminium in the silver chloride, and add a small quantity of hydrochloric acid. The metallic silver thus produced is washed, heated with hydrochloric acid, washed thoroughly to remove all acid and aluminium chloride, then dried and melted, resulting in metal 1,000 fine.—A. S.

*Nickel Steel, Guillaume's Researches on.* S. Jordan. Bull. de la Soc. d'Encouragement, **3**, [7], July 1898, 807—812.

After a short sketch of the history of nickel and nickel-iron alloys, the author refers to Guillaume's researches on the reversible nickel-iron alloys, which contain more than 25 per cent. of Ni, and both lose their magnetism on heating, and regain it on cooling, at the same temperature; and on the irreversible alloys, which contain 25 per cent. of Ni, or less, and, whilst losing their magnetism on heating, regain it on cooling, but only at a much lower temperature, some below 0° C., and others (*e.g.*, steel with 22 per cent. of Ni and 3 per cent. of Cr) not even at the temperature of liquid air. Guillaume's paper before the Soc. *d'Encouragement*, read on March 11, 1898, has led to the discovery of a valuable alloy containing 35—36 per cent. of Ni, of which the dilatation is 10 times less than that of platinum. It is likely to be used largely in the construction of meteorological instruments and chronometers. The same author has investigated the effect of heat treatment on steel. Reference is specially made to certain plates of homogeneous nickel steel, which were capable, without previous cementation, of being hardened on one face by subjection to a cherry-red heat, and rendered malleable on the other face by heating to a dull red heat.—W. G. M.

*Tin and Antimony Alloys, Analysis of.* A. Fraenkel. Mitt. des k. k. tech. Gewere-Museums in Wien, **8**, 1898 [11 and 12], 334.

*See under* XXIII., *page* 876.

*Arsenic in Antimony and other Metals, Estimation of.* O. Ducru. Comptes Rend. 1898, **127**, [4], 227.

*See under* XXIII., *page* 875.

*Lead Pipes, The Cause and Prevention of the Action of Water on.* A. Liebrich. Zeits. angew. Chem. 1898, [30], 703.

*See under* XVIII. B., *page* 862.

*Alkali Oleate, Reagent for Hydrotimetry and the Estimation of Metals forming Insoluble Oleates.* E. Huart. Bull. Assoc. Belge des Chim. **12**, [2], 56.

*See under* XXIII., *page* 878.

*Formaldehyde, Use of Alkaline Solutions of, in Analysis* L. Vanino. Ber. **31**, [11], 1763.

*See under* XXIII., *page* 873.

## PATENTS.

*Steel, or the like, Method and Means for Manufacturing direct in a Cupola.* T. Doherty, Sarnia, Ont., Canada. Eng. Pat. 18,135, Aug. 3, 1897.

Cast iron or steel are to be run at will from one and the same cupola by altering the conditions of working. To accomplish this the cupola is divided into two parts just above the tuyères by a perforated bridge, which serves to support the solid charge of iron and fuel, and yet to allow the melted iron to pass into the lower chamber, into which the air is introduced through specially constructed tuyères. The tuyères are covered with a partially rotatable sleeve with a fireclay nozzle. When the furnace is being run for the production of cast iron, the nozzles are so turned that the apertures are above the level of the melted metal lying on the hearth, but when steel is to be produced, the nozzles are turned so that the air passes through or on to the bath of metal. In either case the blast passes upwards through the aperture by which the accumulating molten metal descends into the hearth.—W. G. M.

*Steel and Silver, Manufacture of a Compound Metal of.* E. Martin, Paris. Eng. Pat. 937, Jan. 12, 1898.

Bars of steel (preferably 200 × 600 mm. by 50 to 100 mm. thick) are cleaned, and are then prepared by one of several alternative processes. Copper is deposited in a thin layer, a film of zinc or tin is deposited either on the copper or the iron, and the whole is afterwards heated to cherry redness and pressed, in order to ensure union between the iron and copper. Or, a film of zinc or tin is deposited on the steel, a foil of copper or bronze is laid on the surface, and a similar bar is placed in contact with it, the two copper surfaces being in juxtaposition but separated by a coating of flake-white to prevent adhesion. The whole is then tied together, heated, and pressed. After cooling, the steel has a coppered surface ready for union with a plate of silver. Prepared plates of steel are then placed in contact with the silver plates, and are heated and pressed or rolled. The compound steel and silver sheets may be re-heated and rolled to any required thickness. If preferred, the steel, having an electrolytic deposit of copper, zinc, or tin, may be heated and placed near the side of the mould, the separating space being filled in with molten silver. The resulting compound bar is then rolled as before.

—W. G. M.

*Steel or Malleable Iron from Cast Iron, Impts. in the Manufacture of.* J. A. Hunter, Philadelphia, U.S.A. Eng. Pat. 7377, March 26, 1898.

Cast iron is heated in a crucible or furnace, and is supplied with oxygen from a mixture of sulphuric acid and an oxide or substance which decomposes the acid to produce oxygen and a sulphate. The substances mentioned are oxide of manganese, hypochlorite of lime, chlorate or nitrate of potash.—A. W.

*Iron or Steel, an Improved Process and Preparation for Coating.* W. P. Thompson, Liverpool. From W. J. Wilder, West Chicago, U.S.A. Eng. Pat. 11,265, May 17, 1898.

In the place of zinc in the galvanising bath, an alloy of zinc, tin, and aluminium, with or without lead, is used, and the steel or iron sheets are cleaned with acid and flux in the ordinary way previous to dipping. A mixture given consists of 84 per cent. of zinc, 14 per cent. of tin, 1·5 per cent. of lead, and 0·5 per cent. of aluminium.—A. W.

*Iron or Steel Plates, Sheet, Process of Oxidising and Finishing.* W. M. Theobald, Wellsville, Ohio, U.S.A. Eng. Pat. 11,689, May 23, 1898.

The plates, whether oxidised or pickled initially, are annealed for about six hours in annealing boxes, from which air is excluded by luting the joints; the boxes are then removed from the furnace and allowed to cool for 25 minutes, by which time the temperature within will be between 900° and 1,400° F. The plates are now exposed to the air until sufficiently oxidised, and then packed one on another to prevent further contact with air, and are passed, at 300° to 900° F., between highly polished rollers. In this way a blue plate with an oxidised finish is produced.

—W. G. M.

*Steel, Impts. relating to the Treatment of.* L. Budzinski and B. Schouwaloff, both of Paris. Eng. Pat. 13,123, June 11, 1898.

The piece of steel, or burnt steel, when red hot, is dipped into a mixture of buckwheat meal, Indian wheat (maize) meal, charcoal, red pepper, and asafoetida, and allowed to cool therein. This treatment softens the metal, produces a finer texture, and improves it for tempering.—A. W.

*Chemical Combinations [in Metallurgy], Impts. in the Process and Apparatus for Decomposing.* H. Niewerth, Berlin. Eng. Pat. 18,327, Aug. 6, 1897.

Three vertical cylindrical chambers are arranged side by side in a furnace. The chambers form part of a gas-circulating system, which also includes two reservoirs for gas and a pump-cylinder with a piston working within it. Horizontal connecting flues are used so that gas may be pumped from one reservoir into the bottom of one chamber, after traversing which it passes from near the top to the middle chamber, and then from the bottom of this to the third chamber, from the top of which it finally escapes to the other reservoir. In use, the ore (for example) to be reduced, is fed into the middle chamber, whilst the two wing chambers are kept filled with carbonaceous material, and the reservoirs are initially filled with carbon monoxide, or with a mixture of that gas and hydrogen. The furnace being heated, the piston, which is valveless, is set in motion, oscillation of the gases takes place, reducing gas is passed from one wing to the central chamber and thence to the other wing chamber, and is afterwards returned in the reverse direction; and the oxidation of the gases by the reduction of ore is thus afterwards neutralised by the reducing material in the wings. The object of producing reversal instead of continuity of direction is the protection of the gas reservoirs from overheating. The reduced material is discharged through a door at the bottom of the central chamber. It is claimed that iron or steel may thus be produced direct from the ore. For some reductions, metallic sodium may be introduced into the wing chambers, and, being vaporised, may then be conveyed into the reducing chamber.—W. G. M.

*Metals from their Ores, Impts. in Extracting Metals, and in Apparatus therefor.* S. H. Johnson, Stratford. Eng. Pat. 4443, Feb. 22, 1898.

This is a modification of Eng. Pat. 19,389 of 1891 (this Journal 1882, 922), in which an apparatus and process were protected for removing soluble salts from finely ground minerals that had previously been treated chemically. In the present specification the whole treatment is carried out in the same apparatus. Several leaching processes may be placed in series, and the solvent passed through them in succession until it is saturated. The mineral in the first vat being first exhausted is discharged, the press is recharged, and then becomes the last instead of the first of the series. The permeable cakes are thus extracted and finally washed. The solvent may also be aërated before or during circulation. The presses are charged initially by mixing the ore to a slurry in a mixing vat, and then forcing the pulp into them. The claim is for the chemical treatment of such cakes in the apparatus described.—W. G. M.

*Iron Ores (Magnetic), Process and Apparatus for Concentrating, and Bricking the same for Market.* B. J. B. Mills, London. From T. A. Edison, Llewellyn Park, N.J., U.S.A. Eng. Pat. 8528, April 12, 1898.

The ore is passed through breaking rolls, then through crushing rolls, next through a drying chamber to pulverising rolls, after which the material is screened, the tailings being conveyed back to the pulverising rolls. The fine material is now passed through magnetic separators, and the concentrated ore is conveyed to dryers, after which the ore may be mixed with a suitable binding agent, such as a mixture of petroleum and resin, formed into briquettes and baked. Or, after the concentrates are dried, they may be again screened, the tailings from these screens being re-pulverised, and the whole may then be passed through a second magnetic separator; the separated concentrates are now dusted and subjected to a final concentration, and are either dried, mixed with a binding medium, bricked, and baked, or else heated, mixed with a binding agent, cooled, bricked, and baked. The whole arrangement is made automatic with the aid of conveyers and elevators, and this arrangement forms the subject of one of the claims.—W. G. M.

*Furnaces, Impts. in Roasting Ovens or, for the Desulphurisation of Volcanic Sulphur, Sulphurous Ores, and Ores containing a greater or less Percentage of Sulphur.* W. P. Thompson, Liverpool. From H. Harland and S. D. Crenshaw, both of Richmond, U.S.A. Eng. Pat. 8570, April 12, 1898.

The ore, &c. is placed upon firebars in a chamber with a roof outlet, and is roasted by air from underneath. In addition to this, air is supplied through the bars into hollow vertical narrow fireclay boxes or conduits running from back to front of the furnace and situated at intervals on the bars and in the ore. These conduits possess side-holes through which air passes to the roasting ore at different elevations.—A. W.

*Gold Bearing and other Ores, Impts. in Separating Liquids from Fine Slimes of, and Apparatus therefor.* C. H. Pead, Boksburg, S. Africa. Eng. Pat. 11,002, May 13, 1898.

The vessel is circular, with a truncated conical hood, to the top of which a filtering screen is clamped. To the under surface of this screen small pipes are led through the hood from a circular tube outside connected with a pump supplying the slime pulp. The vessel fills with the pulp, and on continued pumping, more pulp enters the vessel, the water passing upwards through the filter and running over the top of the hood into a gully, and the slime being left behind. The filter is kept clean by the supply of pulp through the small pipes being made to impinge on its under surface. The action continues, and the slime mud accumulates at the bottom of the vessel in a compact state ready for periodical removal.—A. W.

*Copper Matte, Impts. in Treating.* J. Colquhoun, Clifton, U.S.A. Eng. Pat. 11,180, May 17, 1898.

In the smelting of copper ores by the Bessemer process, the matte and slag from the cupola, instead of being run into a settling pot or being allowed to cool previous to being put into the converter, is now run into a reverberatory furnace, where it is stored, and, if necessary, improved and brought to the right grade and temperature for successful reduction to metal in the converter. Whilst in the reverberatory furnace, the slags from a previous charge in the converter are added for recovering the copper therefrom.
—A. W.

# XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## (*A.*)—ELECTRO-CHEMISTRY.

*Electrolytic Conductivity of Salt Solutions, Contributions to the Study of.* R. Goldschmidt and A. Reychler. Bull. Soc. Chim. 1898, **19**, [15], 675—681.

The authors have devised an apparatus of small capacity, by which electrolytic conductivities may be taken by the method of Kohlrausch. One of the electrodes is permanently fixed at the bottom of an ebonite cup, and is connected to a binding screw by a wire passing through the ebonite; the other is attached to a micrometer screw protected at the lower extremity by an ebonite sheath. The distance between the electrodes may be read to about 0·01 mm. by means of the divided head of the screw. The first apparatus, with electrodes of 9·4 mm. diameter, in a cup of 15·5 mm. in diameter, did not give a sufficiently clear sound in the telephone when the electrodes were 3 mm. or more apart. The second apparatus employed, with electrodes of 14·3 and 40 mm. diameter, in cups of 17·3 and 47 mm. diameter, respectively, gave satisfactory results under certain conditions. The specific conductivity of $\frac{1}{500}$ N-potassium chloride solution was calculated from the observed resistances, R and R¹, of $\frac{1}{50}$ N- and $\frac{1}{500}$ N-potassium chloride solutions, taking Kohlrausch's figure for the conductivity of the former solution.

$$\text{Sp. Cond.} = \frac{R}{R^1} \times 0·002244.$$

The figures obtained, decreased regularly as the distance between the electrodes increased (after the distance of 3 mm., below which the results are irregular).

Satisfactorily constant results were obtained when resistances $R_m$ and $R^1_m$, $R_n$ and $R^1_n$ were measured, for the distances, m and n, between the electrodes. Then

$$\text{Sp. Cond.} = \frac{R_m - R_n}{R^1_m - R^1_m} \times 0·002244.$$

This apparatus might be employed to determine conductivities by a method the inverse of the ordinary. The resistances of a standard solution being measured for different distances of the electrodes, the condition of silence in the telephone using a solution of unknown conductivity can be effected by movement of the micrometer screw, the moveable contact on the bridge remaining at the centre of the wire.—A. C. W.

### PATENTS.

*Calcium Carbide, An Improved Method of and Apparatus for the Production of; applicable also to other Purposes.* H. Maxim, London, and W. H. Graham, Trowbridge. Eng. Pat. 18,989, Aug. 17, 1897.

*See under* II., *page* 833.

*Galvanic Cells, Impts. in.* [*Zinc Carbon.*] A. Heil, Hesse, Germany. Eng. Pat. 24,591, Oct. 23, 1897.

The cell consists of a carbon electrode surrounded by equal parts by volume of graphite, manganese peroxide and lead peroxide, and of a zinc electrode, the electrolyte being zinc sulphate, to which sufficient sodium carbonate has been added to produce a flocculent precipitate of hydrated carbonate of zinc. To maintain the even distribution of this flocculent precipitate, some absorbent material, such as a mixture of gypsum and sawdust, is stirred into it till it forms a pulp.—G. H. R.

*Batteries, Primary, Impts. in or connected with.* [*Peroxide of Lead Negative.*] A. Peters, Copenhagen, Denmark. Eng. Pat. 14,112, June 25, 1898.

Electrically prepared peroxide of lead, which may be supported on a frame or grid, or enclosed in a porous pot, is used as the negative electrode in conjunction with a positive electrode of zinc, iron, or tin, which may be amalgamated. Any suitable electrolyte, such as sulphuric acid, phosphoric acid, or the solution of an acid sulphate or the like, may be employed, or the battery may be set up as a dry cell, in which case tin or amalgamated tin is used for the positive electrode.—G. H. R.

## (*B.*)—ELECTRO-METALLURGY.

*Tin Residues, Electrolytic Treatment of.* Ind. Électrochim. 1898, **2**, 2—8. Science Abstracts, 1898, **1**, [7], 434.

The total loss in the manufacture of tinned iron is stated to amount to thousands of tons annually, the difficulties in the treatment of the residues arising from their large bulk and from the presence, under the layer of tin, of an alloy of tin and iron. In some works caustic soda is used as electrolyte; in this way the tin is dissolved, but the iron left has little value, owing to the relatively large proportion of tin admixed with it.

In a recent process the scraps of metal are packed in wooden frames, which are used as anodes, whilst the tin is deposited upon cathodes of tinned copper. Dilute sulphuric acid is used as the electrolyte, and the iron is eventually completely dissolved.—A. S.

# XII.—FATS, OILS, AND SOAP.

*Vegetable Oil Industry.* E. Fleurent. Rev. Prod. Chim. **1**, [3], 33—34.

When olive oil is heated to 120° C., in thin layers, with free admission of air, the product solidifies on cooling, in the form of large foliaceous crystals, but in proportion as the heating is prolonged, the crystalline structure of the cooled mass is gradually modified by oxidation into the elastic amorphous condition. On the other hand, stearic acid, under the same treatment, is converted into an intermediate liquid form (in the cold) before definitely assuming the consistency of linoxin.

Castor oil affords the sole exception to the rule of the general susceptibility of oils to oxidation, and its influence in thickening varnishes is ascribed by the author to its mechanical diluent action, whereby the evaporation of the solvents present is facilitated.—C. S.

*Stearic and Palmitic Acids, and Mixtures of the Two; Solidification Point of.* L. E. O. de Visser. Rec. trav. Chim. Pays-Bas. **17**, 182—189, Chem. Centr. 1898, **2**, [3], 176.

The author determined the solidification point of carefully purified stearic acid (by about 50 crystallisations), and found it to be 69·32° C., with a hard glass normal thermometer and 69·247° C., with a hydrogen thermometer. Purified palmitic acid, examined in the same way, gave 62·618° C., and 62·532° C. The solidification points of different mixtures of the two acids are set out in the form of a curve, which showed two breaks. The first break (with 54 per cent. of stearic acid) cannot be explained at present, but the second (with 47 per cent. of stearic acid) is explained by the appearance of a "solid solution." The lowest solidification point found was 54·8175° C, with a mixture containing 29·76 per cent. of stearic acid.—A. S.

*Fatty Acids, Bibasic, derived from the Oxidation of Fats; Separation of.* L. Bouveault. Bull. Soc. Chim. 1898, **19**, [13], 562—565.

Having occasion to prepare a quantity of adipic acid, the author adopted the following method of separating the different acids in a mixture resulting from the oxidation of a fat, and consisting principally of succinic, adipic, glutaric, and pimelic acids:—By distilling the mixture under the ordinary pressure until the temperature reached 230° C., the whole of the succinic acid was converted into succinic anhydride, which could then be separated from the other acids by continuing the distillation under a pressure of 20 mm., when it passed over at 135°. The residual mixture of bibasic acids (boiling at 210° to 225° under 20 mm. pressure) was treated with cold ether, which dissolved about one-third of the total quantity. The insoluble portion (m.p. 135° to 140°) consisted chiefly of pimelic and adipic acids, and yielded the latter on crystallisation from boiling water. The adipic acid obtained melted at 140° C., and, though insoluble in water, was found to be extremely deliquescent. The glutaric and pimelic acids dissolved in the ether, and in the mother-liquid of the aqueous crystallisation, were separated by being converted into calcium salts and treated with water, in which calcium pimelate is but slightly soluble in the cold, and insoluble on boiling whilst calcium glutarate is exceedingly soluble. The author considers that his results show the incorrectness of Arppe's statement that pimelic acid is not present in products of the oxidation of fats by nitric acid; in fact, in such oxidations he has found all the usual acids present in varying quantities from succinic up to sebacic acid.—C. A. M.

*Oils and Fats, Fractional Saponification of.—II.* R. Henriques. Zeits. angew. Chim. 1898, [30], 697—702.

The author here gives some supplementary notes to his former paper (this Journal, 1898, 673) on this subject. He finds that his conclusions as to the formation of ethyl esters of the fatty acids with liberation of the glycerin during the saponification of triglycerides with alcoholic potash have already been forestalled by Bouis in 1857, who drew the same deductions from a similar series of experiments (Comptes Rend. **45**, 35; J. prak. Chem. 1857, **52**, 308). Recently Geitel (J. prak. Chem. 1897, 429; 1898, 113) has arrived at the conclusion, from physical considerations, that in the saponification of all partially decomposed triglycerides, mono- and diglycerides are produced. Without attempting to refute this view on theoretical grounds, the author points out that Geitel has brought forward no experimental evidence in favour of his theory except certain results which, in his (the author's) opinion, lend more support to the theory that ethyl esters are produced. Moreover, as in his former paper the author suggested the possibility of diglycerides being present in the residue left on saponifying linseed oil with a smaller amount of alcoholic potash than was requisite to liberate all the glycerin, he has made further experiments on this point with almond oil, and finds that only unaltered triglycerides remain. Even on treating almond oil for eight hours with steam at 250° to 270°, so as to cause a disruption of the molecule and distillation of about 10 per cent. of the substance, the residual portion, when washed free from liberated fatty acids and glycerin, was found to consist of unaltered triglycerides alone.—C. A. M.

*Fats, Determination of Unsaponifiable Matters in.* Schukow and Schestakow. Zap. imp. russk. techn. obschtsch. 1898, **32**, 109.

*See under* XXIII., *page* 878.

*Olive Oil, Method of Detecting Cotton-seed, Sesamé, and Earth-nut (Arachis) Oils in.* M. Tortelli and R. Ruggeri. Chem. Zeit. 1898, **22**, [60], 600.

*See under* XXIII., *page* 876.

*Lubricating Oils used for Machinery in the Cotton Industry.* Leipziger-Färber und Zeugdr. Zeit. 1898, **47**, [8], 334.

*See under* VI. *page* 843.

[*Lubricating Oils*], *The Chercheffsky Viscosimeter.* J. Jean. Rev. Chim. Industr. **9**, [104], 220.

*See under* XXIII., *page* 872.

*Soaps, Analysis of.* R. Hefelmann and E. Steiner. Zeits. für Öffentl. Chem. **4**, 389. Chem. Centr. 1898, **2**, [2], 142.

*See under* XXIII., *page* 878.

## PATENTS.

*Soaps or Cleansing Compositions, Impts. in the Manufacture of.* F. Becker, Prague, and O. Wolff, Dehnitz, Saxony. Eng. Pat. 16,382, July 10, 1897.

It is known that even a soap which is quite neutral in the solid state decomposes in dilute solution, acquiring caustic properties that are injurious to wool fibre and to many aniline dyes. The inventors overcome this difficulty by sulphonating part of the fatty constituents of the soap, thus producing a material which can be used with advantage for greasing and fulling wool, for mordanting cotton, and for bleaching dyed yarns. The soap can be prepared in several ways, of which the following is a type:—100 kilos. of acid Turkey red oil are mixed with 50 kilos. of oleic acid, and the whole is saponified with potash, soda, or ammonia, in the usual fashion. Instead of the castor oil, any oil capable of sulphonation may be employed; and other fatty acids adopted in place of the oleic acid.—F. H. L.

*Oils, Impts. in the Oxidising of.* S. G. Rosenblum and "The Commercial Ozone Syndicate, Ltd.," London. Eng. Pat. 6464, March 16, 1898.

In order to produce quick-drying oils for paint, &c., or semi-solid products for linoleum, linseed oil, or a mixture of equal parts of linseed with cotton-seed or rape oil, is subjected to the action of ozone in conjunction with suitable driers, as described in Eng. Pat. 9529, 1897 (this Journal, 1898, 587). It is stated that the mixture solidifies more rapidly than either of the said oils alone.—F. H. L.

# XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

## (A.)—*PIGMENTS, PAINTS.*

### PATENT.

*Zinc [as Pigment] from Liquors containing Zinc in Solution with Iron and other Metals, Impts. in Separation and Recovery of.* W. J. Wigg and E. G. Ballard, Chester. Eng. Pat. 18,217, May 5, 1897.

The residual liquors resulting from the extraction of copper by the wet process are first deprived of the greater part of the iron contained in them by known processes, and are then treated with milk of lime. Air is then forced into the liquor until the iron oxide precipitated is sufficiently oxidised. The whole is then acidified, and the zinc thrown down is brought back into solution by again injecting air.

The iron oxide, separated, may be prepared as a pigment, while the zinc may be precipitated and smelted. But as the zinc thus obtained still contains iron, and cannot therefore be used as a pigment, the alternative process may be adopted of treating the zinc liquor with air and a small proportion of calcium carbonate. The iron is thus oxidised and precipitated. The zinc solution, which may still contain manganese and other metals, is heated, and being kept neutral, is then treated with bleaching powder solution or chlorine. The solution, after filtration, is precipitated by lime, "or other precipitant suitable for converting the zinc into a pigment." The precipitate is then collected and calcined.—E. S.

*White Lead, Impts. in Chambers for the Manufacture of.* Saxby's White Lead Syndicate, Ltd., and H. G. Percival, Willesden, Middlesex. Eng. Pat. 12,318, June 1, 1898.

The invention relates to the construction of chambers for the manufacture of white lead in such manner that the workmen may not be exposed to deleterious fumes, the process being the ordinary one of exposure of lead to acetic acid vapour, air, moisture, and carbonic acid at suitable temperatures. The floor of the chamber slopes slightly from the sides and ends towards the middle, air pipes passing upwards from channels below, communicating with the external air. Air pipes also lead from the ceiling to a space in the roof, whence the air and gases pass to a chimney. Other air pipes supplied from external shafts lead upwards and open into an air-chamber immediately under the floor, which chamber has rows of dwarf piers spanned by slabs, which carry the floor, consisting of slabs bedded on cement. Steam pipes pass between the rows of piers. The walls are hollow, and the spaces are provided with short pipes having openings to the exterior and interior of the chamber and with lateral openings to the spaces. By these pipes the admission of hot or cold air can be controlled. Carbonic acid gas is admitted by a pipe beneath the floor. The air-pipes are provided with accessible throttle valves. The lead is exposed on trays on wooden shelves throughout the interior of the chamber. The floor is covered with a layer of acetic acid.—E. S.

## (B.)—RESINS, VARNISHES.

### PATENTS.

*Iron Work, A New or Improved Process and Composition for the Covering and Preservation from Rust of Exposed.* K. B. Skeet, Chichester. Eng. Pat. 14,595, July 2, 1898.

The surface is painted with coal tar, and then "the burnt or partially burnt coal dust and smoky deposits in the smoke boxes of marine and other engines" is sprinkled all over it; a second coating being applied after the lapse of a few days. In damp situations the tar and carbon may be mixed together before use. The composition "wears better, looks better, is much cheaper than paint," and is not heavy.—F. H. L.

## (C.)—INDIA-RUBBER, &c.

*India-Rubber, South American [Pará and District].* W. A. Churchill. Dipl. and Consular Report, No. 2,140, 1—35.

In his annual report on the trade of Pará, Brazil, for the year 1897, the Consul deals at length with the subject of india-rubber, the production of which is a staple industry in his district. For the purpose of comparison he gives the following list of all the known varieties of plants which yield elastic gum, together with their habitat :—

*Order Euphorbiaceæ.*—Hevea guianensis, British, Dutch, and French Guiana; H. brasiliensis, Upper and Lower Amazons and Upper Orinoco; H. Spruceana, mouths of Rivers Tapajós and Madeira and Lower Amazons; H. discolor, mouth of River Madeira and near Manáos; H. pauciflora, River Negro and River Uapés, British Guiana; H. rigidifolia, H. lutea, H. membranacea, and H. Benthamiana, River Negro and River Uapés; H. nitida, River Amazons and River Solimões; H. janeirensis, Rio de Janeiro; Manihot Glaziovii, Ceará.

*Order Artocarpaceæ.*—Castilloa elastica, Mexico, Central America and Colombia; C. Markhamiana, Panamá; Artocarpus integrifolia and Cecropia peltata, Central America; Hancornia speciosa, Pernambuco; Vahea gummifera, Madagascar; Landolphia florida, L. Owariensis, L. Kirkii, L. Petersiana, L. Watsoniana, and L. Manni, Africa; Urceola elastica, U. esculenta, Willughbeia Burbidgei, W. Treacheri, W. Edulis, Leuconotus eugenifolius, Chitocarpus flavescens, Chonemorpha macrophylla, Anodendron paniculatum, and Alstonia Constricta, East Indies.

*Order Sapotaceæ.*—Isonandra dichopsis, Sumatra, Borneo, and Malacca; Mimusops Balata, Dutch and French Guianas, Venezuela, Trinidad, and Jamaica.

*Order Moraceæ.*—Ficus elastica, F. macrophylla and F. rubiginosa, India, Ceylon, Assam, Burmah, Java, and East Indies generally; Urostigma Vogelii, West Africa.

During the year 1896, the following quantities of india-rubber were imported into Europe and the United States :—

| From | Quantity. | | |
|---|---|---|---|
| | To Europe. | To United States. | Total. |
| | Tons. | Tons. | Tons. |
| South America.............. | 8,858 | 10,406 | 19,264 |
| Central America ............ | 789 | 984 | 1,773 |
| Africa ....................... | 6,448 | 2,663 | 9,111 |
| East Indies.................. | 776 | 617 | 1,393 |
| Total ............... | 16,871 | 14,670 | 31,541 |

Rubber was first introduced from South America into France in 1736, and into England in 1770, where for the first half century after its introduction, it was used for no other purpose than erasing pencil marks. It was first manufactured into waterproof clothing in France towards the end of last century.

The "Hevea" tree, which in appearance somewhat resembles the English ash, grows to a height of about 60 feet. It blossoms in August, and the fruit becomes ripe in December and January. Like other rubber trees it thrives best on low ground, which is periodically inundated, but will not grow satisfactorily on cleared and open ground. The shade of other trees and a still atmosphere appear to be necessary to its well-being. Since the "Hevea" requires about 15 years to mature, no cultivation has been attempted in the region of the Amazons, as it would not afford a good investment for capital so long as the forest shows no sign of exhaustion. Certain districts become exhausted when overworked, but recover when left alone for some time, and there are some as yet untouched. The area which is known to yield Pará rubber, is estimated at at least 1,000,000 square miles, of which the richest zones are on the banks of the southern tributaries of the Amazons, on the islands in the main river, and near Pará. On the River Aquiry or Acré, as much as 1 ton of rubber per annum is obtained from 100 trees. The northern tributaries of the Amazons, so far as they have been opened up, do not produce much rubber.

The process of extraction in use is very primitive. From 60 to 150 trees are tapped, and on the following days the process is repeated, the gashes being made a little lower down than the first. The rubber, accumulated in three or four days, is cured by being held on a broad wooden paddle over a wood fire (in which the fuel usually consists of the nuts of certain palms), until the milk coagulates. It is stated that this process of curing is very injurious to the eyes, and that many cases of total blindness have been caused by it. Nevertheless the attempts made to introduce improved curing apparatus have not met with popular acceptance.

The best quality of Pará rubber is classified as fine; i containing impurities it is classified as medium; whilst there is also a coarse quality or "Sernamby," which consists of uncured scraps.

About 7 lb. of rubber per day are obtained by each collector in the Lower Amazons, and about three times as much in the richer parts of the Upper Amazons. The season for collecting in the Lower Amazons is from about July—when the waters subside—to January or February. The collectors are chiefly Brazilian immigrants from neighbouring states, and half-castes, but their number is quite inadequate.

The scarcity of labour is the chief difficulty in the india-rubber industry, and the landowner is quite at the mercy of the collector. As a rule the landowner makes advances for outfit, food, &c. to the collectors in return for the rubber. This he sells on the collector's account, making a deduction of 20 per cent. for himself, and then makes further advances for food, &c. Another method adopted is for the landowner to lease a number of trees at a given sum, on the understanding that the lessee shall sell the india-rubber, and buy all the articles he requires from the landowner. Although the landowner's profits are apparently large, it has to be taken into account that 75 per cent. of the collectors to whom they have advanced money either desert or return to their homes ill.

The "Aviador" system is said to be the best means of getting at the rubber. The "Aviador" advances money and supplies in exchange for rubber, and the consul considers that Europeans who wish to engage in the industry, have the best hope of success on that system. He mentions, however, that it was tried some years ago by an American syndicate, but without success. The exploitation of the forests is entirely in the hands of Brazilians and Portuguese, and the general opinion in the country is that none but they can successfully manage an estate. The exporters of india-rubber are for the most part agents of British and American firms, and they procure their supply from the "Aviadores" in the principal centres. Very few "Aviadores" are direct exporters. Liverpool is the market for the Amazonian rubber exported to Europe.

The prospects for the india-rubber crop for 1898–99 are considered to be good, owing to the fact that the rainy season has been less wet than usual, and the collectors have thus been able to get to work at an earlier period than they generally do.—C. A. M.

## PATENT.

*Gutta-Percha, Impts. in Obtaining, and in the Utilisation of the By-Products arising therefrom.* R. J. Friswell and Brooke, Simpson and Spiller, Ltd., London. Eng. Pat. 19,729, August 26, 1897.

The leaves, &c. of the gutta-percha plant are macerated in esparto boilers, the product is treated as in the manufacture of paper pulp, and the stuff is made up into sheets like millboard. The gutta-percha can then be recovered with suitable solvents (*cf.* Eng. Pats. 19,727 and 19,728, 1897; this Journal, 1898, 775), and the residual boards used in the preparation of paper, &c. Or the material from the boilers may be diluted with straw or other pulp so as to yield a waterproof or insulating millboard having gutta-percha diffused throughout its mass. In this case before finishing, the paper should be passed through a quantity of some solvent sufficient to insure the even distribution of the gutta-percha without extracting it.—F. H. L.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Tanning Materials, Practical Examination and Valuation of, by means of an Infusion.* J. von Schroeder. Chem. Centr. 1898, 2, [2], 143.

The author describes a method for the use of the practical tanner, who, up to the present time, has judged tanning materials solely by their appearance, smell, and by the masticating ("Kau-") and breaking tests. He considers it much better to estimate the strength of an infusion of the tanning material, by means of an accurately graduated Baumé hydrometer. The infusion should always be prepared in the same manner; that is, with oak and pine bark, 100 grms. of the finely divided material should always be taken, and with the remaining high grade tanning materials, 50 grms. The weighed sample is treated in a flask at 15° C., with 1 litre of water, and the mixture allowed to stand 24 hours, with repeated agitation. The liquid is then filtered and the strength of the filtrate determined by a hydrometer divided into $\frac{1}{50}$ degrees, so that the reading may be obtained within $\frac{1}{100}$° B.

The author has constructed tables as follows for the individual tanning materials—oak bark, pine bark, valonia, myrabolans, mimosa bark, divi-divi, algarobilla, and sumach.

| Strength of Infusion at 15°. | | Probable Content of Tanning Material. | | Probable Content of Extract. | |
|---|---|---|---|---|---|
| Degrees Baumé. | Sp. Gr. | Tanning Substance. | Per Cent. according to Löwenthal. | Portion Soluble in Cold Water. | Total Extract. |

—A. S.

*Depilation by Sweating and Liming, Von Schroeder's Work on the.* F. H. Haenlein. Dingler's Polyt. J., 1898, 65.

In this work it was demonstrated that a fresh skin or one preserved in salt, could be kept in the sweating room for several days longer than is necessary to loosen the epidermis and the hair, without its undergoing any appreciable alteration in appearance. Between the destruction of the malpighian layer by the Villon's bacteria and the work of destruction and putrefaction caused by other organisms which alter the qualities and aspect of the skin, a relatively long time elapses.

The presence of salt retards the depilation by sweating, therefore it is necessary that in the preparatory treatment salt should be thoroughly removed.

In the case of depilation by lime, the influence of the proportion of lime in the pits and the comparison with regard to the use of old and fresh limes, has formed the subject of considerable investigation. Practical tanners disagree as to the manner of conducting the process of liming, as to the quantity of lime in suspension and on the number of times the same pit may be used, as to whether the liming is better done by using one pit only or by passing the hides successively through series of old and fresh limes. In some tanneries hides are immersed in a thick bath of lime, in others the pit scarcely contains any excess of lime whatever. It is generally accepted that a perfectly new lime does not produce so good an effect as a mellow lime, that is to say, one which has been in use.

In these experiments the pits contained:—

No. I., 1,000 c.c. of water only.

No. II., 1,000 c.c. + 6 grms. of quick lime.

No. IIa., 750 c.c. water + 6 grms. quick lime and 250 c.c. old lime.

No. III., 1,000 c.c. water + 18 grms. quick lime.

No. IIIa., 750 c.c. water + 18 grms. quick lime and 250 c.c. old lime.

No. IV., 1,000 c.c. water + 30 grms. quick lime.

No. IVa., 750 c.c. water + 30 grms. quick lime and 250 c.c. old lime.

No. V., 100 c.c. water + 100 grms. quick lime.

No. VI., 100 c.c. water + 200 grms. quick lime.

Bath No. II. is the normal lime in the German School of Tanning and Experimental Tannery. Bath No. IV. is used in many tanneries. No. VI. is thick and pulpy, not unlike mortar. The pieces of skin experimented upon were examined after 3¼ days of immersion in the baths. In No. I the hair was perfectly tight. Baths No. II—VI showed equal results in the depilation of the skins, in each case the hair was removed with ease. No difference could be observed between the action of the fresh limes and in those in which an addition of old lime liquors had been made.

From these results, it appears that as long as the quantity of lime is between certain limits, it has no action whatever upon the duration of the liming, and the large quantities of lime used by some practical tanners are absolutely useless, and do not in any way shorten the time necessary for the softening and swelling of the skins.

The second series of experiments demonstrated that the presence of salt in the hide does not interfere with the liming process. That, however, does not mean that the tanner should not remove the salt from his hides before liming.

Von Schroeder's experiments demonstrate with certainty that the softening of hides and their preparation for depilation by the lime process are due solely to the alkaline action of the lime and are independent of the development or action of the bacteria.—J. G. P.

*Tanning, the Cause of Error in the Gravimetric Estimation of.* L. Maschke. Dingler's Polyt. J. 1898, 46.

*See under* XXIII., *page* 879.

### PATENTS.

*Tanning Apparatus, Improved.* H. Sefton-Jones, High Holborn, Middlesex. From E. Bardon, Paris. Eng. Pat. 20,134, Sept. 1, 1897.

This invention consists of a tanning agitator drum partly immersed in a vessel or pit containing the tanning liquid, which penetrates into the interior of the drum through orifices formed in the periphery of the latter. Inside the drum is placed the hides or skins to be tanned, the said drum effecting a tanning with a nominal motive power and with the relatively large quantity of tanning liquid. —J. G. P.

*Bateing of Hides and Skins, Impts. in and relating to the Means for the.* J. T. Wood, West Bridgford, Nottingham. Eng. Pat. 12,549, June 4, 1898.

The patent relates to a bate having the essential properties of the natural dung bate, but prepared according to scientific methods. At the present time skins are usually bated by means of a liquid or bate prepared from dog or pigeon dung. The present inventor has discovered that the bateing action of a dung bate is due to the combined action of enzymes or unorganised ferments and to certain amine compounds in the dung: also that the bateing action of the enzymes alone, or the action of the chemical compounds alone, are inefficient, and that the enzymes exert their bateing action in the presence of the chemical compounds, while these latter, in addition, have an independent action. According to this invention, the liquid or bate is made by producing by fermentation an enzyme or enzymes of the same character as the enzymes contained in a dog excrement or produced from bacteria obtained from dung, and to the liquid thus obtained there is added an organic acid and an alkali. For the production of these enzymes, the patentee has got good results by using the following as a nutrient medium: gelatin, 50 parts; potassium or sodium phosphate, 1 part; water, 2,500 parts. The medium is maintained at a temperature of about 30° C., until the required bacteriological action is complete, the time being about seven days. To each litre of the liquor thus obtained there is added 2 to 6 grms. of lactic acid. For bateing skins in a practical manner with the above bate, the method of using must be varied according to the skins treated and the kind of leather it is required to produce.—J. G. P.

*Branding Composition, An Improved.* H. Gibson, Ngaire, New Zealand. Eng. Pat. 13,476, June 16, 1898.

The present branding composition is intended to render unnecessary the tortures inflicted on animals, and damage to their hides, by the application of hot irons. The composition consists of equal parts of barium sulphide and of coal-tar, preferably thinned by a mixture of American potash and water in equal parts by measure, and of spirits of turpentine, each equal in measure to the original composition. Sulphides of the alkalis or alkaline earths may be substituted for the barium sulphide. "The liquid thus resulting is applied to the hair or wool of the animal to be branded by means of an ordinary sheep brand or other suitable branding tool. The hair or wool is destroyed, and the hide branded in a substantially permanent and conspicuous manner," and without injury to the hides of the animals.—E. S.

## XV.—MANURES, Etc.

*Rothamsted, Memoranda of the Origin, Plan, and Results of the Field and other Experiments conducted at. 55th Year*, 1898. J. H. Gilbert, 1—111.

*Rainfall and Drainage.*—The average annual loss (1877–78 to 1896–97) of nitrogen as nitrates in the drainage through 20, 40, and 60 ins. of uncropped soil, in its natural state of consolidation, amounts to 35·07, 30·83, and 33·87 lb. respectively, corresponding with nearly 2 cwt. of nitrate of soda. Of the nitrogen so lost, probably not more than about 5 lb. is derived from the rain-water. The loss of nitrogen as nitrates is very directly dependent on the amount and distribution of the rain and drainage. It is at present doubtful whether the loss of nitrates is diminishing, though there seems to be some indication of a decline in the amount of nitrates found.

*Experiments on the Mixed Herbage of Permanent Grass-Land, 43rd Year.*—The effect of purely nitrogenous manures (especially ammonium salts) is to reduce the total number of species, leguminous herbage being almost exterminated, whilst increasing the growth of the grasses. Non-nitrogenous manures (supplying abundance of phosphoric acid and potash) also reduce the number of species, though to a less extent, and favour the growth of leguminous herbage. The hay produced under the influence of minerals is the best; with mineral and nitrogenous manure combinep the average yield has been nearly three tons (first crop), but the hay is almost entirely gramineous. With exclusive, or almost exclusive, use of artificial manures, certain species are unduly forced at the expense of others. Dung should therefore be applied liberally; in addition, basic slag may be applied with advantage, and also nitrate of soda (1 cwt., or at most 1·5 cwt. per acre).

*Experiments on Barley, 47th Year.*—With mineral manure the produce is greater than without manure; nitrogenous manure alone gives a still greater increase; whilst minerals and nitrogen together give twice, or more than twice, as much produce as minerals alone. Phosphates, both alone and in conjunction with nitrogen, have a much greater effect than the salts of potash, soda, and magnesia. As compared with wheat, barley is more dependent on the surface soil, and therefore more generally requires the direct application of mineral manure (especially phosphates) than wheat under similar soil conditions.

*Experiments on Wheat, 55th Year.*—Without manure the average yield (over 44 years) was nearly 13 bushels per acre, which is above the average of the whole of the United States. With minerals alone, the increase is only slight. Nitrogenous manure alone gave a considerable increase, whilst in conjunction with minerals, the increase was much greater still. It is estimated that, irrespective of fluctuations due to season, the reduction in yield on the unmanured plot amounts to about $\frac{1}{8}$th bushel from year to year.

In the case of wheat (unmanured) alternated with fallow, it was found that whilst the yield is greater than when wheat is grown continuously, a given area of land yields more when the crop is continuous. This is attributed to loss by drainage of much of the nitrogen brought into an available condition under the influence of the fallow.

*Experiments on Leguminous Crops.*—Red clover grown continuously on the same land with mineral and nitrogenous manures yielded practically no crop after a few years Subsequently other leguminous plants, of different (both greater and less) root range were sown on the same land. The various plants grew luxuriantly for a number of years, yielding large amounts of nitrogen. Lucerne yielded an average of 160 lb. of nitrogen over 12 years. The surface soil gained, rather than lost, nitrogen.

*Experiments on Root Crops, 56th Year.*—Root crops depend on liberal application of nitrogenous as well as mineral manure. Without manure they rapidly revert to the uncultivated condition. In a rotation the restorative effect of their growth is due to the large residue of the manure applied for their growth, the considerable manurial value of the leaves which are returned to the land, and to the large proportion of manurial constituents returned as manure after the consumption of the roots by cattle.

The percentage of sugar in roots is generally greatest in small crops, whilst the amount per acre is greatest in large crops. The yield of roots and sugar depends greatly on the amount of nitrogen taken up. The nitrogen-content of feeding-roots is comparatively low, but increases with increased amount of available nitrogen in the soil, and is greatest the more luxuriant, and the less ripe, the crop.

*Experiments on Potatoes 23rd Year.* — The average production of roots over 20 years, was: without manure, 1 ton 11·5 cwt.; with ammonium salts, 1 ton 18·5 cwt.; with nitrate of soda, 2 tons 8 cwt.; and with complete minerals, 3 tons 6·75 cwt. Minerals and nitrate of soda together gave a yield of rather over 6 tons, whilst minerals and ammonium salts gave rather less. This difference is attributed to the greater availability and the more rapid distribution in the soil of the nitrogen of nitrate of soda. With farmyard manure (containing 200 lb. of nitrogen per acre), less produce was obtained than with ammonium salts, or nitrate of soda, containing only 86 lb. of nitrogen per acre.

Nitrogenous manures increase the percentage of nitrogen in potato tubers; with nitrogenous manures alone there is the lowest percentage of ash. The characteristic effect of nitrogen (in presence of sufficient mineral matter, especially potash), is to increase the amount of starch in the tubers. Without manure 1,100 lb. of starch was obtained per acre; with mineral manure, 2,000 lb.; and with complete manure, 3,400 lb.

In wet seasons more diseased potatoes are produced under the influence of nitrogenous manure than without manure. With liberal supply of nitrogen, the juice is relatively rich in nitrogenous and mineral matter, and with unfavourable weather, those tubers suffer most which have the richest juice.

The following papers have been published since June 1891 (see this Journal, 1891, 939):—

Series I.—Reports of Field Experiments, &c.

86. Results of Experiments at Rothamsted on the Question of the Fixation of Free Nitrogen. Abstract of paper read before the Agric. Chem. Section of the Naturforscher-Versammlung, at Halle, a. S. ("Nature," Nov. 12, 1891).

87. The Sources of the Nitrogen of our Leguminous Crops (J. Roy. Ag. Soc. Eng. 1891, [3], **2**, (this Journal, 1892, 253).

88. Allotments and Small Holdings (J. Roy. Ag. Soc. Eng. 1892, [3], **3**).

89. Home Produce, Imports, Consumption and Price of Wheat over 40 Harvest-Years, 1852–3 to 1891–2 (J. Roy. Ag. Soc. Eng. 1893, [3], **4**).

90. Rotation of Crops (J. Roy. Ag. Soc. Eng. 1894, [3], **5**).

91. Upon some Properties of Soils, which have Grown a Cereal Crop and a Leguminous Crop for many Years in Succession (Agric. Students' Gazette, N. S. **7**).

92. The Agricultural Investigations at Rothamsted, England, during a period of Fifty Years (U.S. Depart. of Agric., Washington; Office of Experiment Stations, Bull. No. 22, 1895).

93. The Rothamsted Experiments; being an account of some of the Results of the Agricultural Investigations conducted at Rothamsted, in the Field, the Feeding Shed, and the Laboratory, over a period of Fifty Years (Trans. Highland and Agric. Soc. of Scotland, 1895, [5], **7**).

94. The Depression of Corn Prices; and the Production of Wheat in some of the chief Exporting Countries of the World (J. Roy. Ag. Soc. Eng. 1896, [3], **7**).

95. The Royal Commission on Agricultural Depression and the Valuation of Unexhausted Manures (J. Roy. Ag. Soc. Eng. 1897, [3], **8**).

96. The Valuation of the Manures obtained by the Consumption of Foods for the Production of Milk (J. Roy. Ag. Soc. Eng. 1898, [3], **9**).

97. The Growth of Sugar-beet, and the Manufacture of Sugar in the United Kingdom (J. Roy. Ag. Soc. Eng. 1898, [3], **9**).

Series II.—Reports of Experiments on the Feeding of Animals, &c.

31. The Feeding of Animals, for the Production of Meat, Milk, and Manure, and for the Exercise of Force (J. Roy. Ag. Soc. Eng. 1895, [3], **6**).—N. H. J. M.

*Fertilisers in Horticulture, The Use of.* A. Hébert and G. Truffaut. Bull. Soc. Chim. 1898, **19**, [14], 644—651.

The amount of fertilisers required by a plant is measured by the difference between the nitrogen and mineral content of a healthy plant, and the available nitrogen and mineral constituents of the soil. The available nitrogen of the soil is determined by the quantity of nitrates formed by a given weight of the soil during a given time, the phosphoric acid and potassium by the quantities of these substances soluble in acetic or citric acid. In the experiments of which an account is given, the fertilisers consisted of neutral soluble salts only; they were applied in solutions of 0·03—0·05 per cent. strength at intervals of 7 or 14 days. In the case of *Dracæna Bruanti*, the soil was sufficiently rich in silica and alumina, lime was supplied in quantity by the soil and water. The fertiliser applied was composed of potassium nitrate, ammonium chloride and phosphate, magnesium and iron sulphates in such proportions as the plant required the different elements. The plants were contained in very small pots holding only about 330 grms. of soil. The plants treated as described with 25 grms. of fertiliser, were fine and well developed and weighed about twice as much as similar plants grown under ordinary conditions. The dried plants, treated and untreated, contained practically the same proportions of ash and nitrogen, and the ash had practically the same composition. In short, the method of assimilation is not modified by this process, but the yield is doubled.—A. C. W.

*Fertilisers: A Particular Method of Application by Diffusion.* A. Hébert and G. Truffaut. Bull. Soc. Chim. 1898, **19**, [14], 651—655.

It is to the advantage of plants that fertilisers should be applied in a form in which they are slowly assimilable. The authors employ compressed pastilles composed of fertiliser mixed with kaolin or clay and wrapped in metallic foil. Quantitative experiments show that the diffusion of the soluble substance is regular, and that it is proportional to the number of metallic envelopes. According to this number the time required for complete diffusion may vary from a fortnight to three months (three envelopes). The time required for complete diffusion is also dependent on the proportion of inert material.—A. C. W.

*The Phosphoric Acid dissolved by Soil Waters.* T. Schloesing, junr. Comptes Rend. 1898, **127**, [4], 236—239.

The solutions contained in a soil may be extracted by their displacement by water, poured slowly upon the soil in a regular rain. In such a solution the phosphoric acid is determined after evaporation of 1 litre in a flask, addition of nitric acid to remove hydrochloric acid, and evaporation to dryness in a platinum dish. Calcination follows, then heating with a little ammonium nitrate, extraction with dilute acid, and determination by ammonium molybdate after evaporation to a small bulk. In any one soil, in spite of variations from 5 to 25 per cent. in the original humidity of the soil, the amount of phosphoric acid contained by the soil water is constant. This constancy results from the equilibrium of reactions of opposite tendency, and from it a process may be devised for the estimation of phosphoric acid in soil-waters by working on not more than 1 kilo. of soil.—A. C. W.

*Phosphoric Acid Dissolved by the Waters of the Soil, Estimation of the.* T. Schloesing, junr. Comptes Rend. **128**, [6], 327.

*See under* XXIII., *page* 875.

*Phosphoric Acid in Superphosphates, A Volumetric "Citrate" Method for the Estimation of.* S. Littmann. Chem. Zeit. **22**, [68], 691.

*See under* XXIII., *page* 875.

*Phosphoric Anhydride, Estimation of Water-Soluble and Citrate-Soluble.* F. Martinotti. Staz. sperim. ital. **31**, 160; Chem. Centr. 1898, **2**, [2], 132.

*See under* XXIII., *page* 875.

*Phosphoric Acid, Determination of.* H. Lasne. Comptes Rend. **127**, [1], 62.

*See under* XXIII., *page* 874.

*Phosphoric Acid, Determination of.* L. Vignon. Comptes Rend. 1898, **127**, [2], 191.

*See under* XXIII., *page* 874.

## PATENTS.

*Artificial Manures, Impts. in the Manufacture of.* W. Wolters, Dresden, Germany. Eng. Pat. 21,175, Sept. 15, 1897.

The object of the invention is to produce citrate-soluble phosphoric acid compounds, for application as manures, from phosphorites and bone materials; and citrate-soluble silicic acid compounds from sand, &c. for the same purpose. With high percentage phosphorites, 100 parts are taken to 60 parts of alkaline-earth silicates, and 30 parts of an alkali silicate, and the mixture is melted at a high temperature in a Siemens' furnace, having a hearth formed of the materials themselves containing the phosphoric acid; or, on a small scale of working, the fusion may be effected in graphite crucibles. The alkali silicate may be replaced by glass, in which case the proportions of the ingredients recommended are, glass, 160 parts; chalk, 275 parts; and phosphorite, 250 parts.

To obtain compounds of citrate-soluble silicic acid, a mixture is fused of 100 parts of "crystallised sulphate of potash-magnesia," 25 parts of chalk, and 22 parts of sand.
—E. S.

## XVI.—SUGAR, STARCH, GUM, Etc.

*"Jaundice," a Bacterial Disease of Beetroots.* Prillieux and Delacroix. Comptes Rend. **128**, [6], 338—339.

This disease has been known for several years in the northern parts of France, and generally makes its appearance during the first fortnight in July; it seems to originate in the roots which have been grown for seeding purposes. When a plant is first attacked, the leaves begin to droop and lose their firmness. The outer leaves, and later the inner ones, become mottled with white, translucent spots. Subsequently, the difference between the white and green parts of the leaf diminishes, and the whole assumes a yellow hue, and finally withers. Roots which are badly diseased do not increase in size after the end of July, and although the percentage of sugar remains normal, the total yield may be diminished by one-half. Diseased roots, kept for seeding purposes and replanted in the spring, again yield plants showing all the previous symptoms, but the spike and flowers develop all the same. On microscopical examination, the white patches on the leaves are seen to be full of short, motile, barrel-shaped bacteria. The chloroplasts are decolorised and are less defined than in the healthy cells, and the granulations are more refractive. On diseased seed-beets the bacteria are found not only in the leaves but in the floral bracts and calix, and probably persist as spores in the beet seeds.

The authors have made cultures of the bacteria of beet root jaundice and have produced therewith artificial infections. Young, healthy beet plants, watered with a pure culture of the bacteria, readily contract the disease, whilst similar plants, grown under normal conditions, remain sound. Beets, grown from healthy seeds in soil which has been mixed with the dried leaves of diseased plants, also become infected.—J. F. B.

*Saccharine and Astringent Exudations of the "Grey-Gum" Eucalyptus Punctata DC., and a Product allied to Aromadendrin.* H. G. Smith. Proc. Royal Soc., N.S. Wales, **31**, 177—191.

The author found that when the puncture has not penetrated entirely through the bark, and a flow is set up, the exudation from the trees of the "Grey Gum" is quite white and consists largely of raffinose (melitose). When the puncture has just penetrated through the bark, the exudation becomes dark, and is contaminated with tannic acid and eudesmin, thus indicating that eudesmin is present in the cells of the tree with tannic acid. From a series of experiments it was found that: The manna is derived from the bark of the tree, and the kino from the wood and not from the bark. The principal sugar in the exudations is raffinose, but the dark exudation contains at least two other sugars, one of which is more easily fermentable than the other. Those eucalypts which do not contain eudesmin, &c. in their kinos, do not give manna. Some of the Eucalypts belonging to the Renantheræ contain a yellow dye in their leaves allied to quercetin, and in some respects to aromadendrin. The kino of *Eucalyptus punctata* contains eudesmin, but not aromadendrin.—A. S.

*Chagual Gum, The Glucoses (inactive Galactose and Xylose) Derived from.* E. Winterstein. Ber. **31**, [10], 1571—1573.

The various kinds of gums hitherto investigated consist of anhydrides of glucoses (galactose, xylose, arabinose), and of other substances whose composition is but little known. They are all products of dicotyledonous plants. Since chagual gum is obtained from a monocotyledonous plant, the author thought that it might contain substances differing from those of the other gums. The smaller homogeneous fragments, which were partially soluble in a large quantity of water, gave a solution which did not reduce Fehling's solution, and which rotated the beam of polarised light but slightly to the right. On oxidation with nitric acid (sp. gr. 1·15), the gum yielded 21·25 per cent. of mucic acid, and on distillation with hydrochloric acid (12 per cent.) produced 23·95 per cent. of furfural. The greatest yield of glucose was obtained by boiling the gum for four hours with 5 per cent. sulphuric acid.

In order to isolate the glucoses produced by the hydrolysis, 50 grms. of the gum were boiled under a reflux condenser with a litre of 5 per cent. sulphuric acid, the solution nearly neutralised with barium hydroxide, the barium sulphate filtered off, and the filtrate boiled with animal charcoal and evaporated to a syrup at a gentle heat. The syrup was boiled with 90 per cent. alcohol, and then twice with 70 per cent. alcohol. The residue was dissolved in water, decolorised with animal charcoal, and poured into a large volume of absolute alcohol. The resulting precipitate, when washed with alcohol and ether, and dried, yielded 16 grms. of a snow-white substance not yet examined.

The united alcoholic extracts on evaporation in a desiccator left a clear yellowish syrup, which was successively extracted with alcohol of 98, 90, 80, and 70 per cent. strength. The first extract left on evaporation of the solvent a residue from which, on twice crystallising from 95 per cent. alcohol, small clusters of needle-shaped crystals were obtained. These were identified as xylose.

The portions soluble in dilute alcohol yielded colourless crystals, which were purified by crystallisation from 98 per cent. alcohol, and subsequently from dilute alcohol. They gave an osazone melting at 204° C., and a hydrazone melting at 158° C., and on oxidation with nitric acid yielded 74 per cent. of mucic acid. Their optical rotation was $[\alpha]_{D20} = +4\cdot7^\circ$, but this was not constant in the separate fractions crystallising from the mother liquor, for values subsequently found for $[\alpha]_{D20}$ were $+11\cdot5^\circ$ and $+12\cdot8^\circ$. From the quantity of mucic acid obtained, the author came to the conclusion that the substance was a galactose, but that it could not be *d*-galactose on account of its low specific rotation. On the other hand the melting point of its osazone and hydrazone, and the quantity of carbon dioxide yielded on fermentation with yeast, agreed with Fischer's description of inactive *i*-galactose (Ber. **25**, 1247). A completely inactive preparation could not be obtained, probably on account of the presence of traces of *d*-galactose. The author believes this to be the first recorded instance of the formation of inactive galactose by the hydrolysis of a carbohydrate.—C. A. M.

*Starch, Estimation of, in Cereals.* C. J. Lintner. Zeits. f. angew. Chem. 1898, [32], 725.

*See under* XXIII., *page* 878.

### PATENTS.

*Saccharine Liquids or Solutions, An Improved Process for Electrolytically Purifying and Filtering.* C. A. Meygret, Paris, France. Eng. Pat. 17,315, July 22, 1897.

Electrical energy is employed in place of animal charcoal in the refining of sugar, and the apparatus consists of an electrolytic tank divided into parallel compartments by means of vertical partitions formed of canvas which has been steeped in fish glue, dried, and then dipped into a solution of tannin in chromic acid. Each alternate compartment is filled with the liquor to be filtered and a carbon anode, while the intermediate compartments are filled with water, and contain iron cathodes. On the passage of the current, the potash or other salts contained in the sugar are deposited on the cathodes.—G. H. R.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Beer Yeast, The Action of Oxygen upon.* J. Effront. Comptes Rend. **128**, [6], 326—327.

The author has noticed that pressed yeast, broken into small pieces, and exposed to the air, has the property of absorbing oxygen with a considerable evolution of heat. For instance, when 2 kilos. of yeast were exposed in a layer 37 cm. thick, a rise of temperature of 36° C. was observed after three hours. At the same time there was a large absorption of oxygen and liberation of carbon dioxide. The phenomenon is said to be caused by an oxidising enzyme present in the yeast cells.

Since the presence of other gases has no effect on the absorption of oxygen by yeast, it is suggested that this property may be utilised for the qualitative detection of oxygen when mixed with inert gases. The test is best carried out by passing the gas to be examined through a flask filled with alternate layers of yeast and pumice stone. If oxygen is present, a sensible rise of temperature is observed in the mass in the flask.—J. F. B.

*Yeast, Is the Removal of Fermentation Products of Practical Value for the Investigation of?* Allgem. Anzeig. für Brau. **14**, [24], 806—807.

Although it has been demonstrated by Prior that by completely ridding the fermenting wort of carbon dioxide, Saaz yeast, which otherwise leaves a considerable proportion of unfermented residue, has its fermentative powers greatly increased, it does not follow that such a course is desirable in practice. On the contrary, the author regards the "vacuum" process of fermentation as likely to depreciate the flavour of the beer, by removing the finer, aromatic products of fermentation, together with the hop-aroma; and he considers that when the recovery of fermentation carbon dioxide, for liquefaction, &c. is desired, it is better to remove only the excess, leaving the beer to ferment under ordinary atmospheric pressure.

Again, beer made by the vacuum process requires to be carbonated, and in view of this, as well as of the fact that there are other available means of accelerating primary fermentation, it appears unnecessary to resort to this process, more particularly since the gas re-introduced by carbonating is never retained by the beer so firmly as if absorbed under the normal conditions of fermentation, *i.e.*, by a quiescent surface under direct pressure of the gas. Where carbonating is necessary the author recommends an approximation to these conditions, by exposing the beer to the gas in flat vessels or half-filled horizontal pipes.—C. S.

*Soluble Ferment contained in Germinated Barley and Capable of Acting on Pectin.* E. Bourquelot and H. Herissey. Comptes Rend. 1898, **127**, [2], 191—194.

The authors find that when pectin obtained from powder of gentian is treated with diastase prepared from germinated barley, the product has the property of reducing Fehling's solution. Since neither saliva, which contains amylase, nor the liquid of *Aspergillus niger*, which contains amylase, trehalase, and other soluble ferments, has any action on pectin, it is probable that barley contains a special ferment which is capable of acting on pectin. (See also under XX., page 866.)—E. W. W.

*Malt: Effect of Storage in Sacks.* K. Michel. Allgem. Anzeig. für Brau. **14**, [24], 805—806.

The result of the absorption of moisture by malt kept in sacks or on the open floor is to lower the percentage yield of extract. Lermer's investigations have, however, shown that when stored in boxes and covered with malt culms, the absorption of water is greatly retarded, the percentage decreasing from 10·73 at the surface of the culms, to 7·83 in the malt immediately below, and falling progressively with the depth, so that at 7 ft. only 3·44 per cent. of moisture was found.—C. S.

*Wort, Influence of Mashing Methods on Constitution of.* E. Jalowetz. Gambrinus, **25**, [12], 489—491.

From a series of experiments, made in conjunction with Hubert, on the effect of varying the temperature and duration of the mashing process with different portions of the same malt, the author concludes that these two factors exert an important influence on the carbohydrate constituents of the wort, the general rule being that a quick, high-temperature mash gives a small yield of fermentable extract, and *vice versâ*. The alterations produced by changes in the conditions of mashing are such that though the final attenuation of two beers, mashed in different ways, may prove identical, this affords no guarantee that the unfermentable extract is qualitatively the same in each. And since this circumstance is sure to influence the flavour of the product, it should be the aim of the maltster to vary his methods so as to produce, from barleys of different composition, malts suitable for a uniform method of mashing—of course when the preparation of beer of constant type is in question. Only when the brewer is in possession of a malt exhibiting an unfavourable carbohydrate ratio should an alteration in the system of mashing be resorted to, the changes then made being regulated by the relative proportion of fermentable and non-fermentable matter in the malt, as revealed by analysis.—C. S.

*Hop Constituents: their Solubility and Retention in Beer.* Allgem. Anzeig. für Brau. **14**, [25], 841—842.

The fact that the bitter principles of hops can, by repeated solution in alcohol and reprecipitation of the hop resin by water, be separated from the latter, proves their distinct nature, and the solubility of this bitter principle demonstrates the great difficulty in the way of removing an excess of this substance once dissolved in the beer. At the same time this property indicates the practicability of washing bitter yeast, and so rendering it suitable for many other purposes.

The finer particles of hop resin thrown down by water from alcoholic solution form a very persistent haze, and the fact that further concentration causes some of these particles to coalesce and subside, shows that no special increase in the amount of resin dissolved from the hops can be expected, in practice, to result from the employment of boiling heat. —C. S.

*Wines, Effect of Manganese Salts on the Colour of.* P. Jacob. J. Pharm. Chim. 1898, **8**, [4], 163—167.

Wine treated with its own weight of manganese dioxide assumes a tint similar to that of rum, and appears blackish by reflected light. The taste becomes disagreeably earthy. The total acidity and volatile acids are somewhat diminished, whilst the cream of tartar and tartaric acid are considerably less. The action of 0·2 per cent. of potassium permanganate for five minutes, followed by the addition of 20 per cent. of animal black, and immediate filtration, gives a coloration exactly similar to that of white wines, but the colour rapidly fades until the wine becomes almost colourless. Analysis shows an increase in ash and alkalinity of ash, decrease of total acidity from 4·89 to 0·82, and disappearance of tartaric acid and cream of tartar. A better result is obtained by fining with white of egg after the action of the permanganate: a wine of permanently good colour is obtained, and the composition of the wine is little altered. After the action of permanganate an apparent increase in glycerin is found; this is due to formic acid resulting from the oxidation of tartaric acid.—A. C. W.

*Wines naturally Salty.* E. Bonjean. Bull. Soc. Chim. 1898, **19**, [15], 719—722.

The results of a series of analyses of grape juice, musts, and wines prepared by the author have shown that the juice of the grape from vines grown on a salty soil may contain as much as 4·5 grms. of chlorine per litre, which quantity persists in the must and the wine. Sodium and potassium salts exist in approximately equal proportions in the juice of these grapes; the potassium is partly precipitated during fermentation, and thus a preponderance of sodium may exist in an unsophisticated wine.—A. C. W.

*Wine from Bilberries and Honey, Preparation of.* J. Graftiau. Bull. Assoc. Belge des Chim. **12**, [3], 107.

In addition to the honey wines already described by the author (this Journal, 1897, 817), he has also prepared one by allowing a mixture of 12 litres of bilberry juice and 4 kilos. of honey to ferment spontaneously, the honey being necessary on account of the low sugar content (5 per cent.) of the fruit. The mixture was stored in a vinery, the temperature of which favoured the reproduction and development of the indigenous yeasts. Primary fermentation lasted for about a fortnight, and after the lapse of another week the liquid was racked into another recipient, which was then stored in the cellar about nine months to mature for bottling.

The finished wine is clear, rich in colour, and of agreeable flavour, exhibiting a strongly developed "bouquet" of bilberries. It contains a high percentage of alcohol, tannin, and a sufficient quantity of extract, and is therefore able to bear prolonged storage.—C. S.

*Phlobaphene of the Grape.* A. Girard and Lindet. Bull. Soc. Chim. 1898, **19**, [13], 583—584.

In a former paper (Bull. du Ministère de l'Agricul. 1895, 691) the authors noted the occurrence of a bitter substance in the skin, stalk, and pips of the grapes from different French vines, which was invariably found associated with tannin, and which they then termed "resinous matter." They have since proved its identity with the phlobaphene which Etti extracted from oak-bark and from hops, and which he regarded as the anhydride of tannin (Bull. Soc. Chim. **26**, 413; **35**, 619, **40**, 542). In order to obtain it in a fairly pure state, the stalks or pips are left in contact with ether at 53 C. until the liquid separates into two layers. The lower of these is withdrawn, evaporated *in vacuo* over calcium chloride, and the phlobaphene precipitated by the addition of water to the syrup. The precipitate is washed free from tannin, and dried by exposure on a porous tile, and finally, *in vacuo*, over sulphuric acid. As thus obtained, it is a brown powder but slightly soluble in cold water, more soluble in hot water, soluble in alcohol and ether and in alkalis, from which it is precipitated on the addition of acids. As is evident from its method of preparation, it is soluble in concentrated solutions of tannin, but precipitated on the addition of water. It is precipitated from its solutions by bromine-water, sodium chloride, and ammonium chloride. It gives a green coloration with iron salts, precipitates albumin and gelatin, and yields protocatechuic acid on fusion with potash. All these properties are characteristic of phlobaphene, and the formula deduced from its elementary composition ($C_{30}H_{30}O_{17}$) is the same as that given by Etti.

On boiling the acid solution of stalks and pips from which the phlobaphene has been extracted, a reddish substance is deposited, which also has the composition and properties of phlobaphene. All tannins, but especially oak tannin (Grabowski, Bull. Soc. Chim. **35**, 617; and Etti, *ibid.*, **40**, 512), yield an insoluble red compound when boiled with acids (Quercic Red, &c.). The authors have not found that reducing-sugars are formed under these conditions, though Etti and others have attributed the reduction to the presence of levulin. The sum of the quantities of tannin and phlobaphene is often a constant, any decrease in the amount of the former being balanced by an increase in the latter, and *vice versâ*. The authors consider that it is not improbable that the reserve tannin of the plant is in the form of the anhydride phlobaphene.—C. A. M.

*Volatile Acids produced in a Fermentation, Formula for Estimating by the Duclaux Method.* Bordas and Raczkowski. La Bière, **6**, [6], 87.

*See under* XXIII., *page* 879.

*Methyl Alcohol in Ethyl Alcohol, Recognition and Estimation of.* A. Trillat. Comptes Rend. 1898, **127**, [4], 232.

*See under* XXIII., *page* 879.

*Spirits, Detection of Renatured Spirit in.* H. Herzfeld. Zeits. für öffentl. Chem. **4**, 389; Chem. Centr. 1898, **2**, [2], 133.

*See under* XXIII., *page* 876.

*Malic Acid, Determination of, in Grape Juice.* A. Girard and Lindet. Bull. Soc. Chim. 1898, **19**, [13], 585.

*See under* XXIII., *page* 877.

### PATENTS.

*Alcohol by Saccharification and Fermentation by Mucedineæ, and Apparatus therefor; Improved Process for the Manufacture of.* A. Collette, fils, and A. Boidin, Seclin (Nord), France. Eng. Pat. 13,053, June 10, 1898.

This has reference to a previous specification (No. 19,858; this Journal, 1898, 483), and has the object of rendering the process there described continuous. The mash, sterilised by the heat of steam under pressure, passes through a refrigerator into the first of a series of vessels, where it is inoculated with the required mould. As soon as the liquid reaches a certain level, it passes on into the second vessel, where the same mould may be allowed to continue to act or a fresh mould introduced at will. In like manner it passes into the third vessel, and so on, throughout the series, which in the illustration consists of five vessels. The introduction of the moulds, or moulds and yeast, is so arranged that the saccharification takes place in the earlier vessels and the fermentation in the later, the liquid leaving the last vessel completely fermented. The interior of the steriliser, refrigerator, and vessels, and their connecting pipes, are all rendered sterile before the commencement of the process; and in order to avoid any infection through the valve of the steriliser not fitting quite tightly, two valves are placed on its discharge tube, and steam pressure in excess of that received by the steriliser introduced between them.—C. A. M.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A*.)—FOODS.

*Millet, The Composition and Alimentary Value of.* Balland. Comptes Rend. 1898, **127**, [4], 239—240.

The following table gives the maximum and minimum quantities found of each constituent after an examination of the principal varieties of millet:—

| —— | Minimum. | Maximum. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Water | 10·10 | 13·00 |
| Nitrogenous matter | 8·98 | 15·04 |
| Fatty matter | 2·20 | 7·80 |
| Sugars and starchy matters | 57·08 | 66·33 |
| Cellulose | 3·00 | 10·23 |
| Ash | 1·40 | 6·00 |
| Acidity | 0·055 | 0·098 |

—A. C. W.

*Furfural from Starch and its Derivatives, Formation of.* F. Sestini. L'Orosi, **21**, 109—113; Chem. Centr. 1898, **2**, [3], 182

If bread crust be heated in a tube, furfural begins to form at 110°—115° C.; if, on the other hand, bread crumb be heated, furfural does not begin to form till about 150° C. The author discusses the question whether the furfural in these experiments owes its origin to the admixed pentosans or to the starch. Gridkoff states that starch and cellulose form no furfural by distillation with dilute sulphuric acid, but the author shows that starch heated dry at about 200° C., and in presence of acid at about 100° C., yields furfural. Thus the furfural formed by heating bread, may arise from the starch as well as from the pentosans. As furfural is formed in not inconsiderable quantities by the dry distillation of carbohydrates, even in absence of acid, this circumstance should not be overlooked in the analysis of foods by Tollens' method.—A. S.

*Butter, Variations in Composition of.* C. Masson. Bull. Assoc. Belge des Chim. **12**, [3], 108—109.

In the course of an investigation carried on, under the auspices of the Belgian Government, in order to ascertain the fluctuations in the composition of butter, a sample analysed at Momignies gave the abnormal values: non-volatile fatty acids (Hehner), 91·2; volatile fatty acids (Reichert-Meissl), 20·0; refractometer index (Abbé-Zeiss) at 40° C., 46·2. A check sample prepared by the author from milk from the same dairy, ten days later, gave the corresponding values as 90·9, 22·2, and 44·5 respectively, figures which, according to the table published by Girard, would indicate an adulteration with 42·5 per cent. of margarine. The values determined in a butter of the same origin six weeks afterwards were: non-volatile fatty acids, 87·5; volatile fatty acids, 35·6; Abbé-Zeiss index, 42·5.

From these differences the author concludes that the degree of purity of a butter cannot be reliably computed from its chemical composition alone.—C. S.

*Margarine, Denaturing.* J. Wauters. Bull. Assoc. Belge des Chim. **12**, [2], 58—69.

After reviewing the various methods proposed for the denaturing of margarine and the evidence gathered by André in respect of same, the author expresses the opinion that the addition of sesamé oil is the one most likely to be adopted in Belgium, although a better quality product can be obtained by the use of earthnut (arachis) oil, which will be precluded because the addition of the two kinds of oil is impracticable. Phenolphthalein he regards as unsuitable, owing to the existing doubts as to its harmlessness; and he would prefer the obligatory employment of some such colouring matter as turmeric, which cannot be removed without altering the character of the margarine, whilst the use of this pigment for butter could be prohibited, thus preventing the adulteration of the latter product by the former. In any case the repression of fraud is only possible by international agreement respecting the means of denaturation to be adopted.—C. S.

*Natal Tea, Note on the Analysis of Five Samples of.* C. E. Sage. Pharm. J. 1898, [1466], 106.

Five authentic samples of Natal tea afforded the following analytical figures:—

| Sample. | 1. | 2. | 3. | 4. | 5. |
|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Caffeine | 2·95 | 2·78 | 3·34 | 2·94 | 3·03 |
| Tannin | 8·1 | 10·8 | 10·7 | 8·9 | 6·7 |
| Vegetable extractive matter. | 24·61 | 23·54 | 19·32 | 24·91 | 22·62 |
| Inorganic extractive matter. | 4·24 | 4·45 | 4·95 | 4·46 | 4·38 |
| Total extracted by water.... | 39·90 | 41·57 | 38·31 | 41·21 | 36·78 |
| Moisture | 8·19 | 7·95 | 9·19 | 9·36 | 9·57 |
| Ash | 5·30 | 5·25 | 5·51 | 5·63 | 5·26 |

The designation of these samples was: No. 1, "Flowery Pekoe"; No. 2, "Golden Pekoe"; No. 3, "Pekoe Souchong"; No. 4, "Pekoe"; and No. 5, "Broken Pekoe."

The percentage of tannin in these teas was found to be much lower than in Indian samples; in each case the ash contained a notable proportion of manganese.—J. O. B.

## PATENTS.

*Food for Animals, The Production of a.* C. Estcourt, Manchester. Eng. Pat. 19,979, Aug. 31, 1897.

Claim is made for a mixture of a saccharine material, such as molasses, with an albuminous substance, such as dried or clotted blood, either by themselves or mixed with an absorbent material, such as bran or brewers' grains. Suitable proportions are one part of albumin or dried blood, with one part of molasses, and one part of absorbent material. It is stated that such preparations can be kept for a considerable time without undergoing alteration. —C. A. M.

*Food for Human Consumption, The Production of a New.* C. Estcourt, Manchester. Eng. Pat. 19,980, Aug. 31, 1897.

This food consists of an albuminous substance, preferably fresh blood, mixed with a saccharine substance, such as molasses, treacle, or "natural or artificial sugars." Suitable proportions mentioned are 20 parts of molasses, or its equivalent of saccharine matter, with 80 parts of fluid blood, or its equivalent of albuminous matter. The mixture can either be used alone or in combination with other food substances.—C. A. M.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Water, Purification of, for Drinking Purposes.* P. Guichard. Bull. Soc. Chim. 1898, **19**, [13], 588—592.

In the author's opinion all water intended for drinking ought to be purified by the consumer immediately before use. Filtration through porcelain candles has been widely adopted for the purpose, but has the drawback that unless the filters are frequently cleansed, they become ineffectual, and after a few days' use allow bacteria to pass through. Moreover, the filters do not remove any noxious soluble organic compounds that may be present. Hence the author prefers to use a chemical method of purification before filtration. Girard and Bordas (this Journal, 1895, 881) recommended treating the water with calcium permanganate, and then filtering through a mixture of animal charcoal and lower oxides of manganese. The permanganate is decomposed by the organic matter with the formation of calcium carbonate and manganic oxides, which are reduced by the organic matter or by the charcoal to manganous oxides. The author finds that the addition of the manganese oxides is superfluous, and that filtration through animal charcoal alone is quite sufficient to purify the water after the treatment.

He has examined the action of permanganates, and especially of calcium permanganate, on various kinds of organic matter. Gum causes reduction in the cold, but the oxide of manganese produced remains in solution, probably in the form of a compound similar to that formed by iron oxide with saccharine matter. Starches reduce calcium permanganate rapidly at first, but the rapidity of reduction soon decreases. Cellulose and allied substances cause hardly any reduction, and the addition of precipitated manganic oxide and of charcoal is without influence on the result. Rice and millet grains and powdered cork reduce calcium permanganate very rapidly, and the reduction process continues for several days. The author attributes the great reducing power of these substances to the presence of nitrogenous compounds. Gelatin causes reduction and forms a soluble compound with the reduced manganese oxide. Coagulated white of egg forms a similar compound, which is decolorised by animal charcoal, whilst in the case of the organic substance of bone—"osseine"—the reduced oxide remains insoluble.

Animal and vegetable charcoal cause great reduction especially the former; but the reducing process is not indefinite, and ceases after the addition of a certain quantity of permanganate. In the experiments made by the author the following amount of reduction was caused by these charcoals after boiling for 10 minutes in a neutral liquid :—

| | Permanganate reduced. |
|---|---|
| 1 grm. of charcoal (poplar) washed ............ | 0·072 |
| „ animal charcoal not washed .......... | 0·22 |
| „ „ „ recalcined. | 0·05 |
| „ „ washed ............. | 0·10 |

Animal charcoal, on treatment with hot or cold water, yields organic substances which reduce permanganate. According to Bobierre, animal charcoal has the following percentage composition :—Carbon (containing from 6 to 7 per cent. of nitrogen), 10·8 ; calcium phosphate, 81·7 ; carbonate, 3·0 ; silica, 2·8 ; alumina and iron, 0·7 ; magnesia, 0·2 ; soluble salts, 0·8. As a rule it contains from 7 to 10 per cent. of water. According to some authorities, animal charcoal, even when recalcined, always retains a little organic matter, which can only be removed by fusion with potash ; but, according to others, it loses all its organic matter on calcination.

Precipitated oxide of manganese does not reduce permanganate in the cold, either alone or with charcoal. A great many metals cause more or less reduction, but as a rule the reduction is very slow, as in the case of magnesium, aluminium, zinc, copper, tin, and lead. Iron, however, which reduces permanganates but slowly at first, gives more and more rapid reduction as the metal becomes oxidised The author attributes this to the formation of ferrous oxide or to the hydrogen formed by the action of the iron on the water. In addition to calcium carbonate, a rust is formed during the reaction in which ferrous oxide can be detected, and also manganic oxide, though the latter does not appear to be a necessary factor in the process. The liquid does not retain either iron oxide, organic matter, or manganese, though sometimes very slight traces of iron can be detected by means of sulphocyanide after the oxidation. Attempts to accelerate the reaction by coupling the iron with another metal, such as tin, copper, or zinc and copper, did not give as good results as the iron by itself.

In the application of this reaction to the purification of water on the small scale, the author adds calcium permanganate in excess and then filters the water through a bed composed of finely granulated pumice stone mixed with iron filings or granulated iron. As soon as the iron becomes rusty (*i.e.*, in about two days), the filter bed becomes active and decolorises the water. Using a Mariotte flask with a tubulure near the bottom as the filtering flask, it is possible to obtain not less than 4 litres of purified water in 12 hours by this process.—C. A. M.

*Lead Pipes, The Cause and Prevention of the Action of Water on.* A. Liebrich. Zeits. angew. Chem. 1898, [30], 703—704.

The water-supply, the action of which on the leaden conduit pipes was the immediate object of the author's experiments, was derived from sandy strata beneath marshy ground near the North Sea. It was characterised by an absence of carbonates and by its richness in organic matter and nitrates. It had the following composition in mgrms. per litre :—Dry residue, 350 to 380 ; residue on ignition, 170—190 : chlorine, 30 to 40 ; nitric acid, 40 to 60 ; sulphuric acid, 23 to 27 ; lime, 25 to 30 ; magnesia, 8 to 15 ; silica, 20 to 28 ; ammonia, 0 ; nitrous acid, 0 ; oxygen, 14 ; free carbonic acid, 57. The residue obtained by evaporating a litre down to a few c.c. had a faintly acid reaction, which was attributed to the presence of organic (humic) acid.

The author confirms Muller's statement as to the simultaneous presence of oxygen and carbon dioxide being the chief essential factors in the action of water upon lead. By removing the oxygen with sodium sulphite, the carbon dioxide, even when present in large excess, has no influence, and *vice versa*. The view that a small quantity of carbon dioxide exercises a restraining influence is regarded by the author as incorrect, for he finds that a complete removal of the free acid gives the best results. Whilst recognising the fact that a soft water has a natural tendency to attack pure lead, he states that there is also no doubt that a hard water in the presence of carbon dioxide and air can also dissolve lead, though the quantity dissolved is only trifling.

It was found experimentally that the water in question when left in contact with tubes of pure lead for 12 hours, dissolved over 30 mgrms. of lead per litre. Addition of an excess of calcium carbonate to combine with the free carbon dioxide did not completely prevent the action, the water still dissolving several mgrms. after standing over night. Experiments were then made in which sodium carbonate was substituted either partially or completely for calcium carbonate with the object of obtaining a more rapid neutralisation.

From these experiments the author concluded that with that particular water, at any rate, the addition of sodium carbonate was more effectual than that of calcium carbonate. The practical application of this conclusion to the treatment of the water on the large scale was quite successful, and by the addition of a quantity of sodium carbonate approximately sufficient for neutralisation, the action of the water on the lead was prevented without the water being rendered alkaline.—C. A. M.

*Sewage, Filtration of; First Report : The Bacteriological Examination of London Crude Sewage.* F. Clowes. Issued by the London County Council, June 16, 1898.

This paper is the first report to the Main Drainage Committee on some experiments which are being carried out by the author and Dr. A. C. Houston on the treatment of London sewage by filtration through coke. Pending the installment of the filters, a thorough examination of the nature and number of the organisms present in the crude unfiltered sewage at Barking and Crossness has been made. A description is given of the experimental methods employed for the cultivation and isolation of the different forms of bacteria met with in sewage, and a summary of the results obtained follows, together with details of the experiments arranged in a tabular form.

The sewage at Barking contained on an average nearly four million bacteria per 1 c.c. and 332 spores, whilst that at Crossness contained three and a half million bacteria, and 365 spores per 1 c.c. The large quantity of factory refuse which finds its way into London sewage would therefore appear to have no inhibitive effect on the growth of bacteria.

The number of liquefying bacteria present in the sewage was rather difficult to estimate, as they often liquefy the gelatin plate before they can be counted. It is estimated that London crude sewage contains over 400,000 liquefying organisms per 1 c.c. Probably, though not necessarily, these organisms which liquefy gelatin play a leading part in disposing of the solid matters of the sewage, and a study of them is for this reason of special importance. Many organisms are capable of acting under both aerobic and anaerobic conditions, and in the disposal of sewage, aerobic putrefaction is to be preferred because the gaseous products are less offensive.

Among the species found and recognised were :—*B. Enteritidis Sporogenes*, which was fairly numerous, *B. Coli Communis* in very large numbers, large quantities of various "protean forms," differing somewhat from *Proteus Vulgaris*, which rapidly liquefy gelatin with the evolution of gas. Besides these, *B. fluorescens liquefaciens* and *non-liquefaciens* were present, but comparatively rare, and in some cases they could not be isolated. Bacteria closely allied to *B. Coli Communis* of the same group but differing in details were also found, and small quantities of *B. Mycoides*, *B. Subtilis*, and *B. Mesentericus* were detected. The occurrence of Sarcinae, Yeasts, and Moulds was rare.—J. F. B.

*Nitrites, Estimation of, in Water.* D. de Paepe. Bull. Assoc. Belge des Chim. 12, [3], 98.

*See under* XXIII., *page* 875.

## PATENTS.

*Sewage or other Liquids, Impts. in Apparatus for the Treatment of.* D. Cameron, F. J. Commin, and A. J. Martin, all of Exeter. Eng. Pat. 22,965, Oct. 6, 1897.

This invention embodies improvements in the apparatus described in the patents 3003 and 23,012 of 1896 (this

Journal, 1897, 459 and 753). It comprises the following claims in combination with two or more receptacles:—(1.) A means of controlling the supply and discharge of each receptacle consisting of a pair of independent buckets, so constructed that the overflow of one receptacle fills the bucket whose descent actuates the supply valve of a second receptable; whilst the other bucket which actuates the discharge valve of the receptacle is filled by the overflow of that receptacle. (2.) An aperture which regulates the flow into such bucket. (3.) A means of varying such aperture, such as a cock or detachable nozzle. (4.) A regulating cock or nozzle on the pipe which supplies the bucket actuating the discharge valve. (5.) A series of overflow pipes each fed from one receptacle, and conveying liquid which works an apparatus supplying another receptacle, so that the several receptacles are filled and discharged in rotation. (6.) The connection of such overflow pipes into a continuous ring having branches with 3-way cocks to the several receptacles, so that the supply to any one of them may be cut off at will. (7.) Valves or cocks to cut any one receptacle out of the series. (8.) A check valve to prevent liquid passing back from the continuous ring into a receptacle thus cut out. (9.) A check valve or valves on the overflow pipe or pipes of the receptacle cut out of the series, which without interfering with the overflow prevents the return of the overflowing liquid into that receptacle.
—C. A. M.

*Filtering Water, Impts. in Apparatus for.* A. Puech, Mazamet (Tarn). Eng. Pat. 11,049, May 14, 1898.

An arrangement of plant for filtering large quantities of water. Several compartments or tanks are arranged in groups or separately and at the same level, and are preferably rectangular in shape. Each tank has a false bottom of perforated sheet-metal carried by masonry or iron supports. The bottom is of lime, concrete, or cement, and slopes rapidly down to one end, and from the sides towards the centre. At each inlet and outlet end is a masonry trough separated from the compartment by a partition having a valve or valves at its lower part. The holes in the false bottom vary in size in each compartment. The filtering material is gravel, and the particles forming the layer for each compartment also vary in size. The water to be filtered passes in succession through each compartment, passing first through the coarsest layer, and lastly through the finest. The arrangement of valves is such that the beds may be cleaned whilst in position, and thus used indefinitely without removal, directions for doing this being given. The filtering process is not stopped when one compartment is being cleaned out.—R. S.

## XIX.—PAPER, PASTEBOARD, Etc.

*Waste Liquor from Sulphite Wood-Pulp Manufacture.—IV.* H. Seidel and L. Hanak. Mitt. des tech. Gewerbe-Museums in Wien, **8**, 1898, [11 and 12], 337—347.

In continuation of their previous work (this Journal, 1898, 596) the authors now find that free furfurol is a not inconsiderable constituent of the sulphite liquor, its presence being identified by extracting with ether, adding aniline, and after evaporating the ether, adding hydrochloric acid, when the furfurol reaction is distinctly obtained. Hence the previous statements require modification, since the whole of the furfurol present does not originate from pentoses or pentosans. The presence of these substances is, however, proved by the phloroglucine reaction, and by the fact that after distillation with hydrochloric acid, the following substances gave furfurol:—thick liquor (sulphite liquor evaporated to 28° B.), the solid precipitated by salt or by alcohol, the dry residue from the thick liquor, and finally the dry residue of the filtrate after precipitating with alcohol.

In order to remove sulphur from the organic substance contained in the liquor, the latter was treated with bleaching powder on the water-bath, filtered from gypsum, barium chloride added to the filtrate, the barium sulphate filtered off, and the solution evaporated. The product obtained is exceedingly hygroscopic, is free from sulphur and reduces Fehling's solution strongly. The quantity of oxidising agent necessary for this operation is too large to allow the method to be employed for rendering the sulphite liquors innocuous. Experimental evidence showed that chemically the recovery of the sulphur is a simple matter, but that advantage could only be taken of this technically when it is possible to utilise the organic substance of the sulphite liquor for heating purposes. With regard to the reducing action of the sulphite liquor, this is not due to the sulphurous acid, but to the organic substance containing sulphur. Sulphite liquor reduces completely Methylene Blue, Indophenol, and indigo, and gives with 1·1′ dinitronaphthalene a lilac, and with the 1·4′ derivative a brown dyestuff (see under VI., page 844). A further use for sulphite liquor consists in employing the organic sulphur compound as a substitute for cream of tartar on alumina and chrome mordants for wool.

Various schemes have been proposed in order to render the sulphite liquor harmless, either by rendering the lye innocuous by evaporation, by recovering the sulphur, or by utilising for other purposes the organic substances formed from the non-cellulose portions of the wood in the boiling process. According to the first method, patents have been taken out in Germany for treating the liquor with lime, saturating with carbonic acid, and evaporating the filtrate in a vacuum or multiple-effect apparatus, or else by employing a particular form of evaporating plant. The liquor is evaporated to about 30° B., when it contains more than 50 per cent. of dry substance, and is mixed with wood shavings and burned. In order to effect this evaporation, about nine times the quantity of water has to be evaporated compared with the dry substance obtained, and the calorimetric value of the fuel produced is only about 60 per cent. of that of a medium coal. With regard to the recovery of sulphur, most investigators have assumed that the liquor contains considerable quantities of free and combined sulphurous acid. An exception is Drewsen's method (Ger. Pat. 67,889), in which the liquor is heated with caustic lime under a pressure of six atmospheres, when the residue, consisting of calcium sulphate and monosulphite, together with insoluble organic matter, is employed in place of limestone for the preparation of fresh bisulphite lye. The filtrate is allowed to run to waste. The utilisation of the liquor for other purposes apart from such suggested uses as for cattle food, conversion into the benzoyl compound, or the formation of an antiseptic product by condensation with formaldehyde depends upon the isolation of a tanning or a sizing material. It is well known that ferric chloride gives a decided reaction with sulphite liquor, and hide powder absorbs 28 per cent. of the dry residue. Mitscherlich, by precipitating sulphite liquor with a solution of inferior horn, claws, hoofs, &c. (in water at 100° C.) obtained a substance which dissolved in soda is used for sizing purposes. Cross and Bevan (Eng. Pat. 1548 of 1883) obtained a similar product from sulphite liquor and glue, which they term gelalignosine. Ekman, by salting out, obtains what he terms dextrone, identical with the product formed by precipitation with alcohol. Owing to its powerful reducing properties it cannot be used in calico printing as a substitute for dextrin.—T. A. L.

*Waste Liquors from Sulphite Wood-Pulp Manufacture (Lignorosin).—V.* H. Seidel and L. Hanak. Mitt. des tech. Gewerbe-Museums in Wien, **8**, 1898, [11 and 12], 348.

*See under* VI., *page* 844.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Phosphotungstic Acid, Pure; Preparation of.* E. Winterstein. Chem. Zeit. 1898, **22**, 539.

To a solution of 4 kilos. of sodium tungstate (as free from carbonate as possible), in 4 litres of hot water, 1 kilo. of sodium phosphate (free from ammonia) is added, and the whole is boiled until all the phosphate has dissolved. The solution, whilst still warm, is slightly acidified with a mixture of sulphuric acid and water (1 litre of each), and evaporated until a thin crystalline skin is formed on the surface. After

24—36 hours the syrupy liquid is poured through a sieve to separate the sodium sulphate. When sufficiently concentrated, the liquid has the sp. gr. 1·8 to 2.

200—300 c.c. of the solution are treated with twice their volume of ether, and, after the gradual addition of 70 c.c. of well-cooled 70 per cent. sulphuric acid, are well shaken, light yellow, oily drops of an ethereal solution of phosphotungstic acid fall to the bottom and are run off. Addition of sulphuric acid is continued as long as the oily drops are produced. Addition of acid before ether, and also addition of much acid at a time, are to be avoided, otherwise an emulsion is formed, and the separation of the ethereal solution more or less completely hindered.

The united ethereal extracts are again extracted with ether after addition of water, and finally steam-distilled on a water-bath. Chlorine is then passed through the residue until this becomes light yellow. Most of the phosphotungstic acid separates on cooling, and the rest is obtained after evaporation. The final, coloured, mother-liquors may be utilised in subsequent preparations. The yield is 2·3 kilos. of acid from 4 kilos. of tungstate.

The considerable increase in the price of pure tungstates makes it desirable to obtain the free acid from the tungstates after treating the acid with lime or baryta. This could be done by decomposing these residues with sulphuric acid, filtering from the sulphates, and extracting with ether as described.—N. H. J. M.

### *Bismuth Oxyiodide.* E. Sohet. J. Pharm. Chim. 1898, **8**, [6], 127—128.

Reynold has recommended bismuth oxyiodide as a substitute for iodoform in certain cases, and Matlack, of Philadelphia, generally confirms the recommendation. The oxyiodide is a brownish red powder, which under the microscope is seen to consist of small cubic crystals. It is insoluble in chloroform, ether, alcohol, carbon bisulphide, and a solution of potassium iodide. It can be prepared as follows:—(1.) Bismuth iodide prepared by subliming bismuth with iodine, is boiled with water, filtered, washed with cold water, and dried. (2.) By Mayo's method, 306 parts of bismuth subnitrate are rubbed with 165·6 parts of potassium iodide and sufficient dilute nitric or hydrochloric acid to form a brownish black paste, which is then diluted with water and allowed to remain at least four hours before filtering. (3.) The English process consists in pouring a solution of 306 parts of bismuth subnitrate into 900 parts of pure nitric acid, to which sufficient warm water has been added to just cause turbidity, into a warm solution of 165·6 parts of potassium iodide in 2,500 parts of water. The precipitate is at first brown, but on washing, becomes clearer. The potassium iodide should be in slight excess. This method is the best, and gives a product of characteristic colour, containing the calculated amount of bismuth.

—E. W. W.

### *Absolute Alcohol, Use of Calcium Carbide for the Preparation of; and Detection of Water in Alcohol, Ether, Chloroform, &c.* D. Vitali. Boll. Chim. Farm. **37**, 257—261; Chem. Centr. 1898, **1**, [24], 1225. (See also this Journal, 1898, 180.)

The author recommends the use of calcium carbide for the preparation of absolute alcohol, and in order to remove the unpleasant odour which arises from the solution by the alcohol of the impurities which are developed together with the acetylene, he treats with nitrate of mercury, filters, and distils over dry potassium carbonate.

It is stated that water in alcohol, ether, chloroform, &c. may be detected by treating with calcium carbide, and passing any acetylene evolved into ammoniacal silver nitrate solution.—A. S.

### *Hypophosphites, the Official; The Characters and Tests of.* H. A. D. Jowett. Pharm. J. 1898, 171—176. Read at the Brit. Pharm. Conf. at Belfast, August 1898.

The author finds by experiment that all previously published methods for the determination of hyphosphorous acid and its salts are unreliable; the only one which takes cognizance of the most common impurity—phosphorous acid or its salts—being that of C. T. Tyrer (this Journal, 1897, 764), in which barium chloride is employed to precipitate the impurities. The author finds, however, that barium phosphite is not entirely insoluble, and substitutes lead acetate as the precipitant. The lead salts of all the usual impurities are completely precipitated by this reagent, the excess of lead is removed from the filtrate by $H_2S$, and the hypophosphite remaining in solution is oxidised into phosphate by heating the concentrated solution with potassium chlorate and excess of hydrochloric acid. The amount of phosphoric acid thus obtained is determined by the usual analytical methods, that of titration with standard solution of uranium acetate being most generally applicable. With ferric hypophosphite, a satisfactory method was not available for eliminating the phosphite previous to oxidising the hypophosphorous acid; reliance had therefore to be placed on the qualitative lead test, before conducting the quantitative examination.

For the purpose of experiment, pure specimens of the hypophosphites met with in commerce were prepared.

*Pure Sodium Hypophosphite.*—This salt was obtained in long, transparent, prismatic crystals by recrystallising the commercial salt from strong alcohol. These contained one molecule of water—and were very deliquescent—more so than the commercial granular form. The salts were not decomposed on heating for an hour to 110° C., but became anhydrous. They could be boiled without decomposition in water free from air.

*Potassium Hypophosphite* was purified in a similar manner: the calcium salt was freed from phosphite by treating its aqueous solution with lead acetate, removing excess of lead with $H_2S$, and precipitating the calcium hypophosphite by the addition of alcohol, when it separated in pearly flakes. The solubility of this pure salt was found to be 1 : 6·43 parts of $H_2O$ at 20° C. Although the salt may be boiled in water free from air, without undergoing oxidation, it was found that when evaporated to dryness in the ordinary way on the water-bath, the residue contained only 97·7 per cent. of real calcium hypophosphite. This may account for the phosphite present in many commercial specimens. In the case of the *iron hypophosphites*, the precipitates obtained by the double decomposition of ferrous sulphate with the hypophosphites of calcium or of barium were not satisfactory, being mixtures of the ferrous and ferric salt. When a ferric salt was employed, in the form of iron-alum, a white powder free from ferrous salt, was obtained. This is considered to be the most suitable for commercial use. In the course of the examination of the commercial products, specimens of sodium hypophosphite were found to contain from 99·7 to 91·5 per cent. of the anhydrous salt; potassium hypophosphite was met with containing a considerable amount of carbonate, two samples containing 2·8 and 3·7 per cent. respectively of that impurity. The commercial iron salt was the least satisfactory of any, one sample containing 12 per cent. of $CaSO_4$ and another 9 per cent. of $Na_2SO_4$. Most of the commercial specimens gave indication of traces of phosphite, and some also of chloride and nitrate, with other impurities.—J. O. B.

### *Glycerophosphates, The Organic.* Adrian and Trillat. Bull. Soc. Chim. 1898, **19**, [15], 684–688. (See also this Journal, 1898, 485.)

Glycerophosphoric acid is readily decomposed on concentration, when the free acid reacts with organic bases (quinine, cocaine, &c.) the phosphate of the base is always formed. The following method was adopted. To a solution of neutral calcium glycerophosphate, dilute sulphuric acid is added until the solution is acid to Helianthin, the solution is then made exactly neutral by the addition of a little neutral glycerophosphate. The mixture is warmed, filtered from calcium sulphate, an excess of the base added, and then a large excess of alcohol. After heating, the calcium glycerophosphate is filtered off, the solution evaporated at a low temperature, the residue taken up in a little water, excess of base extracted by shaking with ether, the solution again evaporated, and the residue triturated with strong alcohol. This treatment is twice repeated in order to remove all the calcium glycerophosphate, and finally the residue soluble in alcohol is dried at 50°—60° C.—

$$\mathrm{Ca[O.PO.(OH).OC_3H_5(OH)_2]_2 + 2C_{17}H_{21}NO_4 = C_3H_5(OH)_2O.PO.(OH.C_{17}H_{21}NO_4)_2 + C_3H_5(OH)_2.O.PO.O_2Ca.}$$

Strong bases, as cocaine and quinine, alone appear to be capable of replacing all the calcium in the acid glycerophosphate. Aniline, phenylhydrazine, and pyridine precipitate a certain amount of neutral calcium glycerophosphate, but there remains in solution a double salt, *e.g.*, of aniline—

$$\mathrm{CaO.PO.(OH.C_6H_7N).O.C_3H_5(OH)_2.}$$

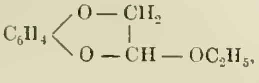
The neutral organic glycerophosphates and the double salts are all amorphous, in the dry state they are stable, but heated with water they rapidly decompose. Quinine glycerophosphate, for example, after a few minutes heating, gives on cooling a precipitate of white needles of quinine phosphate. Quinine and cocaine glycerophosphates are soluble in alcohol, slightly soluble in water, and insoluble in ether; the double salts are almost insoluble in water and alcohol.
—A. C. W.

### *Catechol (Pyrocatechin), Acetals of.* C. Moureu. Comptes Rend. **126**, [23], 1656—1658.

By heating the monosodium derivative of catechol with monochloracetal, the monoacetal derivative of catechol, $\mathrm{C_6H_4.OH.OCH_2.CH.(OC_2H_5)_2}$ is obtained, together with some of the diacetal, $\mathrm{C_6H_4(OCH_2.CH.(OC_2H_5)_2)_2}$. The monoacetal and any unchanged catechol are dissolved out by soda, and after acidulating again, the catechol is removed by repeated washings with water, when a heavy insoluble oil remains. Boiling dilute sulphuric acid hydrolyses this, giving the corresponding aldehyde, $\mathrm{C_6H_4.OH.OCH_2.CHO}$, a dense colourless oil, boiling at 139° C. under a pressure of 9 mm., soluble in warm water, and in alkalis, from which latter solutions, acids reprecipitate it unchanged. It does not give the phenol coloration with ferric chloride, but it reduces ammoniacal silver nitrate, gives Schiff's reaction, and forms a well-crystallised bisulphite compound. It is decomposed at 225° C., losing water. The acetyl-derivative,

$$\mathrm{C_6H_4.OCOCH_3.OCH_2.CHO,}$$

distils at 141° (pr. 8 mm.) and still exhibits aldehydic reactions. The monoacetal, when heated to 210°—215°, loses alcohol, and leaves a new substance, ethoxyl-ethane catechol—



$$\mathrm{C_6H_4}\left\langle\begin{matrix}\mathrm{O-CH_2}\\ \quad\ \ |\\ \mathrm{O-CH-OC_2H_5,}\end{matrix}\right.$$

a colourless oil, boiling at 247° C. and of sp. gr. 1·158 at 0° C.

Boiling weak sulphuric acid hydrolyses this body, eliminating alcohol, and forming the aldehyde described above. Ethoxylethane catechol, in fact, seems to be a kind of acetal, with one alcoholic and one phenolic group attached to the group CH.

The diacetal obtained from the soda-solution of the original product is an oily liquid, distilling, under 9 mm. of mercury, at 195°—197° C., and of density 1·061; it yields an aldehyde when hydrolysed by boiling weak sulphuric acid.—J. T. D.

### *Drugs, &c., Indian.* W. R. Dunstan. Imp. Inst. (Indian Sect.) Annual Report, 1897–98, 23—24.

*Podophyllum.*—As regards their chief constituents Indian and American podophyllum are identical, and a therapeutic test of the podophyllin from the former shows it to be of equal medicinal value with that from the latter variety, whilst the quantity yielded by the plant is apparently higher. The plant also contains quercetin and is found by Hummel to act satisfactorily as a yellow dyestuff.

As the extraction of both resin and dye is simple, there ought to be a large and remunerative business done in this article.

*Aconites.* — Further experiments, particularly with *Aconitum ferox*, are in progress. It has been established that the subsidiary alkaloids in the plant have an opposite physiological action to pure aconitine, and it is therefore considered that the preparation of the latter would be most advantageous for use in medicine.

A comparison of the pseudaconitine from the Indian plant with the true aconitine from *A. napellus* is being instituted.

*Opium.*—The Indian Government has declined the investigations suggested with a view to the improvement of the quality of this article so as to enable it to compete in Europe with the Turkish product, the reason given being that it might prove detrimental to the trade with China. For the same reason it is not desired to utilise any but waste opium for the extraction of morphine.

*Edible oils; Tanning materials; Hyoscyamus.*—Reports are in preparation.

*Burmese India-rubber.*—An encouraging report containing advice on improvements in the methods of collection was received from the expert referee and forwarded to India.

*Mica and Mica Preparations.*—Some special preparations of powdered mica and vaseline were reported on unfavourably.

*Calotropis Floss and Pine-apple Fibre.*—The report on Assam pine-apple fibre was satisfactory.—C. S.

### *Aconites, Indian; Report on the Constituents of Some.* W. R. Dunstan. Imp. Inst. (Indian Sect.) Annual Report, 1897–98, 33—34.

*Nepaul Aconite* (*A. ferox*). — A crystalline alkaloid corresponding with pseudaconitine was isolated from four samples of roots, the percentage ranging from 0·39 to 0·40 per cent. in the samples styled "*A. ferox*," and from 0·41 to 0·50 per cent. in those of "*A. ferox var. Crassi cauli.*" Another sample of *A. ferox* from Darjiling contained 0·35 per cent. of a somewhat different alkaloid, but the quantity was insufficient for a full examination of the product.

It thus appears that the roots in question are richer in pseudaconitine than those of *A. napellus* are in true aconitine, whilst it is probable that the first-named alkaloid will prove to be of equal medicinal value with the latter. Some doubt, however, still exists as to whether *A. ferox* is really the source of Nepaul aconite.

*Aconitum Napellus.*—A sample from Kaghan (Punjab) was found to contain aconitine but in smaller proportion than in roots grown in Europe; and the sample was inferior to the last named.—C. S.

### *Dill Fruits and their Essential Oils, Some Commercial Varieties of.* J. C. Umney. Pharm. J. 1898, [1468], 176. (See also this Journal, 1897, 556.)

Essential oil of dill, distilled in this country is chiefly obtained from English or Indian dill fruits, the latter being imported in considerable quantity for the purpose of distillation. The author has previously shown (Pharm. J., March 13, 1897) that the oil derived from the Indian fruit differs materially from that of English variety, the specific gravity being higher and the percentage of carvol less. Ciamician and Silber state that these differences are due to the presence of Indian dill oil, of a body having all the characters of apiol. (This Journal, 1897, 64.)

Recently a consignment of Japanese dill fruits have reached this country; oil from these resembles that of Indian dill. The following results were obtained with five samples of dill oil.

The oil distilled from German fruits has practically the same characters as that from those grown in England, unless, as is considered to be the case in the sample No. 3 in the above table, a portion of the carvol has been removed. Fractional distillation is stated to be the most satisfactory method for the approximate determination of this important constituent. In view of the presumed powerful therapeutic action of dill Apiol, it is desirable that the official characters of dill oil (sp. gr. 0·905 to 0·920; opt. rot. not less than + 70 in 100 mm.) should be adhered to in order to exclude the Apiol-containing oil derived from fruits of Asiatic origin.

| —— | Sp. Gr. | Opt. Rot. | Fractional Distillation. | | | | |
|---|---|---|---|---|---|---|---|
| | | | Below 200° C. | 200°—210° C. | 210°—220° C. | 220°—230° C. | Above 230° C. |
| | | | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| English, distilled from fruits grown at Market Deeping. | 0·9418 | +72°25' | 22 | 14 | 12 | 50 | 2 |
| English, distilled from fruits grown at Canterbury. | 0·9146 | +80°25' | 21 | 19 | 12 | 46 | 2 |
| German | 0·9002 | +70°25' | 53 | 13 | 12 | 17 | 5 |
| Indian | 0·9486 | +47°5 | 24 | 17·5 | 7 | 10·5 | 39 |
| Japanese | 0·8613 | +50°5' | 21 | 12 | 10 | 8 | 49 |

—J. O. B.

### *Gention Root, Action of Soluble Ferments on the Pectic Products of.* E. Bourquelot and H. Hérissey. J. Pharm. Chim. 1898, **8**, [4], 145—150.

Pectose appears to form an integral portion of the cell membrane, it cannot be extracted as pectose, and only its first hydrolysis product, pectin, is known. In order to study the action of ferments on pectose, powdered gentian itself must be employed. A solution of the soluble ferments of *aspergillus niger* was obtained by three days' contact of distilled water with a mature culture. Comparative experiments, with this solution boiled and unboiled, in the presence of thymol solution, show that the solution contains a ferment (destroyed by boiling) capable of transforming the pectose of gentian root into pectin. (See also under XVII., page 859.) —A. C. W.

### *The Aloïns.* E. Leger. Comptes Rend. 1898, **127**, [4], 234—236. (See also this Journal, 1897, 756.)

Barbaloin is soluble in concentrated hydrochloric and hydrobromic acids, from which solutions it is reprecipitated on dilution. The aqueous solution gives a yellow precipitate with excess of bromine water, readily soluble in alcohol, but non-crystalline.

*Trichlorobarbaloïn*, $C_{16}H_{13}Cl_3O_7 + H_2O$, is obtained by the addition of potassium chlorate to the hydrochloric acid solution of barbaloin. The crystalline mass formed on standing is washed and purified by recrystallisation from strong alcohol. The compound crystallises in lemon-yellow rhomboidal tables, readily soluble in sodium carbonate solution without displacement of carbonic acid. Triacetyl and tribenzoyl derivatives are obtained by the action of excess of the acid chlorides at 100° C. Barbaloin itself, under similar conditions, also yields a triacetyl compound; it contains three hydroxyl groups only, and these are of phenolic nature.

*Isobarbaloïn*, $C_{16}H_{16}O_7$, is found in the last fractions in the crystallisation of the aloins from the Barbadoes aloes. It is purified by recrystallisation from methyl alcohol, and is obtained as opaque tufts of microscopic leaflets, containing $3H_2O$. From water, isobarbaloïn crystallises in very pale prismatic needles containing $2H_2O$. Benzoyl chloride, in presence of pyridine, produces a dibenzoyl derivative exactly similar to dibenzoylbarbaloïn. Trichloroisobarbaloin, $C_{16}H_{13}Cl_3O_7 + 4H_2O$, obtained in the same manner as trichlorobarbaloin, crystallises in shining yellow prismatic needles, and forms a triacetyl compound. Tribromoisobarbaloin is the substance described under the name of tribromobarbaloin. The author considers that barbaloin and isobarbaloin are the only aloins contained in the various commercial aloes.—A. C. W.

### *Quinine, The Basicity of.* David Howard and D. Lloyd Howard. Pharm. J. 1898, [1468], 151. Read at the Brit. Pharm. Conf. at Belfast, August 1898.

The behaviour of quinin towards acids points to the fact that the alkaloid is a "diammonia;" that is, that each of the nitrogen atoms of the molecule represents a basic nucleus, one of which is much more powerful than the other. Thus, with sulphuric acid, quinine will form three definite crystalline salts; one molecule of acid combining with two molecules of quinin to form the ordinary sulphate of commerce; one molecule of acid with one molecule of quinin to produce the commercial "soluble sulphate," and two molecules of acid with one of quinia to form the little-known "tetra-sulphate." Mono-basic acids form salts with both one and two molecules of acid to one of alkaloid.

Confusion has arisen from the fact that French chemists consistently term the "soluble sulphate" as the "neutral sulphate," and the ordinary sulphate of commerce as the "disulphate;" similarly they speak of the soluble hydrochloride as the "neutral" salt, and the ordinary hydrochloride as "basic." This confusion arises from the failure over here to adhere to the old rule which gives a different significance to the Greek and Latin prefixes, and to erroneously regarding a bisulphate as synonymous with a disulphate.

In determining the basicity of quinine by direct titration with sulphuric acid, phenolphthalein is found to be without reaction. With litmus, the formation of the ordinary sulphate is definitely indicated, but no indicator seems to show the formation of the soluble salt with any degree of sharpness.

The existence of these three sulphates of quinine will explain the fact that the addition of sulphuric acid increases the specific rotation of quinine solutions. In the matter of nomenclature, adherence to that at present adopted in this country is advocated, rather than that followed by French chemists. To guard against the confusion which has arisen, the Italian Pharmacopœia gives the following remarkable trio of synonyms:—

"Bisulfate de chinino = sulfato acido de chinino = sulfato neutro de chinino."—J. O. B.

### *Morphine, The Bromine Derivatives of.* H. Causse. Bull. Soc. Chim. 1898, **19**, [15], 707—709.

Ordinary bromination processes give rise to amorphous yellow bromine addition-compounds of tetrabromomorphine, from which sodium thiosulphate (among other reagents) removes bromine, with production of the hydrobromide of β-tetrabromomorphine.

*α-Tetrabromomorphine hydrobromide*, $C_{17}H_{15}Br_4NO_3 . HBr$, is obtained by the action of bromine on morphine, both substances being dissolved in hydrobromic acid of density 1·5. The crystals deposited on cooling are dried over potash, and finally by a gentle heat, then recrystallised from boiling anhydrous methyl alcohol. The compound melts at 218° C., is insoluble in cold water, methyl alcohol, and chloroform, rather soluble in boiling water and ordinary alcohol. The solutions are levorotatory. Sodium alcoholate and silver oxide remove part of the bromine and leave a substance with quinone properties.

*β-Tetrabromomorphine hydrobromide* is obtained as stated, or by the cautious addition of hydrogen peroxide or potassium permanganate to the mother-liquors of the α-compound.

*Tetrabromomorphine*, $C_{17}H_{15}Br_4NO_3 . 2H_2O$, is formed by the action of sodium carbonate on the aqueous solution of the hydrobromide of the α-compound. It is generally blue or violet in colour, is infusible, and has both basic and acid properties. The sulphate and oxalate were prepared by the action of sulphuric acid and acid potassium oxalate on the α-hydrobromide; both salts are insoluble in water and alcohol.

*Tribromomorphine hydrobromide*, $C_{17}H_{16}Br_3NO_3 . HBr$, is obtained on evaporating the mother-liquor of the α-hydrobromide.—A. C. W.

### *Geraniol and Citronellol, New Derivatives of.* J. Flatau and H. Labbé. Bull. Soc. Chim. 1898, **19**, [14], 637—638.

The authors have prepared certain esters of these alcohols.

*Geranyl phthalate* is obtained by recrystallisation from petroleum spirit in shining rhombic tables melting at 47° C. The ester is readily soluble in all solvents in the cold; from petroleum spirit it commences to separate at 10°—12° and is almost insoluble at 5° C. If the solution in this solvent contain the citronellol ester, the quantitative separation requires a week in a freezing mixture. The silver salt, a stable white powder, melts at 138°·8 C.

*Tetrabromo-geranyl phthalic ester*, obtained by the action of 2 mols. of bromine on 1 mol. of the preceding ester in ethereal solution, melts at 114°—115°.

*Fatty acid esters* of geraniol and citronellol have been obtained by heating with an excess of acid and anhydrous sodium acetate in sealed tubes at 150°—160° for 15—20 hours, carefully washing with soda and distilling *in vacuo*.

| | | | | |
|---|---|---|---|---|
| Citronellol acetate | boils at | 172°—173° | under | 34 mm. |
| „ valerate | „ | 194°—196° | „ | 31 „ |
| „ (active) caproate | „ | 168°—170° | „ | 33 „ |
| „ crotonate | „ | 138°—140° | „ | 35 „ |
| Geraniol valerate | „ | 130°—132° | „ | 30 „ |

—A. C. W.

### *Citral* O. Doebner. Ber. 1898, **31**, 1888—1896.

By means of the reaction with pyrotartaric acid and β-naphthylamine (Ber. **27**, 352 and 2020; this Journal, 1898, 598), the author finds in the lemongrass oil of *Andropogon citratus* 80—82 per cent. of citral; in the essential oil of *Citrus limonum* 7—8 per cent.; and in the other oils containing citral, only inconsiderable amounts of the aldehyde. In applying the above test to the oils poor in citral, the principal product of the reaction is α-methyl-β-naphthocinchoninic acid, melting at 310° and soluble in alcohol with far more difficulty than the corresponding citral compound. Lemongrass oil contains, at the most, only traces of other aldehydes mixed with citral. Pseudoionone prepared from citral by the older process with barium hydroxide (this Journal, 1897, 1038) is identical in physical properties with the citral obtained from lemongrass oil by the chloride of lime process (this Journal, 1897, 1038); the latter product, however, is optically active. In the chloride of lime process this substance is at once converted into calcium chloride and hydroxide, and the condensation takes place during the long digestion under the influence of the hydroxide. The ionones prepared by the two processes are identical in boiling point and specific gravity; α- and β-ionone are present in both.—A. C. W.

### 1.4.2. *Dimethylnaphthol* [*from Santonin*]. E. Wedekind. Ber. **31**, [11], 1675—1680.

This substance, which is a decomposition product of santonin, is obtained in very good yield by allowing a solution of santonin in concentrated hydrochloric acid to stand for eight days. The desmotroposantonin which separates out crystallises from alcohol and melts at 260° C. The same result can be obtained, although the yield is not so good, by warming the hydrochloric acid solution to 60° C for about three hours. This substance is then dissolved in 4½ times its weight of 72 per cent. acetic acid, and heated on the water-bath with an excess of zinc dust. The purification is effected by treatment with soda and precipitation with an acid. The melting point of the acid crystallised from alcohol is 175° C. When this is fused in a porcelain dish in a lead-bath at 360° C., with twice its weight of potash, for about 45 minutes, a mixture of the potassium salts of propionic acid and of dimethyl-β-naphthol is produced, from which the latter can be obtained by precipitation with carbonic acid. The product crystallises from alcohol in needles melting at 136° C. Compared with β-naphthol the dimethyl-β-naphthol is characterised by its extreme inactivity. For example, it does not combine with diazo compounds, nor will it condense with nitrosodimethylaniline hydrochloride, and nitrous acid also has no action on it.—T. A. L.

### *Mydriatic Alkaloids, A Note on.* H. A. D. Jowett. Pharm. J. 1898, [1468], 195—196. Read at the Brit. Phar. Conf. at Belfast, August 1898.

Of the mydriatic alkaloids at present known, only atropine, hyoscyamine, and scopolamine (hyoscine) are definitely regarded as chemical entities. For the determination of the purity of commercial atropine and its salts, it is suggested that the behaviour of the solutions with polarised light should be observed; since solutions of pure atropine being optically inert, the presence of hyoscyamine or of scopolamine as impurities may be readily detected by their optical activity. The melting point of the sulphate prepared from pure atropine was found to be 190° C., which is 7° higher than the figure given in the recently-published British Pharmacopœia. The melting points of the aurichlorides of the alkaloids of this group afford a ready means of identifying them. These salts are best prepared by dissolving the bases in an excess of HCl, adding $AuCl_3$ and crystallising the double salts from the hot solution. Atropine aurichloride melts at 137° C, hyoscyamine aurichloride at 160° C., and scopolamine (hyoscine) aurichloride at 198° C. Pure hyoscyamine sulphate, prepared by the author, melted at 204° C., and not at 206° C., as stated in the Pharmacopœia. The melting point of the commercial salt is found to be about 200° C. The author has not met with any commercial scopolamine (hyoscine) hydrobromide melting at 193°—194° C., as stated in the official work. He confirms the statement of Hesse, that, as met with in commerce, it consists of a mixture of stereo-isomers melting at 181° C. The statement in the Pharmacopœia, that the hydrobromide forms with auric chloride a double crystalline gold salt, melting at 198°, is incorrect. The author has shown that under these conditions an additive compound having the formula $C_{17}H_{21}NO_4$.HBr.AuCl is formed, which melts at 215° C.—J. O. B.

### *Melissa, Essential Oil of.* J. Flatau and H. Labbé. Bull. Soc. Chim. 1898, **19**, [14], 636—637.

By shaking with sodium bisulphite solution and decomposition of the precipitate with caustic potash, the presence of 6 per cent. of citronellol in the oil is shown. The oil (600 grms.) was saponified with an equal volume of 5 per cent. alcoholic soda, washed, and distilled *in vacuo*. There were obtained 325 grms. boiling at 115°—160° under 40 mm. pressure, 120 grms. boiling at 160°—280° under the same pressure, 90 grms. boiling above 280°. From the first fraction 240 grms. of phthalic ester were obtained, from which 120 grms. of geraniol were isolated. The same fraction contains 72 grms. of linalol.—A. C. W.

### *Caparrapi, Essential Oil of.* F. J. Tapia. Bull. Soc. Chim. 1898, **19**, [14], 638—644.

This essential oil is obtained from deep incisions in the wood of *Nectandra caparrapi*, a tree belonging to the *Laurinaceæ* found in the Republic of Colombia. The oil varies in colour from a pale yellow to deep brownish red. In commerce the two varieties are distinguished as white and black. The odour is sweet, more agreeable in the white oil, and recalls that of chloral. The density varies from 0·9336 for white oil to 0·9163 for the black oil. The oil has a feeble action on polarised light, is very soluble in methyl and ethyl alcohols, glacial acetic acid, ether, chloroform, benzene, petroleum; and carbon bisulphide; less soluble in amyl alcohol, it readily dissolves fats, waxes, resins, caoutchouc, naphthalene and paraffins. All samples have an acid reaction towards litmus.

*Caparrapic Acid*, $C_{15}H_{26}O_3$.—By extraction of the white oil with soda solution of 2° B., and addition of hydrochloric acid, crystals are formed. These are boiled with milk of lime and the calcium salt recrystallised from alcohol. The pure acid is obtained from this salt; it forms white needles, melting at 84°·5 C. upon mercury and remains in a state of superfusion for several days; it shows a weak dextro-rotatory power, is little soluble in water, readily soluble in alcohol. If the alcoholic solution be heated with a mineral acid, the acid loses its property of crystallising. Heated to 120°—140°, the acid loses weight and is transformed into a thick reddish-yellow liquid, resembling the resinous acid which can be extracted from the black oil. Probably the presence of the resinous acid in these oils is a consequence of the heating of the original essence to drive off water.

Ferric chloride produces a voluminous light yellow precipitate in solutions of the acid or its salts. The calcium salt, $Ca(C_{15}H_{25}O_3)_2 + 5H_2O$, forms long silky needles, which melt at a high temperature with decomposition; it is very soluble in hot dilute alcohol. The silver salt forms brilliant rectangular leaflets melting at 170° upon mercury.

The black essential oil contains no aldehyde or ester. The first fractions of a distillation, boiling at 170°—185° under 40—45 mm. pressure, appear to consist almost entirely of a sesquiterpene alcohol, $C_{15}H_{26}O$, the acetic ester of which decomposes on distillation, forming a hydrocarbon which unites with hydrochloric acid to yield the hydrochloride $C_{15}H_{24}.2HCl$. This hydrocarbon, caparrapene, is also obtained by the action of phosphoric anhydride on the alcohol; its density at 16° C. is 0·9019; rotatory power, $a_D = -2°·21$; refractive index, 1·4953. The white essential oil from which the acid had been extracted was distilled with water. The distillate consists of the alcohol, $C_{15}H_{26}O$, caparrapiol, density, 0·9146; rotatory power, $a_D = -18°·58$; refractive index, 1·4843; boiling point, 260° C. under 757 mm. pressure.—A. C. W.

*Oil of Cade (Oleum Cadenum), Notes on.* Cathelineau and Hausser. Bull. Soc. Chim. 1898, **19**, [13], 577—580.

Oil of cade, an oil obtained amongst the distillation products from Juniper wood, is in general use as a therapeutic agent, but reliable information as to its composition and the principle to which it owes its healing virtues in certain diseases of the skin is still wanting. Although Wallach (Annalen, **238**, 82) isolated a certain substance which he termed *cadinene* by distilling the crude oil in a current of steam, the method cannot, in the author's opinion, be employed in a quantitative analysis. It is true that a small quantity of a substance, the bulk of which has a boiling point between 260° and 280° C., passes over with the steam, together with traces of acid, but the yield is exceedingly small, and the distillation difficult to carry out on account of bumping, whilst, on the other hand, the tempetature (100° C.) causes alterations in the oil and produces new compounds differing from those in the original product.

To avoid these drawbacks, the authors recommend the treatment of the oil with 5 per cent. sodium carbonate solution, which effects a preliminary separation into two classes of products, which can be further subdivided without undergoing material alteration. 400 c.c. of the oil are shaken in a separating funnel with 500 c.c. of 5 per cent. sodium carbonate solution, and after standing for two hours, the alkaline liquid is withdrawn. The residue is shaken with 100 c.c. of water and 500 c.c. of petroleum spirit (b.p. 40° to 60° C.), and the aqueous portion separating on standing added to the first alkaline liquid. During the treatment with petroleum spirit, a small quantity of resin separates, and is collected on filter.

The alkaline solution is extracted with two successive portions (250 and 125 c.c.) of petroleum spirit, and the extract on evaporation of the solvent leaves a liquid (9 c.c.), of which, on fractionation, 4 c.c. distil between 245° and 265°; 3 c.c between 265° and 285°; leaving a residue of 2 c.c. These different fractions are added to similar fractions subsequently obtained.

The alkaline liquid, after extraction with petroleum spirit, is extracted twice with similar quantities of amylic alcohol. This extract on evaporation leaves a residue which, when dried at 125°, and subsequently at 145°, consists of a resinous substance which becomes hard on cooling. In the author's experiments it amounted to about 36 grms.

After the double extraction, the alkaline liquid is neutralised with sulphuric acid and distilled in a current of steam until about a litre of water has been collected. The portion of oil (about 6 c.c.) carried over by the steam distils between 205° and 225° C. The aqueous distillate also contains some organic acid (about 3 c.c.). The neutralised alkaline solution deposits, on cooling, a resinous substance, of which about 19 grms. can be collected. After separation of the resin, the solution is evaporated on the water-bath. The sodium sulphate crystallises, and an oily fraction which rises to the surface can be extracted with cold alcohol. It amounts to about 4 c.c.

The second main fraction of the original oil, *viz.*, the substances insoluble in sodium carbonate solution, are fractionated by distillation on a sand-bath. The bulk distils between 241° and 285° C., leaving a residue of about 50 c.c. The results obtained by this process from 400 c.c. of oil of cade are summarised in the following table:—

I. PRODUCTS SOLUBLE IN SODIUM CARBONATE SOLUTION.

| | | |
|---|---|---|
| Extraction with petroleum spirit..... | 9 | 77 c.c. |
| " " amylic alcohol ...... | 36 | |
| Oil volatile with steam............... | 6 | |
| Resin.............................. | 19 | |
| Soluble oil not volatile with steam.... | 4 | |
| Soluble acids volatile with steam..... | 3 | |

II. PRODUCTS INSOLUBLE IN SODIUM CARBONATE SOLUTION.

| | | |
|---|---|---|
| Distilling between 100 and 245°...... | 3 | 313 c.c. |
| " " 245 " 260°...... | 120 | |
| " " 260° " 285°...... | 140 | |
| Residue.............................. | 60 | |
| Total................................ | | 390 c.c. |

The 9 c.c. of I. (extraction with petroleum spirit) are identical with products contained in II. The authors promise a communication on the nature of the fractions thus obtained, and a comparison of them with the products yielded by the plant on extraction with various solvents.—C. A. M.

*Oil of Cade.* P. Adam. Bull. Soc. Chim. 1898, **19**, [13], 580—583.

The author's object was to determine whether it was possible for a pharmacist to obtain a product answering to the requirements of the French Codex. The different specimens examined were obtained from reliable French firms and tested in accordance with de Schulz's directions (Arch. des Sci. Biol. St. Petersburg, 1897, 345—374). On treatment with caustic potash, all the samples absorbed oxygen very rapidly from the air. They were fractionated by distilling 300 grms. under ordinary pressure, and each fraction was dissolved in ether and shaken with an alkaline carbonate solution to remove the free acids. The ethereal layer was shaken with a solution of caustic potash, which dissolved the phenols and polyphenols, leaving the phenol esters, ketones, and other neutral bodies. In each case different results were obtained. All the samples were completely soluble in amylic alcohol, ether, benzene, chloroform, carbon bisulphide, and aniline, but gave a more or less turbid solution in olive oil, in which de Schulz found the pure oil to be completely soluble. Turpentine should dissolve the oil completely (de Schulz), but in none of the specimens examined by the author was there complete solution. The results of other tests are given in a table. The fact that in every instance the total amount obtained on distillation was greater than the quantity of oil used is attributed to rapid oxidation. The author considers that these results show that it is practically impossible to obtain a pure oil of cade, and that therefore that oil ought to be removed from the Codex.—C. A. M.

*Eucalyptus Punctata (Grey Gum) and its Essential Oil.* R. T. Baker and H. G. Smith. J. and Proc. Royal Soc. N. S. Wales, **31**, 259—280.

Recourse to *Eucalyptus punctata* as a commercial source of eucalyptus oil is advocated. This "grey gum" tree is widely distributed, and although not hitherto used for distilling eucalyptus oil, yields a product which compares favourably with that of *E. globulus*. Specimens of oil distilled by the authors gave from 46·4 to 64·5 per cent. of eucalyptol, as compared with 49·8 per cent. found in the oil of *E. globulus*. The specific gravity and optical rotation of the different specimens, even when obtained from trees growing in close proximity, were found to vary slightly; as a rule the lightest coloured oils are richest in eucalyptol. The yield of oil from leaves and terminal shoots was good. —J. O. B.

### *Lemon Oil, A New Constituent of.* J. C. Umney and R. S. Swinton. Pharm. J. 1898, [1468], 196. Read at the Brit. Pharm. Conf. at Belfast, August 1898.

By first removing the limonene from lemon oil by distillation *in vacuo*, and then eliminating the aldehydes from the residue by means of sodium bisulphite, or by repeated saponification with an alkali, a product was obtained which had the odour and general characters of geraniol. This is stated to exist in the original oil in the form of the acetic ester to the extent of 1·2 to 1·4 per cent. of geranyl acetate, and is found to the same extent in both the "Palermo" and "Messina" varieties of lemon oil. —J. O. B.

### *Lemon Oil, Concentrated.* H. T. W. Idris. Pharm. J. 1898, [1468], 161—162. Read at the Brit. Pharm. Conf. at Belfast, August 1898.

Oil of lemon free from terpenes is best prepared by distilling off 90 per cent. of the original oil under reduced pressure. The residual 10 per cent. is then distilled with steam, when the yellow, highly fragrant, "concentrated" lemon oil comes over. The yield amounts to between 6 and 7 per cent. of the crude lemon oil used.—J. O. B.

### *Geranium, Volatile Oil of; The Acids in.* Flatau and Labbé. Comptes Rend. 1898, **126**, [26], 1878—1879.

From *East Indian geranium oil* the authors have isolated, by the usual method of saponification, and subsequent fractional distillation of the liberated acids *in vacuo*, a solid acid having the melting point 28°·2 C. The silver salt gave figures corresponding to the formula $AgC_{14}H_{27}O_2$, and similar results were obtained with the barium, calcium, and copper salts, the latter containing one molecule of water of crystallisation. The formula of the acid is doubtless $C_{14}H_{28}O_2$; it is therefore an isomeric form of myristic acid. It was noted that geraniol is extremely soluble in this acid, and even in solutions of its salts, so that several fractional distillations were necessary to purify the acid, after liberation, from adhering traces of that alcohol. The authors consider that the oily acid isolated by Messrs. Schimmel in 1894, and regarded by them as a mixture of caproic and acetic acids, was nothing but an oily solution of geraniol in the higher acid now discovered. In addition to this acid, acetic acid and traces of butyric acid were found. *Bourbon geranium oil* was found to contain an acid $C_{10}H_{18}O_2$, to the extent of 0·5 per cent.; half of this was present in the free state, and half in the form of an ester. In addition to this, acetic acid and a trace of valerianic acid were present.—J. O. B.

### *Jalapinolic Acid.* N. Kromer. J. Prakt. Chem. 1898, **57**, [10 and 11], 448—466.

Jalapinolic acid is formed, as previously shown, by the hydrolysis of the resin of false jalap. The acid is most readily isolated in a pure state by liberating it from its methyl and ethyl esters, since these bodies are readily obtained in a crystalline state. These esters were prepared by passing dry HCl gas into a solution of the acid in the respective alcohols, and subsequently purifying the esters by recrystallisation from petroleum ether and ether. The methyl ester occurs in shining colourless scales, the ethyl ester in needles. The former melts at 50°– 51° C., and congeals at 43°—42° C.; the latter melts at 47°—48° C. and congeals at 41°—40° C.

From these esters the pure acid is obtained, in the usual way, by saponification. It is concluded that jalapinolic acid is an oxyhexadecylic acid of the composition $C_{16}H_{32}O_3$, and is an isomer of normal hexadecemolic acid. The hydrogen of the hydroxyl group is replaceable by acid radicles, by the action of anhydrides on the ester of the oxy-acid. The ester obtained, having the composition $C_{15}H_{30}.OCOCH_3.CO_2C_2H_5$, was a liquid, which distilled under reduced pressure without decomposition. The oxy acid does not form a bromo-additive compound, but gives with bromine a substitution product. When reduced with hydriodic acid, the oxy acid is transformed into a hexadecylic acid, $C_{16}H_{32}O_2$, which is an isomeride of the acids of this formula already known. When oxidised with permanganate in alkaline solution, jalapinolic acid is split up into methylethylacetic acid, sebacic acid, and an isomeric form of the latter melting between 89° and 91° C.—J. O. B.

### *Albumoses, Contributions to the Knowledge of.* (*Fourth Communication.*) H. Schrötter. Monatsh. f. Chem. 1898, **19**, 211—222.

The author has previously shown (this Journal, 1894, 273) that the manufactured article known as "*Peptone Witte*" consists principally of two albumoses—one rich, the other poor in sulphur, and whose hydrochlorides can be easily isolated.

By dissolving "*Peptone Witte*" in water, and adding sodium nitrite and sulphuric acid, a substance having acidic properties separated, and this when purified was insoluble in water, partly soluble in strong alcohol, but soluble in weak alcohol, separating from the latter as a pale yellow crystalline powder. It had the elementary composition C, 51; H, 5·7; N, 15·7; S, 12·1 per cent. respectively, contained 0·28 per cent. of ash, and gave no biuret or nitroso reaction. A barium and calcium salt are described. Attention is drawn to the close similarity of this product with Maly's oxyprotosulpho acid, obtained by the oxidation of unpeptonised albumin with potassium permanganate (Monatsh. f. Chem. **6**, 107; **9**, 255; **10**, 26); the author does not, however, consider that these acids are identical.

By acting on the two albumoses (*loc. cit.*) with nitrous acid under the before-mentioned conditions, it is shown that the albumose containing the more sulphur is the one principally concerned in the reaction.—J. L. B.

### *Emetine and Cephaeline.* B. H. Paul, A. J. Cownley, and O. Hesse. Pharm. J. 1898, [1466], 98—100.

When they first isolated the two alkaloids of ipecacuanha, Paul and Cownley provisionally adopted the empirical formula $C_{15}H_{22}NO_2$ for emetine and $C_{14}H_{20}NO_2$ for cephaeline. Hesse has repeated the analysis of these alkaloids, and has obtained figures agreeing with those of the first-named investigators. From the determination of the methoxyl by Zeisel's method, and the molecular weight by Beckmann's boiling-point method, Hesse attributes to emetine the formula $C_{26}H_{30}N_2(OCH_3)_4$, and to cephaeline, $C_{28}H_{32}N_2O_2(OCH_3)_2$.

As the result of the methoxyl determination, Hesse infers that, in addition to cephaeline, there may be present in ipecacuanha a base richer than it is in methoxyl, having the formula $C_{26}H_{31}N_2O(OCH_3)_3$. The behaviour of cephaeline shows that it contains a free hydroxyl molecule. It is suggested that the alkaloids may all be derived from "nor-emetine" by a complex formula which is given.

With reference to these deductions, Paul and Cownley point out that, although the formulæ suggested by Hesse differ from those obtained by them to the extent of two atoms of hydrogen, the latter's analytical figures are more closely in accordance with their empirical formulæ than with those which he has adopted. In applying Beckmann's boiling-point method for the determination of the molecular weight, Paul and Cownley found difficulty in obtaining a solvent which had no action on the alkaloids; both chloroform and absolute alcohol give rise to apparent alteration. The dry alkaloids are not readily soluble in pure ether, although the nascent alkaloids, in the presence of water, are dissolved by it. From the fact that Hesse employed ethylic alcohol and chloroform in his molecular weight determinations, these results alone are not regarded as conclusive. It is mentioned incidentally that for medicinal purposes the crystalline hydrobromide of emetine has been found to be the most convenient salt for use. As produced on the commercial scale the salt contains two molecules of water—

$$C_{15}H_{22}NO_2HBr.2H_2O.$$

When crystallised from water, it forms beautiful silky needles containing nine molecules $C_{15}H_{22}NO_2HBr.9H_2O$. On exposure to air, these gradually effloresce until seven molecules of water have been lost, when the composition of the salt remains constant; in this form it is quite stable, and is readily soluble in water.—J. O. B.

*Spt. Æther Nit., Assay of.* C. E. Smith. Chem. and Druggist, 1898, **53**, [951], 59; from Amer. J. Pharm.

*See under* XXIII., *page* 880.

## PATENTS.

*Perfumes* [*Violet*], *Impts. in the Manufacture and Production of.* A. Verley, Paris, France. Eng. Pat. 11,613, June 16, 1897.

The manufacture and application to perfumery of alcohols of the general formula—

$$(C_3H_7)(CH_3).C_6H_3.CO.CRR'.OH$$

and their esters are claimed. The processes claimed for the production of these esters consist in acting on cymene with acid chlorides in the presence of aluminium chloride, brominating the ketone produced in the α-position by the action of bromine in the cold, and acting on the bromo-derivative with sodium acetate, propionate, &c., or in producing the substituted ketone directly from cymene by the action of chloracetyl chloride in the presence of aluminium chloride and converting into the ester as before. The substances obtained by the foregoing reaction are claimed as new products.—A. C. W.

*Antiseptic Substances, Impts. in the Manufacture of.* W. L. Wise, London. From Knoll and Co., Ludwigshafen-on-Rhine, Germany. Eng. Pat. 18,178, Aug. 4, 1897.

The manufacture of an almost odourless compound of iodoform with an albuminous substance (egg albumin, peptone, casein) by bringing solutions of the two substances into contact, and heating the precipitate for a few hours at about 120° C., subsequently washing with an iodoform solvent if required, is claimed. The heating at the comparatively high temperature brings about combination between the iodoform and albumin.—A. C. W.

*Pharmaceutical Compounds* [*Protein Silver Compounds*], *The Manufacture or Production of.* H. E. Newton, London. From Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 18,478, Aug. 9, 1897.

The process is claimed for the production of soluble silver protein compounds, consisting in dissolving the insoluble compounds obtained by the action of proteins on silver salts or silver oxide, in solutions of albumoses or the soluble compounds obtained by the action of formaldehyde on the solutions of natural proteins. The products are claimed as new articles of manufacture, and their use, for therapeutic purposes.—A. C. W.

*Iodised Albumin and Iodised Peptones, An Improved Process for the Production of.* S. S. Bromhead, London. From The Chemische Fabrik in Helfenburg, Dresden, Germany. Eng. Pat. 7740, March 31, 1898.

The process for the production of iodine compounds of albumin and peptone is claimed, which consists in mixing the powdered substance with iodine and 90 per cent. alcohol, drying at 35° C., washing the dry powder with absolute alcohol, dilute sodium carbonate and thiosulphate solutions, water, alcohol, and ether, and again drying at 35° C.
—A. C. W.

# XXI.—PHOTOGRAPHY.

*Ammonium Persulphate as a Reducer for Negatives.* Lumière Bros. and Seyewetz. Bull. Photo. Club, Paris, 1898, **8**, 232.

Most reducers slowly dissolve the deposited silver, working from the top of the film inwards. Thus in any short time the effect on the weakest parts of the negative is greater than on the stronger parts. A hard or too dense negative, the result of under-exposure and over-development, cannot be successfully treated by such a reducer. It can, however, by an aqueous solution of ammonium persulphate not stronger than 5 per cent., in a bath of which the fixed and well-washed negative is placed, and gently rocked till the desired weakening is nearly reached, when it is taken out, kept for a few minutes in a 10 per cent. solution of sodium sulphite, and then well washed. The authors consider that the persulphate penetrates to the lower layers of the film, and there dissolves silver according to the equation—

$$SO_4(NH_4) + Ag = AgNH_4SO_4,$$

and that then the reaction—

$$2(SO_4NH_4) + 2H_2O + 2AgNH_4SO_4 =$$
$$2(NH_4)_2SO_4 + 2H_2SO_4 + Ag_2 + O_2$$

occurs; this can, however, only take place in the upper layers or at the surface of the film, where the ammonium persulphate is in excess. Thus the solution of silver is greater, and its reprecipitation relatively less, in the stronger than in the weaker parts of the film.—J. T. D.

*Substrata for Negatives.* E. Vogel. Phot. Mittheil. 1898, **35**, 73.

Substrata are used to cause the sensitive film to adhere more firmly to the glass. For wet collodion plates, dilute albumin made slightly alkaline with ammonia is very suitable, for the silver salt coagulates the albumin, and gives a very adherent film. It is not so good for collodion emulsion, as here the silver salt does not come into contact with the albumin, which remains soluble in water. In this case a solution of 1 grm. of india-rubber in 1,000 grms. of benzene answers well, but if many prints have to be taken from the negative, the film is apt to peel off or to crinkle. For wet plates, collodion emulsion, or collodion or gelatino-bromide dry plates, an excellent substratum consists of 1 grm. of emulsion gelatin, 300 c.c. of water, and 6 c.c. of a 2 per cent. solution of chrome alum, poured two or three times over the plate. Almost equally good is 3 grms. of gelatin in 1,000 c.c. of water, to which 10 c.c. of formalin have been added; the formaldehyde hardens the gelatin, and acts as an antiseptic.—J. T. D.

*Colour Sensitisers for Gelatino-bromide Plates.* Paul Ruh. Phot. Corr. 1898, **35**, 243.

Various colouring matters were compared as to their effect in increasing the sensitiveness of plates in the less refrangible part of the spectrum. Erythrosin (the sodium

| Colouring Substance. | Extent of the Sensitive Band. | | Time to reach Medium Intensity. | Relative Delicacy. Erythrosin = 1. |
|---|---|---|---|---|
| | From | To | | |
| | | | Seconds. | |
| Erythrosin | D | D ½ E | 1 | 1 |
| Eosin | D ½ E | E | 4 | ½ |
| Rose Bengal | C ¾ D | D ½ E | 2 | ¼ |
| Octoiodofluorescein | D | D ½ E | 4 | ¼ |
| Phloxin | D ½ E | D ½ E | 4 | ¼ |
| Bromonaphthofluorescein. | D | D ½ E | 6—7 | ⅙—⅕ |
| Napthofluorescein | B<br>C ½ D<br>D ½ E | D<br>D ½ E | 2 | ⅓ |
| Dibromonitrofluorescein. | D ½ E | D ½ E | 7 | ⅓ |
| Cyanosin | D | D ½ E | 4 | ½ |
| Methyleosin | D ½ E | D ½ E | 2 | ¼ |
| Ethyleosin | D ½ E | D ½ E | 2 | ⅓ |
| Monochlorofluorescein | From blue<br>D ½ E | D ½ E<br>D ½ E | 15 | 1/7 |
| Acridine Yellow | *b* | G | 7 | ⅓ |
| Chrysaniline | From blue | F | 7—8 | ½—¼ |
| Quinoline Red | C ½ D | D ½ E | 4 | ½ |
| Nigrosin B | *a*<br>C ½ D<br>D | B<br>D<br>D ½ E | 60 | 1/20 |
| Benzylfluorescein | D ½ E | E ½ F | over 15 | under 1/16 |
| Fluorescein | D ½ E | D ½ E | „ 15 | „ 1/16 |
| Tetranitrofluorescein | D | D ½ E | „ 15 | „ 1/16 |

derivative of tetraiodofluorescein) was taken as a standard. The plates were dipped for two minutes in a bath of 2 per cent. ammonia, then for two minutes in the colour bath (100 c.c. of water, 2 c.c. of ammonia, 6 c.c. of a solution of erythrosin of 1 to 500), and finally dried. These plates were very delicate, and showed a sensitive band from D, three-fourths of the distance to E. The table gives a summary of the author's results.—J. T. D.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Explosives, The Annual Report (Twenty-second) of Her Majesty's Inspectors of, for* 1897.

In the above report it is pointed out that no modification of the law took place during the year, but an Order in Council has been made, declaring that acetylene, when in a liquid state, "or, when subject to a pressure above that of the atmosphere, capable of supporting a column of water exceeding 100 inches in height," shall be deemed to be an explosive within the meaning of the Act, and when it is considered an explosive, its use is prohibited.

The growth of the trade in explosives has not diminished: three new factories have been added since 1896; 10 applications for new licenses are still under consideration. The following have been added to the list of authorised explosives:—Amvis, Carbo-gelatin, Coalite or Pembrite, Cannonite No. 3, Nobel Ardeer Powder, Nobel Carbonite, Nobel Gelignite, Norres patent safety fuze lighters.

The amount of foreign nitroglycerin compounds imported is still large, being 1,153,550 lb. in 1897, against 1,259,200 lb. in 1896. Carbonite and gelatin dynamite account for nearly 100,000 lb. of this, the amount of gelatin dynamite being rather in excess of the carbonite.

Dr. Dupré reports that the quality of the various explosives examined shows a distinct improvement. A revival of activity among inventors took place, due, no doubt, to the new Act regulating the use of explosives in mines, which came into force at the beginning of this year. Eleven new explosives were brought to the notice of the Department. A considerable amount of chemical examination has been involved in connection with explosives proposed for use in coal mines, and it has been found necessary to fix the proportions of certain of the constituents within somewhat narrow limits, as a slight variation might very seriously affect the character of the explosive so far as liability to set fire to explosive mixtures of gas or dust and air is concerned.

Dr. Dupré describes an interesting experiment in which practically pure acetylene under different degrees of compression was exposed to the action of a strongly heated platinum wire. It was found that at ordinary pressure the gas undergoes slight local decomposition, which does not, however, extend to the mass of the gas. Under a pressure of 15 ins. of mercury above the atmospheric a similar result was obtained, but when the pressure was increased to 29 ins., the gas exploded the instant the wire became red hot, and when subsequently the carbon produced was collected, it was found that the whole of the acetylene had been decomposed.

During the use of explosives of the first division of the nitro-compound class, a number of accidents have taken place through a pick or tool striking into unexploded portions of the charge. As these imperfect explosions could be largely traced to the use of detonators of insufficient strength, a circular was issued to manufacturers and importers of detonators calling their attention to the necessity of observing a correspondence between the charges in their detonators and standard charges as laid down in the official dictionary of explosives.

An account is given of the principal outrages both at home and abroad, the most serious one in this country being the explosion on the Underground Railway at Aldersgate Street Station.

From the circumstances of a great petroleum fire which occurred at Purfleet, the advantage is very apparent of having large quantities of petroleum surrounded by a ditch to prevent outflow of the oil to adjacent premises.

A testing station has been established at the Royal Arsenal, Woolwich, in which explosives can be tested in connection with the Coal Mines Regulation Act, 1896. This station is under the charge of H.M. Inspectors of Explosives. Major A. Cooper-Key, R.A., who has been appointed an additional Inspector of Explosives since the beginning of 1898, and has had the immediate control of this testing station since its establishment, furnishes a special report.

Explosives for use in coal mines must be on the "permitted list," and are only admitted to this list after having been admitted to the Home Office list of authorised explosives, and then passed the special tests at Woolwich. These tests consist in firing shots into a highly explosive mixture of coal-gas and air. The explosive passes the test if it fails to ignite the mixture in the first 20 shots, or if there is only one failure in the first 30, or two in 40 shots.

The gaseous mixture into which the shots are fired contains 9 per cent. of coal-gas and 91 per cent. of air.

The charge of explosive used, when of the class intended to be fired by a detonator, is taken as the equivalent of 2 oz. of No. 1 dynamite, and when not fired by a detonator, as the equivalent of 6 oz. of R.F.G. gunpowder.

It is pointed out that although an explosive may be on the permitted list, no relaxation in the way of precaution should take place, as no test can ensure absolute freedom from risk when the explosive is used in fiery mines.—W. M.

*Liquid Air as a Constituent of Explosives.* W. Schulz. Glückauf, 1898, **34**, 341.

Experiments have been made with coal-dust soaked with liquid air in the same manner as kieselguhr is soaked with nitroglycerin. The mixture can be detonated by fulminate. If the liquid air contains the normal (23·17 per cent. by weight) proportion of oxygen, the theoretical figures for the work and the temperature of explosion are lower than the corresponding figures for dynamite; but if the liquid air contains 50 or 60 per cent. of oxygen the figures are higher than those for dynamite. No definite experimental results as to the explosive efficiency seem to have been obtained, and the cost is high.—J. T. D.

### PATENTS.

*New Explosive Compound.* C. D. Abel, London. From Gesellschaft für Linde's Eismaschinen, Wiesbaden, Germany. Eng. Pat. 18,929, Aug. 16, 1897.

This invention relates to the use of mixtures of liquid oxygen and oxidisable substances as explosives. Such mixtures burn away quietly when ignited in the ordinary manner, but when fired by a detonator explode violently.

The proposal is to produce the explosive at the place where it is to be used, by there employing a machine for liquefying air, from which, by evaporation, the larger portion of the nitrogen is eliminated; the liquefied gas is then mixed with an oxidisable body such as wood-charcoal powder, cotton wool being added to prevent the scattering of the charcoal powder when it is introduced into the boiling oxygen. Sulphur may also be used when a more disruptive explosive is desired.

The mixture must be placed in a case which is a bad conductor of heat, before being placed in the bore hole. If the oxidisable body be liquid—petroleum, for instance—it must first be absorbed in cotton wool, so as to offer a large surface to the liquid oxygen.—W. M.

*Matches adapted to prepared Striking Surfaces, Impts. in the Manufacture of.* S. Neuburg, Prague, and I. Neuburg, Vienna, Austria. Eng. Pat. 19,418, Aug. 23, 1897.

"This invention relates to improvements in headless matches free from phosphorus, and which are intended to ignite by striking against a suitably prepared surface."

The chief point is the substitution of a metallic sulphate for ammonium sulphate in compositions containing sodium chlorate for coating the matches.

The splints in bundles are first dipped in a solution of sodium chlorate, 87·5 parts; gum arabic, 12·5 parts; sulphate of iron, 7·5 parts; and water, 125 parts. After drying they are then dipped in a waterproofing solution composed of resin, 25 parts; turpentine, 67·5 parts; oleic acid, 125 parts; and linseed oil, 37·5 parts.

The friction composition for the striking surface on the box consists of amorphous phosphorus, 25 parts, 12·5 parts; glass powder, 3·75 parts; water, 25 parts; and gum arabic, 6·25 parts.—W. M.

*Safety Tamping for Explosives used for Blasting Operations in Coal and other Mines, and in Apparatus for Use in Connection therewith; Impts. in or relating to.* E. S. Clark, Wrexham. Eng. Pat. 20,223, Sept. 2, 1897, and Eng. Pat. 11,674, May 23, 1898.

These two patents have the same title and the same object in view. The invention consists in preparing solid cylinders, prisms, or other pieces of a dry hydrated salt, such as soda crystals, with central or other passages. Such prisms are placed at the bottom of the bore hole and above the explosive; the products of explosion permeate this stemming rapidly, and give up heat in liberating gaseous products from the stemming.—W. M.

*Explosives, Impts. in the Manufacture of.* U. Alvisi and E. Pulfici, Rome. Eng. Pat. Sept. 21, 1897.

The inventors prepare ammonium perchlorate by treating sodium perchlorate prepared from the chlorate, with ammonium nitrate, when ammonium perchlorate separates in fine crystals. Mixtures of ammonium perchlorate with picric acid or with trinitrocresol, called "cremonite," are claimed; also a black gunpowder in which ammonium perchlorate replaces potassium nitrate, and to which powders the name of *manlianite* is given.—W. M.

*Phosphorus and other Materials, Impts. in the Process of Manufacturing.* C. S. Bradley, Avon, New York, and C. B. Jacobs, East Orange, New Jersey, U.S.A. Eng. Pat. 10,290, May 5, 1898.

Raw materials containing phosphorus, such as mineral phosphates, bones, or calcium phosphates in any form, are mixed with sufficient carbon to combine with the oxygen present, and to form a carbide with the calcium, and the mixture is subjected to an electric current of from 25 to 50 kilowatts, in a furnace having a dome, an inclined carbon hearth, a feed-pipe communicating with a valved hopper, and a discharge pipe for the phosphorus vapour liberated, leading into a vessel containing water. The reaction in the case of ordinary calcium phosphate is stated to be—

$$Ca_3P_2O_8 + 14C = 3CaC_2 + P_2 + 8CO.$$

The calcium carbide formed flows on to the hearth of the furnace, and may be removed from time to time through a tap hole.—E. S.

# XXIII.—ANALYTICAL CHEMISTRY.

## *APPARATUS, ETC.*

[*Lubricating Oils.*] *The Chercheffsky Viscosimeter.* F. Jean. Rev. Chim. Industr. 9, [104], 220—221.

The advantages claimed for this apparatus are that, owing to the adjustability of the working parts, it may be approximated to, and will give results comparable with, the other standard viscosimeters in use, according as the conditions of the experiment are modified in one way or the other. Thus, the diameter of the effluent jet, the length of the effluent tube, and the working temperature can be varied, and the oil may be either maintained under a constant pressure, as in the Barbey ixometer, or under variable pressure, like the Engler apparatus.

The instrument consists of a water bath surrounding the oil-cup—both being of copper—the former supporting the usual thermometers, and a tapped burette for maintaining a constant supply of oil when required. The outlet tube can be closed by a steel cone attached to a vertical spindle working in guides. For keeping the oil at any desired constant level, an overflow pipe is provided, which extends downwards through the bottom of both vessels and can be adjusted to any height, its exact position being recorded by a pointer and arm moving on a scale on one of the legs of the tripod stand.

A measuring glass, placed under the effluent jet, is graduated in one-sixths of a cubic centimetre, and thus records in ten minutes the rate of outflow in c.c. per hour. According to the inventor the standard degree of fluidity of an oil is expressed by the number of c.c. per hour discharged through a jet 5 c.c long and 1 mm. in diameter, the constant level of the oil being 5 c.c. above the outlet orifice, and the working temperature 35° C.

To approximate the apparatus to the Barbey ixometer, a jet 3 cm. long and 1 mm. diameter, with a working pressure 4, is required; and in the author's experiments in comparison with the Engler viscosimeter it was found that with a jet 3 mm. in diameter and 5 cm. long, the difference in the time required for the passage of 200 c.c. of oil in the two instruments did not exceed 0·02—0·05 of a minute.—C. S.

## *INORGANIC CHEMISTRY.—QUALITATIVE.*

*Formulæ for Test Papers.* Pharm. Post; through Pharm. J. 1898, 4, [1469], 226.

*Potassium Ferricyanide and Citric Acid.*—The papers have likewise the property of precipitating albumin from urine.

*Wurster's Dimethyl-paraphenyldiamine Paper* (Wurster's red ozone paper).—Filter paper impregnated with a solution of paraphenyldiamine. Indicator of ozone, sulphuretted hydrogen, &c.

*Wurster's Tetramethyl-paraphenyldiamine Paper.*—Filter paper impregnated with tetramethyl-paraphenyldiamine. Assumes an intense bluish-violet colour in the presence of traces of active oxygen in neutral or acetic acid solution. Reagent for ozone and peroxide of hydrogen.

*Reagent Paper (Geissler and Oliver) for Detecting Albumin.*—(*a*) *Picric and Citric Acids:* A precipitate results if a strip of the paper is dipped into urine containing albumin. (*b*) *Sodium Tungstate and Citric Acid:* The presence of albumin, mucin, uric acid, peptones, and creatinine is revealed by a precipitate resulting after strips of the above paper have been dipped into the urine.

*Detection of Sugar in Urine—Indigo, Carmine, and Carbonate of Sodium Papers.*—These papers furnish the Mulder reaction (a successive green, red, and yellow coloration) in the presence of sugar in urine.

*Griess Test Paper.*—Filter paper saturated with an alcoholic solution of metaphenylene-diamine. Gives a yellow to brown colour reaction in the presence of nitrous acid and nitrites.

*Griess Red Test Paper.*—Filter paper saturated with an alcoholic solution of sulphanilic acid and alpha-naphthylamino hydrochloride. The presence of nitrous acid and nitrites produce a dirty red coloration of the paper.

*Palladium Chloride Paper.*—Swedish filter paper saturated with a solution of palladium chloride. Reagent for coal-gas, carburetted hydrogen, carbonic acid, &c., shows liberation of metallic palladium.

*Phenolphthaleïn Paper.*—Filter paper impregnated with an alcoholic phenolphthaleïn solution employed as indicator in alkalimetry.

*Phloroglucin-Vanillin Papers.*—Filter paper saturated with alcoholic solution of phloroglucin-vanillin, employed as a reagent for detecting the free hydrochloric acid of gastric juice.—*Deutsch. Amerik. Apoth. Zeit.*, xviii., 100.

*Azolitmine Paper.*—A reddish-violet bibulous paper impregnated with azolitmine. It is coloured red by acids, blue by alkalis.

*Lead Acetate Paper.*—Filter paper saturated with a solution of neutral lead acetate, for detecting the presence of sulphuretted hydrogen.

*Lead Glazed Paper.*—Glazed paper impregnated with carbonate of lead, for detecting the presence of sulphuretted hydrogen.

*Brasilin Papers.*—Filter paper impregnated with a solution of brasiline; serves as an indicator for acids.

*Blue Carmine Paper.*—Filter paper impregnated with a solution of indigo carmine, for detecting oxygen.

*Red Carmine Paper.*—Paper saturated with a solution of ammonia-carmine, an indicator of acids, giving a yellowish-red colour reaction.

*Turmeric Paper.*—Filter paper saturated with an alcoholic tincture of turmeric. Indicator of caustic alkalis and alkaline earths, giving a reddish-brown colour reaction. It is coloured brown by boric acid, sulphur-yellow by acids. Sensibility for KHO about 1:180,000, for $NH_3$ 1:35,000.

*Fuchsin Paper.*—Paper saturated with an alcoholic fuchsine solution, employed for detecting sulphuric acid.

*Dahlia Paper.*—Filter paper impregnated with an alcoholic solution of the colouring matter of *Dahlia variabilis.* Indicator of acids and alkalis.

*Hæmatoxylin Paper.*—Filter paper saturated with aqueous solution of hæmatoxylin. Extremely sensitive indicator for ammonia, alkalis, alkaline earths, and certain metals.

*Bilberry Paper.*—Paper saturated with an alcoholic or aqueous extract of bilberries. Employed as an indicator of ammonia and fixed alkalis.

*Blue and Red Litmus Paper.*—Very sensitive indicators obtained by immersing filter paper with a neutral alcoholic litmus tincture. Sensibility of red paper for KOH 1:20,000; of blue paper, for HCl 1:50,000.

*Cobalt Paper.*—Filter paper impregnated with cobalt chloride and methyl blue solution. Indicator of zinc, copper, and nickel.

*Congo Paper.*—Prepared by impregnating filter paper with a solution of Congo red. Turned blue by acids Indicator of free acids.

*Albumin Paper.*—Paper impregnated with an aqueous solution of albumin. Principally employed in photography.

*Lacmoid Paper.*—Filter paper saturated with an aqueous solution of lacmoid. Obtainable in red and blue, and used in the same way as litmus paper. The red lacmoid paper is greatly superior to red litmus paper as an indicator for alkalis. It should be kept in a well-stoppered bottle, as it is very apt to spoil.

*Methyl Orange Paper.*—Filter paper saturated with an aqueous solution of methyl orange. Employed as an indicator of acids and alkalis. The methyl orange paper is coloured deep brownish-yellow by mineral acids.

*Ozone Paper.*—Potassium iodide starch paper. (Schonbein's ozone paper.) Filter paper saturated with starch paste which has previously been mixed with potassium iodide. Gives a blue reaction when ozone is present.

*Thallium Paper.*—Filter paper saturated with thallous hydrate. Gives a brown reaction when ozone is present.

*Boettger's Test Paper.*—Filter paper saturated with neutral gold chloride, coloured violet in the presence of ozone.

*Pole Test Paper.*—Unsized paper saturated with an alcoholic solution of phloroglucin-vanillin. The paper, when moistened, turns red when applied to the negative pole of an electric current.

*Silver Paper.*—Filter paper impregnated with an aqueous solution of silver nitrate. Indicator of chromic acid, arsenious acid, &c. Delicate reagent for arsenic, specially suited for toxicological examinations.

*Tropæolin Paper.*—Filter paper impregnated with a saturated alcoholic solution of tropæolin. Gives a lilac reaction in the presence of free hydrochloric acid in gastric juice.

*Potassium Mercuric Iodide and Citric Acid Paper.*—Albuminous urine shaken with this paper gives a voluminous precipitate.

### *The Alkaline Earths, Magnesia and Manganese Oxide; Detection and Separation of, in the presence of Acids forming Insoluble Salts with these Bases.* A. Villiers. Bull. Soc. Chim. 1898, **19**, [15], 710—711.

In the ordinary course of qualitative analysis, the precipitate obtained on the addition of ammonia, after boiling off sulphuretted hydrogen, oxidising with nitric acid, and addition of ammonium chloride, may contain the whole or a part of the calcium, barium, strontium, manganese, and magnesium, in addition to iron, aluminium, and chromium. Zinc, nickel, and cobalt do not form precipitates insoluble in a sufficient excess of ammonia and ammonium chloride even when acids are present with which these metals form insoluble salts. Of the acids which form insoluble salts with the above metals, silicic acid may be separated by evaporation of the acid solution; organic acids should be removed by ignition after the sulphuretted hydrogen treatment, for their presence may prevent the precipitation of the sesquioxides. It is useless to endeavour to separate boric acid: the borates are too soluble in the presence of ammonium salts to be found in the washed precipitate. Oxalic and phosphoric acids remain to be considered. Although the phosphates of the alkaline earth metals and of manganese are soluble in acetic acid, it is not sufficient to add the quantity of ferric salt theoretically sufficient to precipitate all the phosphoric acid; even if a considerable excess of iron be present, the precipitate on boiling may still contain a large proportion of the alkaline earth metals and manganese. The complete separation of phosphoric acid from these metals can only be effected by the use of a very large excess of iron (see following abstract).

—A. C. W.

### *Detection of Fe, Cr, Al, Ca, Ba, Sr, Mg, Mn, in the presence of Acids forming Insoluble Salts with these Metals.* A. Villiers. Bull. Soc. Chim. 1898, **19**, [15], 712—713.

Destroy organic matters, if present, by ignition. Remove silica by evaporation to dryness with hydrochloric acid. Test for phosphoric acid with ammonium molybdate. If phosphoric acid be present, oxalic acid is indicated by the formation of carbonic acid on boiling with nitric acid (a manganese salt to be added if not already present). In the absence of phosphoric acid, test for oxalic acid by calcium chloride and sodium acetate. If oxalic acid is found, destroy it by ignition or by boiling with nitric and hydrochloric acids, adding a manganese salt if necessary; then in the absence of phosphoric acid the sesquioxides are precipitated by ammonia, and the process follows the ordinary course. In the presence of phosphoric acid add ammonia until precipitation begins, dissolve the precipitate in the least possible quantity of hydrochloric acid, and add a small quantity of an alkaline acetate. If iron be in large excess, this occasions no precipitate in the cold. If a precipitate form, add a little ferric chloride and let stand a minute; if necessary, add more ferric chloride until a clear solution is obtained. If a drop of acetate solution produces a precipitate, add more acetate and ferric chloride. Dilute if the coloration is intense. Boil and add more acetate if necessary to produce a colourless solution. Filter boiling. Heat the solution with a slight excess of ammonia, collect the precipitate (if any) on a second filter and add to the first. Concentrate the solution, and test for the alkaline earth metals and manganese in the ordinary way.—A. C. W.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Formaldehyde, Use of Alkaline Solutions of, in Analysis.* L. Vanino. Ber. **31**, [11], 1763—1765.

Gold can be determined quantitatively by adding to its solution as chloride, ordinary commercial formalin, followed by a few drops of sodium hydroxide, and warming for a short while on the water bath. The precipitated gold is filtered (the filtrate being tested with additional formalin and alkali to see that precipitation is complete), well washed with water and alcohol, and either dried at 180° C. or carefully heated to redness in a crucible. The author's experiments (on quantities of chloride varying from 0·1203 to 0·5803 grm., and in fairly dilute solutions) gave results varying from 64·75 to 64·94 per cent., the calculated percentage being 64·92.

Silver, in solution as nitrate, can also be determined accurately by the same method, either in the cold or on the water bath; the only precaution necessary is to wash well with absolute alcohol to remove adhering water. Silver chloride is also completely and rapidly reduced by the same reagent, and the reaction can be used, not only in analysis, but as a means of preparing pure silver from silver residues.

The author promises a further communication on the use of alkaline formaldehyde in analysis, with especial reference to copper salts.—J. T. D.

### *Ferrous Salt in Alkaline Solution, Volumetric Analysis by.*
A. Job. Comptes Rend. **127**, [1], 59—60.

A FERROUS salt to which excess of sodium pyrophosphate has been added, to alkaline reaction, remains colourless, and can be titrated quite well with permanganate or iodine. The application to reductions in alkaline solutions is obvious.—J. T. D.

### *Combined Sulphuric Acid, The Volumetric Estimation of.*
F. Marboutin and M. Molinié. Bull. Soc. Chim. 1898, **19**, [15], 713—714, and 714—717.

IN the author's process for estimating combined sulphuric acid (this Journal, 1897, 1041), the results are obtained by a comparison of the process carried out with distilled water and the water to be examined. Telle (this Journal, 1898, 378) describes a similar method, in which solutions of a definite strength are employed. In such a method the solubility of barium chromate may give rise to a considerable error; when equal volumes of equivalent solutions (Ba = 137·2, Cr = 52·1) are mixed, the clear liquid decanted from the precipitate always contains barium and chromium. The amount of chromium remaining in solution after mixing 50 c.c. of $\frac{1}{10}$ N solutions, is equivalent to 1·5 mgrm. of $SO_3$; this quantity is 10 per cent. of the maximum amount contained in the waters for which the process is used. The method may be utilised for the examination of sewage, waste liquors, &c., if the organic matter be previously destroyed. This is accomplished by evaporating 200 c.c. with 20 drops of nitric acid on the sand bath, treating the residue with fuming nitric acid, and again evaporating, finally expelling excess of the acid by hydrochloric acid. The results agree with those of gravimetric determinations. The processes of Villiers (this Journal, 1897, 832) and Lindet (Comptes Rend. **125**, 246) for the destruction of organic matter by the use of nitric acid in the presence of manganese and vanadium salts, may be used preparatory to gravimetric estimations of sulphuric acid, but nitric acid alone must be used for the volumetric process. The authors find that it is not necessary to allow precipitated barium sulphate to stand in a warm place before filtering; the observed differences in the weights obtained were of the order of errors of experiment. They, however, allow the liquid to stand 12 hours at 10° C., to enable the precipitate to agglomerate before a gravimetric determination. In the volumetric process the barium chromate clears the liquid.

—A. C. W.

### *Boric Acid, Determination of.*
Vadam. J. Pharm. Chim. 1898, **8**, [3], 109—111.

IN estimating boric acid volumetrically, the author uses mannite in preference to glycerin (Barthe, J. Pharm. Chim. 1894), as the former gives a sharper final reaction. To determine the amount of boric acid contained in a butter, 50 grms. are extracted with 20 c.c. of warm water, and to the aqueous extract, which is usually acid, litmus is added, and then decinormal caustic soda solution until the colour changes to blue. The blue solution is divided into two portions, the one being retained for comparison of colour, whilst to the other 1—2 grms. of mannite are added, and the acid is then titrated with decinormal alkali. The number of c.c. of alkali multiplied by 0·0062 gives the weight of boric acid contained in the solution titrated.

According to the author, since it is immaterial as regards the calculation whether a triatomic or a hexatomic alcohol is used, it is probably not the boric acid compound of the alcohol which is neutralised, but the boric acid itself, which is liberated from this compound by the action of the alkali.

—E. W. W.

### *Nitric Acid, A New Method for the Estimation of.*
W. Ackermann. Chem. Zeit. **22**, [68], 690—691.

THIS method depends on the reduction of the nitric acid to ammonia in alkaline solution by means of ferrous hydrate. One grm. of potassium nitrate, 30 c.c. of caustic soda solution (sp. gr. 1.33), and 40 grms of ferrous sulphate, dissolved in water, are brought into a flask of not more than 500 c.c. capacity. The total volume of water used for washing in the nitrate and dissolving the ferrous sulphate should amount to about 160 c.c. A pinch of reduced iron is added to facilitate the boiling of the thick mixture, and the contents of the flask are distilled into standard acid in the usual manner for the estimation of ammonia. Half an hour is generally sufficient for the distillation; and just before the end of the operation, the point of the condenser is washed free from acid, and the last few drops of the distillate are tested with litmus paper. If the reduction has not proceeded satisfactorily, the distillate has an alkaline reaction to the very end, and the neutrality of the last few drops is a very good test of the success of the determination. The reagents themselves nearly always yield more or less ammonia, so that it is essential to correct the results obtained by a blank experiment. Good crude caustic soda is preferable to the purified article, and the ferrous sulphate should be prepared in the laboratory from iron wire and pure sulphuric acid. The above quantity of water may be doubled or even trebled, but at greater dilutions the action of the ferrous hydrate is diminished and reduction is incomplete; prolonged boiling also prevents the proper reduction of the nitric acid. A large excess of caustic soda especially must be avoided, and only slightly more than the quantity requisite for the decomposition of the ferrous sulphate must be used.

The presence of chlorides, sulphates, and acetates has no effect on the applicability of this method, but tartrates hinder the action; and if phosphates are present, as in the case of mixed fertilisers, a modification of the procedure must be adopted.

The solution of the fertiliser to be analysed is brought into the flask, and sufficient calcium chloride solution of known strength is added to convert all the soluble phosphate present into tricalcium phosphate; the contents of the flask are then shaken whilst the caustic soda solution is added drop by drop. An excess of soda is used, corresponding to the calcium chloride taken, and finally the ferrous sulphate and the iron are added and the distillation carried out as before; an increase of 10 c.c. of water is advisable to counteract the thickening of the mixture due to the calcium phosphate. Small quantities of iron in the fertiliser are without influence on the results.

The author demonstrates the accuracy of his method by the results of several determinations of nitric acid in known quantities of pure saltpetre.—J. F. B.

### *Phosphoric Acid, Determination of.*
H. Lasne. Comptes Rend. **127**, [1], 62—64.

VIGNON has stated that the amounts of phosphoric acid obtained by precipitating separately the water solution and the ammonium citrate solution of a superphosphate are together considerably less than that got by mixing the two solutions before precipitation. The author's experiments show that if the precipitates stand for a few hours before filtration, this is never the case. They also show that the precipitation is not influenced by any of the substances usually associated with phosphoric acid in superphosphates, except silica and fluorine, which do not go into the solutions; and that the ammonium-magnesium phosphate is absolutely insoluble in a liquor containing a third of its bulk of 22 per cent. ammonia solution—equal bulks of phosphate solutions of relative strengths 1, 10, 100, 1,000, giving exactly proportional amounts of precipitate.—J. T. D.

### *Phosphoric Acid, Determination of.*
L. Vignon. Comptes Rend. 1898, **127**, [2], 191.

IN the analysis of a superphosphate, the author (this Journal, 1898, 696) estimates the amount of citrate-soluble phosphoric acid by difference, from determinations of the quantity of acid soluble in water alone and the quantity soluble in water and citrate. In reply to Lasne (see preceding abstract) he states that this method is more rapid and more accurate than the direct determination.—E. W. W.

### *Phosphoric Acid dissolved by the Waters of the Soil, Estimation of the.* Th. Schloesing, jun. Comptes Rend. **128**, [6], 327—329.

When it is not convenient to determine the amount of phosphoric acid in solution in the waters of soils by the method of extraction by displacement (see under XV., page 857), the author recommends the following procedure :—

A quantity of soil, equal to 300 grms. in the dry state, is weighed out and gently agitated in a flask of 1·5 litres capacity with 1.300 c.c. of ordinary water, allowing for that already contained in the soil. The flask must be slowly rotated for 10 hours at the rate of two revolutions per minute, and at the end of that time the phosphoric acid is determined in 1 litre of the liquid after decanting through a filter.

In this manner an extract is obtained containing the same proportion of phosphoric acid as the water of the soil extracted by displacement. The flask must not be violently agitated, nor the treatment be prolonged for more than 10 hours, or the results will be too high.—J. F. B.

### *Phosphoric Acid in Superphosphates, A Volumetric "Citrate" Method for the Estimation of.* S. Littman. Chem. Zeit. **22**, [68], 691—693.

Orthophosphoric acid may be estimated in the presence of other acids by double titration with standard caustic soda. The solution is first titrated with methyl orange as an indicator, whereby the other acids are completely neutralised and the phosphoric acid converted into the monobasic sodium salt $NaH_2PO_4$. Further addition of alkali changes this into the dibasic salt $Na_2HPO_4$, which, though alkaline to methyl orange, is acid to phenolphthalein. After the point of neutrality to this latter indicator is passed, the tribasic normal salt is formed, which is alkaline to both. This estimation can be made with accuracy when only acids or alkaline bases are present, but in the case of superphosphates, the other bases present interfere with the titration.

The phosphates of the fertiliser are converted into sodium phosphate, and the precipitation of the bases is prevented by the use of a neutral solution of sodium citrate. This solution is prepared by dissolving 30 grms. of pure caustic soda in about 120 c.c. of water, and accurately neutralising it with a concentrated solution of citric acid in presence of phenolphthalein. After 24 hours it is filtered, again neutralised if necessary, and made up to 250 c.c.

For the estimation, 10 grms. of superphosphate are shaken with about 400 c.c. of water for half an hour in a 500 c.c. flask, and then made up to the mark and filtered. Fifty c.c. are measured out and titrated with pure decinormal caustic soda, free from carbonate, using methyl orange as indicator, to complete neutrality. By this quantity of soda the acids are neutralised and the phosphates are brought into the monobasic condition; 10 c.c. of the citrate solution are then added to convert the calcium and other phosphates into the monobasic sodium salt. Phenolphthalein is next added as indicator, and soda solution is run in to distinct redness, exact neutrality being obtained by titrating back with standard acid. The solution remains quite clear and after a little practice very sharp results can be obtained. The phosphoric acid is now all in the state of dibasic sodium salt, and the percentage of $P_2O_5$ in the sample can be calculated by multiplying the number of c.c. of decinormal soda required for the second titration by the factor 0·71.

Some superphosphates, containing much iron and aluminium, do not give a sharp end-reaction with methyl orange at the above concentration, but satisfactory results can be obtained by diluting the 50 c.c. of superphosphate extract to five times the volume and titrating; the titration in presence of phenolphthalein is then performed on another 50 c.c. of the extract after the addition of 10 c.c. of citrate solution, and the quantity of soda required by the first portion is deducted.

By this method the soluble phosphoric acid may be estimated in 45 minutes with very fair accuracy; a large number of determinations of $P_2O_5$, in superphosphates from various sources, quoted by the author, show a mean difference by this method of only 0·13 per cent. higher than by the gravimetric citrate method. This method of course is not applicable to the estimation of meta- and pyrophosphoric acids; but by treatment of the superphosphate with acid it may be used for the determination of the total phosphoric acid as well as the soluble, although, on account of the large amount of acid to be neutralised, the results are not quite so accurate.—J. F. B.

### *Phosphoric Anhydride, Estimation of Water-soluble and Citrate-soluble.* F. Martinotti. Staz. sperim. agric. ital **31**, 160—164; Chem. Centr. 1898, **2**, [2], 132.

The modification proposed by Appiani, and now used in place of the old (official) Italian method for the examination of superphosphate, is accurate, but is inconvenient and takes a long time. The author states that by the following method, results equally accurate can be obtained in a shorter time :—5 grms. of the superphosphate are treated in a 250 c.c. flask with 100 c.c. of water, then neutralised with caustic soda, using phenolphthalein as indicator, 100 c.c. of the official ammonium citrate solution added, the whole made up to the mark, and agitated for one hour by means of suitable apparatus. 50 c.c. (corresponding to 1 grm. of superphosphate) are now filtered off, warmed with a few drops of nitric acid, in order to convert any pyro- and metaphosphate present into orthophosphate; then 100 c.c. of water and 25 c.c. of ammonia added, and the phosphoric acid precipitated by 50 c.c. of magnesia mixture.—A. S.

### *Nitrites, Estimation of, in Water.* D. de Paepe. Bull. Assoc. Belge des Chim. **12**, [3], 98—102.

From an examination of the various colorimetric methods proposed for this purpose, the author accords the preference in point of delicacy, to—

(*a*) The Zambelli reaction :—α-Naphthol sulphanilic acid in an alkaline medium. Detects $\frac{1}{50}$ mgrm. per litre when a stratum of 10 cm. thickness is employed.

(*b*) The Riegler reaction:—β-Napthol naphthionic acid in an ammoniacal solution. Sensitiveness equal to *a*.

(*c*) The third Griess reaction :—*a*-naphthylamine sulphanilic acid, modified by Lunge and Lwoff. Capable of detecting $\frac{1}{100}$ and even $\frac{1}{1000}$ mgrm. per litre in a 10 cm. stratum.

The defect attaching to this last-named reaction, *viz.*, that the coloration develops slowly and fades quickly, may be obviated by heating the water to be tested, nearly to boiling before adding the reagent, and by subsequently maintaining it at about 80° C.—a procedure which will also restore the fugitive coloration, even after 48 hours, if air has been excluded.

The effect of various organic and inorganic matters in the water was investigated, and in the case of the antipyrin reagent proposed by Schuyten (this Journal, 1896, 743), only strong mineral acids, alkali sulphites and sulphides, salts containing basic hydrogen (*e.g.*, $Na_2HPO_4$), oxalic acid, and oxidising agents like $KMnO_4$, $CrO_3$, &c., exert a decolorising influence. Lime and the alkalis only destroy the reaction when in sufficient quantity to neutralise more than one-half of the acetic acid in the reagent.

Similar results were obtained with the Griess reagent above mentioned. In this case the reduction of the colour is manifested by a change from garnet-red to dirty orange-red. In water analysis the sulphides present are too dilute to have any effect on the reaction; and the oxidising influence on nitrites, with which ferric salts are credited by Schuyten, is believed by the author to be non-existent, his researches in this direction with ferric chloride having yielded negative results. Only in cases where—as in the antipyrin reaction—the coloration is weak, the colour of the acetic solutions of ferric salts may mask the true colour reaction. —C. S.

### *Arsenic in Antimony and other Metals, Estimation of.* O. Ducru. Comptes Rend. 1898, **127**, [4], 227—229.

In processes for the determination of arsenic in antimony, which involve solution in aqua regia, precipitation of the sulphides, oxidation of these, and precipitation of magnesium-ammonium arsenate, errors occur through the volatility of arsenic chloride and through the solubility of the arsenate. The distillation method of Clark (this Journal, 1887, 352) for the estimation of arsenic in copper may be employed with accuracy for many metals—iron, steel, lead, tin, zinc, bronze, &c. The method as applied in such cases is as follows:—About 5 grms. of the sample in coarse powder are placed in a distilling flask or tubulated retort, with condenser, the lower end of which dips into 50 c.c. of water contained in a graduated flask; 100 c.c. of ferric chloride solution and 200 c.c. of pure hydrochloric acid are added. When 100 c.c. have distilled over, 100 c.c. of acid are added to the contents of the retort; this is repeated until about 300 c.c. have been collected. The distillate contains a little antimony; it is redistilled, the receiver being washed out with hydrochloric acid. The first 300 c.c. contain all the arsenic; if 50—100 c.c. of sulphuric acid be also added, it is sufficient to distil only 150 c.c., but in this case the distillation should be assisted by a slow current of air. The distillate is diluted and precipitated by sulphuretted hydrogen, and the sulphide collected on a weighed filter. In the distillations, to prove that all the arsenic has come over, dilute a portion of the distillate with its own volume of water and pass in sulphuretted hydrogen: the absence of a precipitate after some minutes indicates the absence of arsenic.

The ferric chloride solution is prepared by dissolving 800 grms. of iron in 2 litres of hydrochloric acid and 2 litres of water, saturating with sulphuretted hydrogen, allowing to settle, and filtering, boiling off excess of the gas, oxidising by chlorine, and driving out the excess by an air current, and finally diluting to 5 litres with pure hydrochloric acid. This reagent and the hydrochloric acid should be tested for arsenic by distillation with 15 grms. of pure ferrous sulphate.—A. C. W.

*Tin and Antimony Alloys, Analysis of.* A. Fraenkel Mitt. des k. k. tech. Gewerbe-Museums in Wien. 1898, **8**, [11 and 12], 334—336.

The author proposes the following method for the analysis of alloys containing tin, antimony, lead, copper, and phosphorus. After treating the alloy with nitric acid, the residue containing the oxides of the above metals, together with stannic phosphate, is fused in a silver crucible with caustic soda, the melt is dissolved out with water, one-third the volume of alcohol is added, and the solution is filtered after about 24 hours. The alkaline filtrate (F) contains sodium stannate and phosphate and the lead oxide, whilst the residue (R) consists of sodium antimonate and copper oxide. The alkaline filtrate F is treated with sulphuretted hydrogen, and the lead determined in the usual manner. The tin sulphide is precipitated from the filtrate by sulphuric or acetic acid, converted into oxide, and weighed, whilst the phosphoric acid is determined in the concentrated filtrate. The residue R is dissolved in tartaric-hydrochloric acid, the antimony and copper precipitated with sulphuretted hydrogen, and separated by digesting the precipitate with ammonium sulphide, the antimony being precipitated from the filtrate with an acid, collected on a tared filter, and an aliquot part heated in a current of carbon dioxide to determine the amount of antimony trisulphide. It is also possible to precipitate the antimony as insoluble oxide by evaporating the nitric acid solution and moistening the residue with a few drops of nitric acid. If, however, it is suspected that some of the antimony is dissolved in the acid, it can be determined as above, along with the copper, after removing the lead with sulphuric acid; and the principal amount being present in the residue R, preferably the ammonium sulphide solutions are all mixed together.

—T. A. L.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

*Acetone, New Reaction of, and New Method for Distinguishing Aliphatic Amines.* E. Rimini. Annali Farmac. e Chim. 1898, 193—196; Chem. Centr. 1898, **2**, [2], 132.

Acetone gives, with primary amines in presence of sodium nitroprusside, a red-violet coloration that is quite distinct from the colour obtained in Legal's and Le Nobel's reactions. With secondary and tertiary amines, Legal's reaction takes place—formation of an orange-red colour. No other aldehydes or ketones give this reaction with primary amines; but pyruvic acid gives a similar coloration. The test may be used for the direct detection of acetone in presence of aceto-acetic acid and urine, as the latter do not give the coloration; and, in conjunction with the reaction of aldehydes with secondary amines in presence of sodium nitroprusside, it may be used for distinguishing between amines. If a fatty amine, in presence of sodium nitroprusside, gives a red coloration with acetone, it is a primary amine; if it gives a blue coloration with aldehyde and sodium nitroprusside, it is a secondary amine; and if it gives no coloration at all, it is a tertiary amine.—A. S.

*Spirits, Detection of Renatured (Repurified) Spirit in.* H. Herzfeld. Zeits. für öffentl. Chem. **4**, 389; Chem. Centr. 1898, **2**, [2], 133. (See also this Journal, 1898, 782.)

The author states that a denatured spirit may be completely freed from pyridine by adding 10—15 times as much sulphuric acid as is necessary for combining with the pyridine, and then distilling without first neutralising. For the detection of renatured spirit, it is recommended to test for acetone rather than pyridine, as the acetone cannot be separated from denatured spirit by distillation, even with a good rectifying apparatus.—A. S.

*Olive Oil, Method of Detecting Cotton, Sesamé, and Earthnut (Arachis) Oils in.* M. Tortelli and R. Ruggeri. Chem. Zeit. 1898, **22**, [60], 600—603.

The authors have combined their process for the detection of cotton-seed oil (this Journal, 1898, 607) with modifications of Baudouin's test for sesamé oil and Renard's test for earthnut oil. The liquid fatty acids obtained in the manner described, though in this case from 20 grms. of the oil instead of 5 grms., are first tested for cotton-seed oil by their modification of Becchi's test. Another portion of the liquid acids is tested for sesamé oil with furfural and hydrochloric acid. The authors have found that the whole of the substance which gives the violet coloration in sesamé oil dissolves in the liquid fatty acids, whereas none of that which causes some kinds of pure olive oil to give a very similar coloration, in this test (this Journal, 1898, 275) is ever present in them. It is stated that as little as 1 per cent. of sesamé oil can be detected in this way.

In order to detect the presence of earthnut oil, the lead soaps remaining insoluble in ether, are washed with ether, and shaken in a flask with 220 c.c. of ether, and a little 20 per cent. hydrochloric acid, in order to liberate the fatty acids. The ethereal solution of these is washed with water, filtered, the ether distilled off, and the residue cautiously shaken in a stoppered flask over the hot water bath, with 100 c.c. of 90 per cent. alcohol, until a clear solution is obtained and the temperature has risen to about 60° C. The flask is then transferred to a white surface and the solution allowed to cool. If earthnut was present in the original oil, a deposit consisting of a mixture of fine needle-shaped crystals (lignoceric acid) and thin tabular crystals (arachidic acid) is obtained. This crystalline precipitate is thoroughly characteristic of earthnut oil, and appears when the olive oil contains even less than 5 per cent. of that oil. The temperature at which the crystallisation commences gives an approximate idea of the amount of earthnut oil originally present, as is shown in the following table:—

*Solution in* 100 *c.c. of* 90 *per cent. Alcohol at about* 60° *C.*

| | | | | |
|---|---|---|---|---|
| Temperature at which crystallisation commences. | 35°—38° | 31°—33° | 28°—30° | 25°—26° |
| Original amount of earthnut oil in the mixture. | 100 | 60 | 50 | 40 |
| Temperature at which crystallisation commences. | 22°—24° | 20·5°-21·5 | 18°—20° | 16°—17 |
| Original amount of earthnut oil in the mixture. | 30 | 20 | 10 | 5 |

If a more exact determination is required, the precipitate which forms in the alcohol, after standing for about three hours at 15°—20° C., is collected on a filter and washed with three successive portions (10 c.c. each) of 90 per cent. alcohol and then with 70 per cent. alcohol. It is dissolved off the filter with boiling absolute alcohol, the latter completely evaporated, and the residue re-crystallised from 90 per cent. alcohol. The precipitate is collected and washed in the same way as before, and is finally dissolved off the filter into a weighed basin by means of boiling absolute alcohol. The solvent is evaporated and the residue dried to constant weight at 100° C., and its melting point determined. By this process a mixture of lignoceric and arachidic acids, melting at 74° to 75·5°, is obtained, and from the weight of the residue, plus the amount which has dissolved in the 90 per cent. alcohol, the percentage of earthnut oil in the original mixture can be calculated. The co-efficients of solubility of arachidic and lignoceric acids in alcohol are different, but the necessary allowance can be made by reference to the subjoined numbers experimentally determined by the authors :—

| Of a Mixture of the Acids melting at 74°—75·5°. | 100 c.c. of 90 per Cent. Alcohol dissolve at | | |
|---|---|---|---|
| | T=15° | T=17·5° | T=20° |
| | Grm. | Grm. | Grm. |
| 1. Weight between 2·7 and 0·50 grm. | 0·070 | 0·080 | 0·090 |
| 2. „ „ 0·47 and 0·17 „ | 0·050 | 0·060 | 0·070 |
| 3. „ „ 0·11 or less „ | 0·033 | 0·040 | 0·045 |

The authors state that according to their experiments the quantity of the mixture of these acids normally present in earthnut oil fluctuates between 4·30 and 5·40 per cent., but 4·80 per cent. may be taken as the mean for practical purposes. Hence the combined residue and dissolved portion multiplied by 5 gives the original percentage of that oil in the mixture.

By the original method of Renard and its modifications, 4 to 5 per cent. of arachidic acid, melting between 69·5° and 72°, is obtained from earthnut oil, whilst by the author's process the proportion of the two acids and their melting point is as mentioned above. This difference is attributed partly to loss during manipulation and partly to too low a solubility coefficient in Renard's process. Renard found that 100 c.c. of 90 per cent. alcohol dissolved 0·022 grm. of arachidic acid at 15° C. and 0·045 grm. at 25°, and these co-efficients have since been used in calculating the percentage of earthnut oil in a mixture. In no case, however, have the authors obtained so small a co-efficient of solubility in their practical determinations, even when the percentage of earthnut oil in a mixture was as low as 5 per cent. The table of the solubility of the mixed acids which is given in brief above, is given again more fully in an appendix, and the authors conclude their paper with a table giving the results of the determination of earthnut oil mixed in known proportions with olive oil. According to these the method is capable of estimating the oil within 1 or 2 per cent. of the actual proportion.—C. A. M.

*Carbolic Acid, on the Estimation of.* L. de Koningh. Nederlandsch Tijdsch. voor Pharm. Chem. en Tech. 1898.

When using Koppeschaars's bromine process it often happens that the liquid on being titrated with thiosulphate keeps turning blue again. This is caused by bromine (or iodine ?) being retained by the oily tribromocresol, and only slowly dissolving in the supernatant fluid. When this happens, the author pours off the liquid and dissolves insoluble matter in a little alcohol; a 20 per cent. solution of potassium iodide is added, and after the addition of some starch-water the titration is continued.

To estimate the amount of cresol in powders containing slaked lime, he operates as follows:—10 grms. of the powder are rubbed in a mortar with water, and the diluted sulphuric acid is added until the mass has a distinctly acid reaction. After the lapse of a quarter of an hour, it must be seen to once more that the acid reaction is persistent. The whole is now transferred to a suitable measure and made up with water to 1,007 c.c.; the extra 7 c.c. compensate for the volume occupied by the undissolved matter. After repeated shaking, the liquid is allowed to settle clear, and 100 c.c. are then pipetted off for the Koppeschaar process.

*Albumin, Testing of Commercial.* Ann. Chim. Anal. Appliq. 1897, 241; through Leipziger Färber- u. Zeügdr. Zeit. 1898, **47**, [7], 289.

Commercial albumin is generally adulterated with gelatin, dextrin, and gum. It may be spoiled through partial coagulation.

The gelatin present may be approximately estimated by taking 2 grms. of albumin and stirring up with a small quantity of water. A possible addition of flour is thus easily detected. The whole is then diluted with water to 200 c.c. when the solution should remain clear. If turbid, partial coagulation has taken place.

To 100 c.c. of this solution, add 35 c.c. of a 1 per cent. solution of tannic acid, and about 0·2 grm. of powdered tartar; shake up and filter. One-half of the filtrate is mixed with a half per cent. solution of gelatin (25 c.c. of which correspond with about 0·1 grm of pure commercial tannic acid); if the albumin is pure no precipitate here occurs; if the gelatin produces a precipitate, the tannic acid is obviously in excess, and in this case the albumin contains adulterant or has partially coagulated.

The other half of the filtrate is treated with a further addition of tannic acid, and any precipitate indicates that the albumin has been adulterated with gelatin or similar substances. The amount of tannic acid necessary for precipitation gives approximately the amount of gelatin present, since the latter can precipitate four times as much tannic acid as the same weight of albumin.

The presence of gelatin and dextrin in albumin can also be shown by heating its solution on the water bath. The albumin coagulates and is separated by filtration. If the albumin is pure, the filtrate produces no precipitate with tannic acid.

Dextrin and gum can be determined in the filtrate by the usual methods of examination.—J. E. H.

*Alkyl Phosphates and Phosphoric Acid, Analysis of a Mixture of.* J. Cavalier. Comptes Rend. **127**, [1], 60—61.

The salt $PO_4R_2H$ (R = Ethyl, methyl, or allyl) is acid both to methyl orange and to phenolphthaleïn, and is rendered neutral by one molecule of alkali. The salt $PO_4RH_2$ is rendered neutral to methyl orange by one molecule of alkali; to phenolphthaleïn by two; and its barium salt is soluble in water. Phosphoric acid is rendered neutral to methyl orange, in the cold, by one molecule of alkali, then at 60° C. by a second; and if the base be baryta, the whole of the phosphoric acid is thrown down as $PO_4BaH$, while the precipitate does not interfere with the delicacy of the indicator. A mixture of $PO_4R_2H$, $PO_4RH_2$, and $PO_4H_3$, then, is titrated with baryta and methyl orange in the cold; this gives the sum of the three. The titration is continued at 60° C. with phenolphthaleïn; this gives the last two. The liquid is now filtered; filtrate and precipitate are separately treated with known amounts of standard hydrochloric acid, so as to get clear solutions acid to methyl orange, and the titration between neutrality to methyl orange and neutrality to phenolphthaleïn is in each case undertaken again. Thus we get separately the amounts of dihydrogen and trihydrogen compounds, which should add to their sum as obtained before.—J. T. D.

*Malic Acid, Determination of, in Grape Juice.* A. Girard and Lindet. Bull. Soc. Chim. 1898, **19**, [13], 585.

The tartaric acid (free and combined) is first determined by the method of Berthelot and Fleurieu, in which the tartar is precipitated by a mixture of alcohol and ether, and the free tartaric acid by saturating about one-third of the liquid (containing the alcohol and ether) with potash and adding the other two thirds. After filtering off the total hydrogen potassium tartrate, the alcohol and ether are expelled by evaporation, water added, and normal lead acetate added to the boiling solution until there is a permanent turbidity.

The boiling liquid is filtered and the lead malate allowed to crystallise. The next day the mother-liquor is treated in the same way, and the further deposit of lead malate collected. The process is again repeated with the mother-liquid, and so on, until no more crystals deposit on cooling. The total lead malate is dried, detached from the filter, and weighed. Since lead malate is soluble to some extent in acetic acid, a correction must be made at the end of the determination for the amount of free acid in the mother-liquid. This can be done by means of the following table :—

| Quantity of Acetic Acid in 100 c.c. of Mother-Liquid. | Lead Malate dissolved in 100 c.c. of Mother-Liquid. |
|---|---|
| 0·21 | 0·10 |
| 0·40 | 0·13 |
| 0·55 | 0·16 |
| 0·73 | 0·18 |
| 0·90 | 0·20 |

The authors state that they have obtained very satisfactory results in test experiments with this method.—C. A. M.

*Fats, Determination of Unsaponifiable Matters in.* Schukow and Schestakow. Zap. imp. russk. techn. obschtsch. 1898, **32**, 109.

The differences between different methods of determination of unsaponifiable matters in fats are due to the splitting up with water of neutral soaps, with production of acid soaps, which are soluble in benzene or ether, but insoluble in water, and which can only be separated from the unsaponifiable matters by thorough washing with weak alkali. The authors have obtained very exact results by the following combination of Allen and Thomson's method with that of Demski and Morawski:—Mix 5 grms. of the fat in a glass basin with 25 c.c. of 8 per cent. alcoholic sodium hydroxide, evaporate on the water-bath, dissolve the soap in 50 c.c. of water, bringing it and the insoluble portion into a separating funnel. After cooling, add alcohol till the solution clears, avoiding excess; then add 50 c.c. of ether, and shake. Separate, and repeat twice the treatment with ether. Evaporate the combined ethereal extracts (filtering first if any precipitate of calcium soap separates). To the residue add phenolphthalein, treat several times with petroleum spirit (b.p. 80°), filter into a tared flask, evaporate the petroleum spirit, and weigh the dried residue. Bone fats contain from 0·4 to 2·0 per cent. unsaponifiable matter; beef- or mutton-tallow, 0·2 to 0·5 per cent.—J. T. D.

*Alkali Oleate, Reagent for Hydrometry and the Estimation of Metals forming insoluble Oleates.* F. d'Huart. Bull. Assoc. Belge des Chim. **12**, [2], 56—57.

The reagent is prepared by mixing normal alkali solution with an equivalent weight of pure oleic acid, and dissolving the product in a minimum volume of 96 per cent. alcohol, the whole being finally made up to 10 times the original volume of alkali solution taken. Any foam produced is dissipated by a little alcohol.

If the article taken up as "pure oleic acid" be found to contain traces of mineral acids, the solution must be standardised by means of decinormal silver nitrate and adjusted with alkali, so that 1 c.c. of the silver solution in 50 c.c. of water will froth when agitated with 1·1 c.c. of the oleate reagent (0·1 c.c. being needed, to form the froth).

The solubility of the neutral oleate in alcohol being relatively low, the solution may become turbid; but the acid oleates (*e.g.*, with five equivalents of oleic acid) give a clear solution, which may be standardised for use by decinormal silver solution.

In hydrometric tests, the oleate reagent directly expresses the degree of hardness (French scale), provided that 50 c.c. of water be taken instead of 40 c.c.

To obviate the retardation of frothing, occurring in the case of concentrated solutions, owing to the bulk of the precipitate formed, it is advisable to operate on solutions of the same concentration as decinormal reagents.—C. S.

*Soaps, Analysis of.* R. Hefelmann and E. Steiner. Zeits. für öffentl. Chem. **4**, 389—396; Chem. Centr. 1898, **2**, [2], 142.

The authors describe the different methods for the separation and drying of the fatty acids from soap solutions. Späth (this Journal, 1896, 139) takes up with petroleum spirit the fatty acids separated by normal acid, but the authors found that no complete solution could thus be obtained in the case of cocoanut-oil soap, white flakes of an oxy-fatty acid being left behind. In the drying of the evaporation residue, two sources of error come into consideration: (1) evaporation and (2) oxidation of the fatty acids. These errors, however, compensate each other as a rule, and, with pure oleine soaps, cause no appreciable difference. Drying in presence of indifferent gases, in order to avoid oxidation, as advised by Späth, is thus not necessary, especially if the drying be carried out at 55° instead of 100°. The error due to volatilisation is much more considerable in the case of soaps containing lower fatty acids, as cocoanut-oil soap, though this fact appears to have been wholly overlooked up to now. It was found impossible, in the case of cocoanut-oil soap, even when guarding against oxidation, to bring the ether or petroleum spirit residue to a constant weight at 100° C.; the longer the time of drying, the greater the volatilisation of the lower fatty acids, and at the same time oxidation takes place. Thus there is a corresponding diminution of the acid and iodine numbers, and increase of the refractometer number, as was proved by experiments with a tallow-cocoanut oil soda soap and a pure cocoanut-oil soap, the losses being particularly considerable in the case of the latter. In order to avoid these losses, the authors neutralise the ethereal fatty acid solution with absolute ½N alcoholic potash or soda, using phenolphthalein as indicator, evaporate, and dry the mixture of neutral fats and alkali salts of the fatty acids to constant weight at 100° C. From the weight obtained, together with the amount of alkali used, the amounts of fatty acids and of total fat may easily be calculated. The results obtained by this method correspond with those obtained by direct drying of the evaporation residue, if the drying be carried out at about 55° C., as recommended by Saupe; and the authors state that correct and constant results can be obtained in all cases, if this low temperature be used.—A. S.

*Starch, Estimation of, in Cereals.* C. J. Lintner. Zeits. f. angew. Chem. 1898, [32], 725—729.

The starch in three different barleys was determined by the following methods :—

1. Three grams of the finely ground barley were triturated with 25 c.c. of a 1 per cent. solution of lactic acid and 30 c.c. of water, and heated for three hours at 3½ atmospheres, filtered, and washed with hot water until the volume of the filtrate was 200 c.c. To this was added 15 c.c. of hydrochloric acid (sp. gr. 1·125), and the mixture inverted by heating for three hours in a boiling water bath, neutralised, made up to 500 c.c. and 25 c.c. of the filtrate used for the reduction by Allihn's method. The quantity of starch was found by multiplying the glucose by the factor 0·9.

2. Three grams of material were converted to a paste by heating with 50 c.c. of water, cooled to 70° C., and kept at this temperature for twenty minutes with 20 c.c. of malt extract (20 grms. of malt extracted for two hours with 200 c.c. water); 5 c.c. of 1 per cent. tartaric acid solution was added and the whole heated for half an hour at three atmospheres, then cooled to 70° C., and digested again with 10 c.c. of malt extract until the disappearance of the iodine reaction. The solution was made up to 250 c.c., 200 c.c. of it inverted, and the glucose determined in it as in the first process, a correction being made for the reducing power of the malt extract.

3. Three grams of material are treated with ether on a filter until the fat is removed, and the dry filter and substance boiled for half an hour with 100 c.c. of water, cooled to 65° C., 10 c.c. of malt extract (prepared as above) added, and digested at 65° C. for half an hour, then boiled for a quarter of an hour, cooled, and treated again at 65° C. with

10 c.c. of malt extract, cooled, made up to 250 c.c., and 200 c.c. of the filtrate inverted as in the first process, the subsequent operations and corrections for the malt extract being the same.

The results obtained by these different methods were :—

| Wheat. | First Method. | Second Method. | Third Method. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| 1 | 68·50 | 67·51 | 63·22 |
| 2 | 67·90 | 65·98 | 63·68 |
| 3 | 69·00 | 67·50 | 63·93 |

It will be observed from these figures that the longer the heating under pressure with acids is continued, the greater is the amount of apparent starch. The influence of the pentosans upon the results of the starch estimations has been determined by Tollens' phloroglucinol method (this Journal, 1896, 744). When the percentage of pentosans is subtracted from that of the starch found by the above described methods, the value becomes about 61·1 per cent. Corrections were also made for the pentosans contained in the malt extract. Wiley and Krug (this Journal, 1898, 498) have stated that when diastase acts on the starch of cereals, &c., pentosans do not enter into solution ; but the author cannot confirm this observation.

Several points in the above analyses are discussed, and stress is laid on the question as to whether the hydrolysed products of the pentosans have a different reducing power to glucose ; for, if such be the case, an obvious error arises. Although there are differences of opinion affecting the relation of the reducing powers of arabinose and glucose, the author considers, on the evidence afforded by the comparison of the amounts of pentosans as determined by Tollens' method, and, on the other hand, by determining the reducing power of a fermented inverted starch solution, that the reducing powers are practically similar. It is therefore permissible to subtract the percentage of pentosans from that of the apparent starch.

To obtain as true a value as possible of the starch in cereals the author recommends this process : 3 grms. of finely ground material are mixed with 200 c.c. of water and 15 c.c. of hydrochloric acid (sp. gr. 1·125) and inverted in a boiling water bath for three hours, then neutralised, cooled, made up to 500 c.c., filtered, and the reducing power determined in 25 c.c. of the filtrate, as described in the first method (*loc. cit.*). The pentosans are estimated by distilling 133 c.c. from 200 c.c. of the inverted solution, adding to it 33 c.c. of hydrochloric acid (sp. gr. 1·18) and then treating according to Tollens' directions (*loc. cit.*)—J. L. B.

*Methyl Alcohol in Ethyl Alcohol, Recognition and Estimation of.* A. Trillat. Comptes Rend. 1898, **127**, [4], 232—234.

Among the products of the oxidation of ethyl and methyl alcohols by potassium bichromate and sulphuric acid are acetal and formal, $CH_2(OCH_3)_2$, respectively. These compounds form condensation products with dimethylaniline, tetramethyldiamidodiphenylethane, and tetramethyldiamidodiphenylmethane, $CH_2[C_6H_4N(CH_3)_2]_2$. Each of these compounds gives a blue coloration on oxidation, but the coloration obtained from the former disappears rapidly under the influence of heat, whilst the coloration resulting from the latter becomes more intense. By the following process 0·5 per cent. of methyl alcohol in absolute alcohol may be detected. To a mixture of 20 c.c. of the alcohol and 300 c.c. of 10 per cent. bichromate solution, 100 c.c. of sulphuric acid (1 : 4) are slowly added, with cooling. After standing an hour the liquid is distilled; the first runnings are rejected, the distillate is exactly neutralised, redistilled and the distillate made up to 400 c.c. The distillations are pushed very far. In a small bottle, to 100 c.c. of the distillate are added 2 c.c. of carefully distilled dimethylaniline and 1 c.c. of sulphuric acid (10 per cent.). The bottle is stoppered and warmed at 65°—70° C. on the water bath for 5 hours, the contents are made alkaline and the excess of dimethylaniline removed by a current of steam. The blue coloration, indicative of the presence of methyl alcohol, is developed on acidifying a portion of the liquid with acetic acid, and adding a little lead dioxide suspended in water. In order to make a quantitative determination, the coloration obtained, must be compared with standards. The intensity is proportional to the amount of methyl-alcohol.—A. C. W.

*Tannin, The Cause of Error in the Gravimetric Estimation of.* L. Maschke. Dingler's Polyt. J. 1898, 46.

Most chemists are now agreed on the superiority of the hide-powder method for the estimation of tannin, but there is still great differences in the manner of carrying this out. Some chemists employ the hide-filter method as recommended by Procter. The successful working of this method, however, depends on the operator's skill in the filling of the hide-filter, and on this account is rapidly losing favour, and the shake method is rapidly replacing it. When a good hide-powder is used, this method gives results which are absolutely comparable with one another. The advantage of the shake method lies in the fact that one can introduce a correcting factor for the solubility of the hide-powder, but unfortunately, unless the hide-powder be of first-class quality, even this method is not perfectly accurate. Below is a table of analyses of six materials made each with two hide-powders, the factor of solubility of one of which for 50 c.c. equalled 0·039 grm. and of the second quality 0·1725 grm. The analyses were in every case conducted simultaneously.

| — | Tannins. | Non-Tannins. | Tannins. | Non-Tannins. | Tannins. |
|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Oak wood extract. | 25·20 | 20·00 | 34·80 | 10·20 | 9·60 |
| „ „ | 26·00 | 20·50 | 40·90 | 5·60 | 14·90 |
| „ „ | 26·50 | 12·90 | 38·30 | 2·00 | 11·80 |
| Quebracho extract. | 57·20 | 12·20 | 68·20 | 8·10 | 11·00 |
| „ „ | 74·30 | 12·80 | 87·20 | .. | 19·90 |
| „ „ | 70·30 | 14·30 | 79·30 | 5·60 | 9·00 |
| Quebracho wood. | 19·20 | 1·80 | 22·50 | 1·50 | 3·30 |
| „ „ | 20·40 | 0·70 | 22·40 | 1·30 | 2·00 |
| Valonia...... | 23·30 | 15·10 | 31·00 | 7·30 | 7·70 |
| „ ...... | 27·80 | 12·10 | 36·00 | 4·20 | 8·20 |
| Pine bark.... | 13·10 | 9·00 | 17·40 | 3·10 | 4·30 |
| „ „ .... | 13·60 | 8·80 | 19·60 | 2·80 | 6·00 |
| Mimosa bark. | 36·80 | 11·60 | 44·30 | 5·10 | 7·50 |

As will be seen from the above figures, the differences are enormous, and more marked when the substances analysed contained a large percentage of non-tannins. Up to now no hide-powder has been made which contained no soluble matter, and we are bound to use the hide-powder which contains the least amount of soluble substances. But until a perfectly pure hide-powder is produced, it will be impossible for chemists to get identical results, and a good hide-powder will only be produced by the combined action of the interested parties, analytical chemists, or technical tanners who will undertake to instruct the manufacturer in the best processes for the manufacture of hide-powder and see that it is made according to instructions and from selected material.—J. G. P.

*Volatile Acids Produced in a Fermentation, Formulæ for Estimating by the Duclaux Method.* Bordas and Raczkowski. La Bière, **6**, [6], 87—88.

The following table expresses the results of a fractional distillation :—

| 1. Fraction. | 2. c.c. of Lime-Water. | 3. Total Volume of Lime-Water. | 4. Ratio $\frac{n_{10} \times 100}{N}$ | 5. Ratio $\frac{N \times 100}{N_r}$ |
|---|---|---|---|---|
| c.c. | | | | |
| 10 | $n_1$ | $n_1$ | $r_1$ | $a_1$ |
| 20 | $n_2$ | $n_1 + n_2$ | $r_2$ | $a_2$ |
| 30 | $n_3$ | $n_1 + n_2 + n_3$ | $r_3$ | $a_3$ |
| 40 | $n_4$ | $n_1 + n_2 +$ | $r_4$ | $a_4$ |
| 50 | $n_5$ | $n_1 + n_2 +$ | $r_5$ | $a_5$ |
| 60 | $n_6$ | $n_1 + \quad +$ | $r_6$ | $a_6$ |
| 70 | $n_7$ | $n_1 + \quad +$ | $r_7$ | $a_7$ |
| 80 | $n_8$ | $n_1 + \quad +$ | $r_8$ | $a_8$ |
| 90 | $n_9$ | $n_1 + \quad +$ | R | $a_9$ |
| 100 | $n_{10}$ | $n_1 + n_2 \ldots = N$ | 100·00 | A |

$N_1$ representing the volume of lime water required to neutralise the acids in the distilling flask. The nature of the acids is determined by the fourth column.

In the case of a single acid—

$$\frac{N \times 100}{N_1} = A, \text{ i.e., } N_1 = N \times \frac{100}{A};$$

and if $n$ represent the $\frac{1}{10}$ normal $H_2SO_4$ required to neutralise 20 c.c. of lime water, and $m$ the molecular weight of the acid in the liquid, then the weight of such acid per 110 c.c. of liquid is—

$$\frac{N \times 0{\cdot}00014 \times m \times n}{28 \times A},$$

which may be simplified, by substituting F for the constant factor—

$$\frac{0{\cdot}00014 \times m}{28 \times A},$$

into $A = F \times n \times N$.

The value of F is, for formic acid, 0·00039; for acetic acid 0·00037; for propionic acid, 0·00038; for butyric acid, 0·00045; and for for valerianic acid, 0·00051.

Where there are two acids the volumes $N'$ and $N''$ of lime water corresponding to each have to be determined. Here—

$$\frac{n_{10} \times 100}{N} = R, \text{ whence } n_{10} = \frac{NR}{100}$$

$$\frac{n'_{10} \times 100}{N'} = R', \text{ whence } n'_{10} = \frac{N'R'}{100}$$

$$\frac{n''_{10} \times 100}{N''} = R'', \text{ whence } n''_{10} = \frac{N''R''}{100}$$

from which it follows that $NR = N'R' + N''R''$, and $N = N' + N''$. The values of $N'$ and $N''$ are:—

$$N' = \frac{N(R - R'')}{R' - R''}, \quad N'' = \frac{N(R' - R)}{R' - R''}.$$

$\frac{1}{R' - R''}$ is constant for each acid mixture.

By the aid of the equations,—

$$F' = \frac{0{\cdot}014 \times m'}{28 \times A} \times \frac{1}{R' - R''}, \text{ and}$$

$$F'' = \frac{0{\cdot}014 \times m''}{28 \times A} \times \frac{1}{R' - R''},$$

the simplified formulæ $A' = F' \times n \times N(R - R'')$ and $A'' = F'' \times n \times N(R' - R)$ are obtained, representing the proportion of the acids in the mixture. The subjoined table has been drawn up to indicate these values in the mixtures of most frequent occurrence:—

| — | Formic. | | | | Acetic. | | | Propionic. | | Butyric. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Acetic. | Propionic. | Butyric. | Valerianic. | Propionic. | Butyric. | Valerianic. | Butyric. | Valerianic. | Valerianic. |
| Formic acid .......... | A' | A'' | A'' | A'' | .. | .. | .. | .. | .. | .. |
| Acetic acid .......... | A' | .. | .. | .. | A'' | A'' | A'' | .. | .. | .. |
| Propionic acid ........ | .. | A' | .. | .. | A' | .. | .. | A'' | A'' | .. |
| Butyric acid.......... | .. | .. | A' | .. | .. | A' | .. | A | .. | A'' |
| Valerianic acid ....... | .. | .. | .. | A' | .. | .. | A | .. | A | A |
| F'.................... | 0·000077 | 0·000028 | 0·000025 | 0·000025 | 0·000046 | 0·000035 | 0·000035 | 0·00011 | 0·000076 | 0·00020 |
| F''.................... | 0·000081 | 0·000030 | 0·000022 | 0·000020 | 0·000045 | 0·000030 | 0·000025 | 0·000095 | 0·000058 | 0·00018 |
| R'.................... | 84·4 | 93·0 | 97·0 | 79·6 | 93·0 | 97·0 | 99·5 | 97·0 | 99·5 | 99·5 |
| R''.................... | 79·6 | 79·6 | 79·6 | 95·9 | 84·4 | 84·4 | 84·4 | 93·0 | 93·0 | 97·0 |

—C. S.

*Spt. Æther. Nit., Assay of.* C. E. Smith. Chem. and Druggist, 1898, **53**, 951], 59; from Amer. J. Pharm.

The author alleges that Allen's method does not give reliable results, and he proposes the following modification of the chlorate process. 5 c.c of the spirit to be tested is mixed, in a flask or bottle of white glass, provided with a loosely-fitting stopper, with 10 c.c. of distilled water, 5 c.c. of a cold, aqueous, saturated solution of potassium chlorate, and 5 c.c. of 10 per cent. nitric acid. The mixture is shaken frequently during 30 minutes, then 10 c.c. of decinormal silver nitrate added, the whole shaken briskly, 10 drops of ferric ammonium sulphate solution added, and the excess of silver quickly titrated with decinormal potassium sulphocyanide. The end point is reached when, after momentary shaking, upon addition of the last drop of solution, the red colour produced is not entirely dispersed, but faintly tinges the whole of the liquid. The colour will frequently disappear after persistent shaking or after standing awhile, but no attention need be paid to this. The amount of ethyl nitrite present in the sample is calculated from the number of c.c. of silver nitrate used in precipitating the chloride formed, 1 c.c. of $\frac{1}{10}$N $AgNO_3$ corresponding to 0·0225 grm. of ethyl nitrite.—A. S.

*Coal, Indian: Chemical Examination of.* W. R. Dunstan. Imp. Inst. (Indian Sect.) Annual Report, 1897–98, 40—44.

*See under* II., *page* 850.

*Furfural from Starch, and its Derivatives; Formation of.* F. Sestini. L'Orosi, 21, 109—113; Chem. Centr. 1898, 2, [3], 182.

*See under* XVIII., A, *page* 861.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Hydrogen Peroxide as a Factor in Spontaneous Combustions.* L. H. La Wall. Amer. J. Pharm. 1898, **70**, [6], 291—293.

It is stated that, under favourable conditions, hydrogen peroxide may give rise to spontaneous combustion. An instance in which a piece of cheese cloth, saturated with hydrogen peroxide and bound round the wrist of a patient in the form of an antiseptic dressing, smouldered and caused severe burns, induced the author to experiment with these materials. It was found that when cheese cloth was merely moistened with the peroxide and exposed to the heat of a drying oven, charring did not take place, until the temperature reached 65° C. It is probable that the slight trace of free sulphuric acid present aided the action, since little or no charring was produced when neutralised peroxide was employed, whereas the addition of 1 per cent. of sulphuric acid markedly increased the charring effect.—J. O. B.

*Graphitic Acid, Preparation of.* L. Staudenmaier. Ber. **31**, [10], 1481—1487.

Gottschalk found that five successive operations were necessary to oxidise graphite completely, by the action of fuming nitric acid of sp. gr. 1·525 and potassium chlorate, to graphitic acid; and that the graphite passed through successive stages of oxidation, being converted after three operations into a grass-green substance. The author finds that this substance can be readily oxidised to graphitic acid by heating on the water-bath with potassium permanganate solution and dilute sulphuric acid. He prepares graphitic acid in considerable quantity, in a comparatively short time (24 to 48 hours), and without danger of explosion, by stirring very finely divided graphite (25 grms.) into a mixture of concentrated sulphuric acid (1 litre) and nitric acid of sp. gr. 1·4 ($\frac{1}{2}$ litre), and adding at intervals potassium chlorate (about 450 grms. in all). The mixture is kept at the ordinary temperature, is well stirred after each addition of chlorate, and each portion of chlorate is added only after the gas-evolution from the previous addition has completely ceased. When a sample treated with permanganate and acid becomes of a pure yellow colour, the acids are thoroughly washed with cold water from the powder, which is then oxidised with permanganate. Any excess of permanganate is destroyed by hydrogen peroxide, and the resulting graphitic acid washed successively with dilute nitric acid, alcohol, and ether.

—J. T. D.

### *Calcium Hydride, Preparation and Properties of.* H. Moissan. Comptes Rend. **127**, [1], 29–34.

Calcium is placed in a nickel boat in a glass tube, through which pure hydrogen is passed till all air is expelled. The end of the tube is then sealed and the hydrogen supply kept up under a pressure of 30 or 40 c.m. of water. The tube is gradually heated, and at low redness the calcium takes fire, rapidly absorbs hydrogen, and is converted into a white substance, calcium hydride. Not more than five or six grams of calcium should be contained in a single boat, lest the intense heat cause the metal to alloy with the nickel; but several boats may be contained in series in one tube. The hydride is white, with crystalline fracture; density, 1·7; not sensibly dissociated in a vacuum at 600° C.; not farther affected by hydrogen at the softening point of Bohemian glass; not affected by chlorine in the cold, but decomposed at low redness with incandescence, forming calcium chloride and hydrochloric acid; similarly acted on by bromine or iodine. It does not oxidise in air even at a bright red heat; but at the highest temperature of a blowpipe it burns, forming lime, which fuses and forms a protective coating to the portions beneath. In oxygen it takes fire at a low red heat, and the whole is converted into a fused mass of lime. In sulphur vapour action begins only at a very high temperature, but then goes on vigorously, calcium and hydrogen sulphides being formed. On selenium it has no action, nor has it on nitrogen. Phosphorus at 500° C. decomposes it, evolving hydrogen, and forming a substance which produces phosphine when thrown on water. Boron at 700° C. is without action. Heated with carbon in a brasqué crucible to 700° or 800° C., it forms calcium carbide; but neither boron nor silicon has any action under the same circumstances. Potassium or sodium fluoride, at 500° C., acts vigorously, with evolution of hydrogen and vapour of potassium or sodium. Silver fluoride acts violently, even in the cold, giving calcium fluoride and silver, with evolution of hydrogen; and at 400° C. a similar reaction takes place with fluoride of lead or of zinc. Sodium chloride, at red heat, acts very regularly, sodium vapour being given off. Fused potassium iodide does not act on the hydride, but silver iodide acts vigorously. Oxidisers, as potassium chlorate, perchlorate, chromate, or permanganate, are reduced with violence amounting to explosion. Hydrogen sulphide at a red heat forms calcium sulphide and hydrogen, nitrogen dioxide forms ammonia, and carbon dioxide forms carbon and calcium carbide. Strong sulphuric acid, if hot, is reduced by calcium hydride, but strong nitric acid is hardly attacked. Dilute acids act vigorously, forming calcium salts and hydrogen; in the case of hydrochloric acid the action is rapid, whether the acid be dilute or strong. Ethyl alcohol slowly attacks the hydride, but the alkyl chlorides or iodides, benzene, or turpentine, are without action. Carbon tetrachloride vapour at 400° C. acts vigorously, depositing carbon and giving off hydrogen and hydrochloric acid. Water decomposes it violently, forming lime, while the hydrogen both of the hydride and the water is evolved. The interest of this hydride lies in the fact that the hydrogen behaves like a non-metal; the substance is radically different in character from the potassium-hydrogen and sodium-hydrogen alloys of Troost and Hautefeuille, or from Graham's hydrogenised palladium.—J. T. D.

### *Magnesium Sulphide, Crystalline Anhydrous.* A. Mourlot. Comptes Rend. 1898, **127**, [2], 180—183.

According to the author, amorphous magnesium sulphide is best prepared by passing hydrogen sulphide over magnesium filings mixed with a little sulphur heated in a porcelain tube, or by the action of hydrogen sulphide on magnesia or anhydrous magnesium sulphate at 1,200° C. By heating the amorphous sulphide in a carbon tube in an electric furnace, using a current of 1,000 ampères at 50 volts, or by similarly heating a mixture of magnesium chloride (1 mol.) and stannous sulphide (1 mol.), magnesium sulphide was obtained in spherical masses of yellowish colour, whose fractures often exhibited cavities bordered with cubical crystals. These crystals, of sp. gr. = 1·85, have two cleavage planes at right angles to one another; they have no action on polarised light. This crystalline sulphide is not so readily attacked as the amorphous sulphide; its reactions are similar to those of crystalline barium sulphide (this Journal, 1898, 502), which it resembles too in resisting the action of carbon in the electric furnace. It reacts energetically with chromyl chloride, forming a green substance which contains chromium and sulphur. Attempts to prepare magnesium sulphide by reducing the anhydrous sulphate with carbon in the electric furnace resulted only in incomplete reduction to sulphide, some oxide being also formed.—E. W. W.

### *Monobarium and Monocalcium Phosphates, Decomposition of, by Water at 100° C.* Georges Viard. Comptes Rend. 1898, **127**, [2], 178—180. (Compare this Journal, 1894, 634—635.)

The author finds that when monobarium phosphate, $BaH_4(PO_4)_2$, is heated with water in a sealed tube at 100° C., or boiled with water under the ordinary pressure, it is decomposed into phosphoric acid and dibarium phosphate, $Ba_2H_2(PO_4)_2$, which is deposited in a crystalline form. As the amount of phosphate is increased, the ratio R, obtained by dividing the total phosphoric acid by the amount of combined phosphoric acid, increases at first very rapidly, then more slowly, until a constant value of 2·79 is obtained, when for every 100 grms. of water 75 grms. of phosphate are used. Monocalcium phosphate behaves in a similar way, yielding dicalcium phosphate, $Ca_2H_2(PO_4)_2$, and R attains a constant value of 2·34, when 50 grms. of phosphate are treated with 100 grms. of water.—E. W. W.

### *Perborates and their Constitution.* S. Tanatar. Zeits. physik. Chem. **26**, 132—134; Chem. Centr. 1898, **2**, [3], 171.

Sodium perborate is formed by the electrolysis of a concentrated solution of sodium orthoborate, or still more easily by the addition of 120 c.c. of 3 per cent. hydrogen peroxide to a solution of 20 grms. of borax and 4 grms. of caustic soda in a little water. It separates after some time in the cold, as small, slightly-soluble crystals, having the composition $NaBO_3 + 4H_2O$. The crystals are filtered off and washed with alcohol and ether. 15 grms. of the pure salt are obtained from 20 grms. of borax. It is quite stable if preserved dry, but in solution, especially in the warm, it is decomposed with evolution of oxygen. It reacts with permanganate in the same way as hydrogen peroxide. The ammonium salt, $NH_4BO_3 + H_2O$, is prepared like the sodium salt, but is much more soluble in water, and must be precipitated by alcohol from the solution in which it is formed. Perborates are very energetic oxidising agents, and are preferable to hydrogen peroxide, on account of their stability and large content of active oxygen—10 and 16 per cent.

—A. S.

### *Chromous Salts, Decomposition of Water by, and their use as Absorbers of Oxygen.* Berthelot. Comptes Rend. **127**, [1], 24—27.

The heat of formation of $Cr_2Cl_4O$ from $CrCl_2$ and oxygen is greater than the heat of formation of water. Chromous chloride, however, in perfectly neutral solution, does not decompose water at all at ordinary temperatures, though above 250° C. it does so. But if the solution be even slightly acidified with hydrochloric acid, slow decomposition sets in, with gradual evolution of hydrogen. The same thing occurs with the chromous chloride solution obtained by acting with zinc and hydrochloric acid on a solution of chrome alum. It follows that acidified chromous chloride is not a reagent to be employed in accurate work, either as an absorbent in gas analysis, or for the purification from oxygen of any gas save hydrogen.—J. T. D.

*Copper Selenate, A Source of Selenic Acid.* R. Metzner. Comptes Rend. **127**, [1], 54—57.

SELENIOUS acid, in solution not stronger than 10 per cent., is completely oxidised by a current of chlorine, and the selenic and hydrochloric acids are then nearly neutralised by copper oxide prepared from the nitrate by calcination, the neutralisation being completed by the precipitated, well-washed, boiled hydroxide. The cupric selenate is separated easily by crystallisation from the chloride, as its solubility varies rapidly with the temperature, a litre of the saturated solution containing, at 15°, 35°, and 55° C. respectively, 257, 346, and 435 grms. of salt. By electrolysing the solution of the purified crystals (5 amps. at 2 to 3 volts.), a solution of pure selenic acid is obtained, free from copper, chlorine, and selenious acid.—J. T. D.

*Acetylene and Cuprous Oxychloride, Compound of.* R. Chavastelon, Comptes Rend. **127**, [1], 68—69.

IF the compound $C_2H_2.Cu_2Cl_2$ (Comptes Rend. June 25, 1898) be dropped into water, acetylene is slowly evolved, and the crystal becomes violet on the surface. After several days this violet colour becomes uniform throughout the crystal, and when this point is reached, an analysis of the crystal shows it to have the formula $C_2H_2.Cu_2Cl_2.Cu_2O$; its formation is expressed by the equation—

$$2(C_2H_2Cu_2Cl_2) + H_2O = C_2H_2.Cu_2Cl_2.Cu_2O + C_2H_2 + 2HCl.$$

—J. T. D.

*Toluene, Some Fluorine Derivatives of.* F. Swarts. Bul Acad. roy. Belgique, **35**, [3], 375—420; Chem. Centr 1898, **2**, [1], 26.

Two fluorine derivatives of toluene were prepared by the action of antimony fluoride on benzo-trichloride; the attempt to obtain the third monofluordichlortoluene in this way, proved unsuccessful. The fluorine compounds were best obtained by adding two-thirds of the weight of antimony fluoride to one part of benzotrichloride, heating the mixture to boiling as quickly as possible, and keeping it boiling for about five minutes. In this way were obtained difluorchlortoluene, $C_6H_5CF_2Cl$, a colourless liquid with constant vapour density; b.p., at 770 mm., 142·6° C.; sp. gr., at 13° C., 1·25415; refractive index, 1·46969; and trifluortoluene, $C_6H_5CF_3$, a colourless liquid also with constant vapour density; b.p., 103·5° C., sp. gr., at 14° C., 1·19632; refractive index, 1·41707. Trifluortoluene or phenylfluoroform is stable towards water, alkalis, phenols, aniline, reduced copper, phenylhydrazine and high temperatures, but is converted by fuming nitric acid into *m*-nitrotrifluortoluene.—A. S.

*Nitrophenates, Action of Carbonic Acid on.* Monnet and Benda. Bull. Soc. Chem. 1898, **19**, [15], 688—692.

THE nitrophenols dissolve in alkaline carbonate solutions, with liberation of carbonic acid; in consequence, it is generally stated that the nitrophenates are not decomposed by carbonic acid. The authors find this is not the case. If pure carbon dioxide be passed into aqueous solutions of sodium *o*-, *m*-, or *p*-nitrophenol, the nitrophenol is precipitated in quantity. In the case of *p*-nitrophenol, the acid salt, $(C_6H_4.NO_2.ONa)(C_6H_4.NO_2.OH)2H_2O$, is first thrown down, but from more dilute solution, the free nitrophenol separates on cooling, or can be extracted with ether.

—A. C. W.

*d-Gluconic Acid, Transformation of, into d-Arabinose.* O. Ruff. Ber. **31**, [10], 1573—1577.

CROSS, Bevan, and Smith (this Journal, 1898, 591) found in the course of their experiments on the action of hydrogen peroxide on carbohydrates in the presence of iron, that there was no evidence that the oxidation products of glucose, were pentoses. As this conflicted with the author's experience he describes his experiments on the oxidation of gluconic acid. Starting from the idea that hexoses are transformed into pentoses in the living cells of plants he subjected the calcium salt of gluconic acid, the first oxidation product of glucose, to a gentle process of oxidation by means of basic ferric acetate in sunlight. After four days, during which carbon dioxide was evolved, a dark yellow liquid was obtained which reduced Fehling's solution and ammoniacal silver solution, and which contained a small amount of formic acid. This liquid yielded an osazone from which a fraction was obtained, which melted at 162°—163° C., and which had a composition corresponding to the formula of the pentosazone, $C_5H_8O_3(N.NHC_6H_5)_2$.

If calcium gluconate be oxidised with bromine and lead carbonate, or with hydrogen peroxide and basic ferric acetate, solutions are obtained, which, on treatment with hydroxylamine, yield *d*-arabinose oxime, $C_5H_{11}O_5N$, which melts at 138°—139°. It is multi-rotatory, its normal specific rotatory power being $[\alpha]_D = -13·2$ in 8 per cent. aqueous solution. A specimen of *l*-arabinose oxime was found to agree with the oxime just described, except that it was dextro-rotatory, its normal specific rotatory power being $[\alpha]_D = +13·31°$.—C. A. M.

## *PRIZES.*

*Matches without Yellow Phosphorus, to Strike on any Surface; Competition for the Invention of a Paste for.* [Full Details.] Bull. Assoc. Belge des Chim. **12**, [2], 79—80. (This Journal, 1898, 702.)

THE Ministry of Industry and Labour (Belgium) has thrown open to general competition a prize of 50,000 francs, for the best paste for matches, destitute of white phosphorus, to strike on any surface. The conditions are as follow:—

1. *The Paste* must be able to withstand shock and friction sufficiently well to preclude the risk of any dangerous explosion during the course of manufacture; and it must, in addition, be free from substances capable of injuriously affecting the workpeople employed.

2. *The Matches* must strike on friction surfaces of various kinds, including cloth. They must also be able to bear exposure to alternations of moisture and dryness, heat and cold, within average limits, and must preserve their inflammability without, on the other hand, being liable to spontaneous decomposition; *i.e.*, must be chemically stable.

They must not in igniting give rise to any sparks, or the like, capable of burning the person using them, or of causing danger of fire, neither must they disengage poisonous fumes. Preference will be given to matches made of paste free from poisonous ingredients.

Moreover, the matches must be able to withstand a certain amount of friction, so as to preclude ignition in the pocket of the user, and in order that they may be transported and stored without danger, in the packages usual in commerce.

Inventors may compete up to 1st January 1899, under the following conditions:—

1. A sealed envelope to be deposited containing the name, baptismal name, and address of the competitor, together with his adopted device or mark, corresponding to that inscribed on the cases containing the samples of paste and matches. Of these samples 250 grms. of paste in the moist state must be submitted, enclosed in a stoppered bottle, encased in a recipient of sheet- or tin-plate, and packed in a wooden case with walls at least 2 cm. in thickness, a free space—to be entirely filled with some elastic substance, such as hay, straw, wood-wool, &c.—of at least 3 cm. intervening in every direction between the bottle and the iron recipient.

Furthermore, a sample of at least 10,000 matches must be deposited, packed in boxes, cardboard or paper; each package to contain not more than 500 matches and to be marked with the device selected by the inventor. The boxes must be enclosed in a tin-plate case hermetically sealed on all sides to exclude moisture, the whole being placed in a wooden box with walls at least 1 cm. thick.

All communications and packages to be sent to Mons. Woeste, President of the Commission, at the "Ministère de l'Industrie et du Travail," 2, Rue Latérale, Brussels, any time up to 1st January 1899.

Inventors whose matches fulfil the conditions laid down must afford proof that the manufacturing process is feasible on an industrial scale, by making—at a place to be mutually determined, on Belgian territory,—at least a million matches either before the Commission or a delegation of the latter. The Commission is empowered to extend, if necessary, the time limit fixed for the competition, and to ask inventors to provide additional explanatory information.—C. S.

### *The Disposal of Distillers' Refuse: A 2,000l. Premium.*

Chem. Trade T., Sept. 3, 1898, 150.

The question of river pollution has long been troubling the Scotch distillers, and 21 representatives of distilleries who guarantee the sum, met in Elgin last week, and formally agreed to offer a prize for a successful method of treatment. The following are the conditions under which it is to be given, and all competitors must sign them:—

The subscribers hereby offer a premium of 2,000*l.* to a person devising and handing over to them for their sole use and behoof such a scheme for treating the by-products of distilleries as will effectually purify them and be adopted by the Combination, and that on the following conditions, *viz.*:—

1. The money shall not be paid (*a*) unless and until the Combination are satisfied by practical tests, carried out by themselves or others authorised by them, that the scheme proposed satisfactorily treats all by-products of distilleries, and for this purpose competitors shall be bound to place their scheme, if required, at the disposal of the Combination to be experimented upon by them, with the assistance of the competitor, either on a large or small scale, and that for a period of one year from the time the scheme is placed at the disposal of the Combination, or such longer or shorter period as may be agreed upon; (*b*) unless and until the Combination are satisfied that it can be accomplished at a cost which, in the opinion of the Combination, is reasonable.

2. Should two or more equally effectual remedies be devised by two or more different persons, the Combination shall have the power of deciding to whom the premium shall be awarded.

3. The power of selecting the persons who shall compete shall be in the absolute discretion of the Combination.

4. Samples of the by-products will be placed free on rail to any person or persons the Combination may approve.

5. The premium shall not be paid unless and until the scheme is handed over to the Combination and vested in them as their absolute property.

6. All questions or differences arising between the Combination on the one hand and a competitor or competitors on the other hand, whether as to the meaning of these presents or any other question or difference, shall be settled by the combination, who appoint themselves as sole arbiters for this purpose.

Communications should be addressed to "The Distillers' Combination for the Purification of By-Products," D. Mustard, Secretary, 1, North Street, Elgin, N.B.

# Trade Report.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

(*From the Board of Trade Journal.*)

### SERVIA.

#### *Additional Octroi Duties.*

*Note.*—Kilogramme = 2·204 lb. avoirdupois.
Dinar = $9\frac{1}{2}d.$

Additional articles which a recent law of the Skuptshina provides shall henceforth be subject to Octroi duty on importation into Servia:—

Medicinal wines, such as quinine, pepsine, quinquina, laroche, meat, and other wines, per 100 kilos., 100 dinars.

### UNITED STATES.

#### *Customs Classifications.*

Phenacetin is dutiable under paragraph 67, Act of 24th July 1897, as a medicinal preparation in the preparation of which alcohol is used.

Indigo artificially prepared from coal-tar is dutiable at 30 per cent. *ad valorem* as a coal-tar colour or dye under paragraph 15, Act of 1897.

### DUTY ON CARBONATE OF BARYTA.

*Eng. and Mining J., Sept.* 10, 1898, 304.

In the matter of the protest of Gladding, McBean, and Co. against a decision of the collector of customs at San Francisco, the Board of General Appraisers decides as follows: "The merchandise is ground carbonate of baryta or witherite. It was assessed for duty as a manufactured article under section 6, Act of July 1897, and is claimed to be exempt from duty under paragraph 489. The Act of 1894, in paragraph 395, gave free admission to carbonate of baryta in its unmanufactured condition only; but the corresponding paragraph of the Act of 1897, paragraph 489, reads: 'Baryta, carbonate of, or witherite.' This provision contains no limitation as to whether the merchandise is ground or not. The protest is sustained accordingly, and the merchandise will be admitted free of duty."

## *EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.*

### SUGAR BOUNTIES IN FRANCE.

*Bd. of Trade J., Sept.* 1898, 328.

The Board of Trade have received through the Foreign Office, copy of a French Presidential decree, with reference to the granting of sugar bounties, as follows:—

Article 1.—The bounties, which from 1st September 1898 will be granted on home or French Colonial sugar, declared for exportation to other countries and French Colonies, not subject to the Metropolitan Customs Tariff, are fixed as follows:—

Raw sugar in powder or small crystals, yielding at least 98 per cent. in the case of beetroot sugar or 97 per cent. for colonial sugars, calculated before the deduction for waste in refining, per 100 kilos. of refined sugar, 2·77 francs. (Sugars of this category liable to duty and exported direct by the manufacturer himself, when they polarise at least 99·75 per cent., will be calculated according to their gross weight as refined sugars, without any deduction whatsoever.)

Raw sugar yielding from 65 per cent. to 98 per cent. in the case of beetroot sugar, or from 65 per cent. to 97 per cent. in the case of colonial sugars, per 100 kilos. of refined sugar, 2·42 francs.

Candied sugars, calculated at their legal coefficient, refined sugars in loaves or pieces, perfectly refined, hard, and dry, per 100 kilos. (net weight), 3·11 francs.

"Vergeoises," per 100 kilos. of refined sugar, 3·11 francs.

Refined sugar in powder or crystals, yielding at least 98 per cent., per 100 kilos. of refined sugar, 2·77 francs. (When the sugars of this last category polarise at least 99·75 per cent., they will be considered as pure refined sugars, and certificates of exportation admitted in the discharge of the obligations for temporary admission for their whole weight, without any deduction whatever.)

### DEVELOPMENT OF THE GERMAN CEMENT EXPORT TRADE.

*Bd. of Trade J., Sept.* 1898, 326.

H.M. Consul-General at Hamburg encloses a memorandum stating that the growing importance of the export trade of German cement from all parts of the German Empire is illustrated by the latest official statistics, which show that the aggregate exportations of this article, which already in 1894 had reached 7,110,701 cwt., amounted to 9,350,000 cwt. in the year 1897.

The three ports which do the largest export trade in German cement, or about 75 per cent. of the entire shipments from Germany, are Hamburg, Swinemünde (Stettin), and Bremen, *viz.*:—

*Exportations of German Cement.*

| From | 1896. | 1897. |
|---|---|---|
| | Cwt. | Cwt. |
| Hamburg | 3,753,912 | 4,871,919 |
| Swinemünde | 1,920,940 | 1,644,460 |
| Bremen | 815,265 | 980,090 |
| All other parts of Germany | 2,659,883 | 2,050,531 |
| Total | 9,150,000 | 9,350,000 |

By far the most important German port of exportation for cement is Hamburg. In 1897 the shipments from Hamburg amounted to more than 50 per cent. of the aggregate exportations from the rest of Germany. Though there are four or five cement factories in the vicinity of Hamburg (employing together about 3,000 workmen) which ship a certain proportion of their annual production to foreign countries, the larger share of the cement exported is stated to be derived from inland German factories.

The prominent customer for German cement is the United States of America; and next to that country Brazil, Australia, and Japan.

*Exportations of German Cement from Hamburg.*

| Countries to which exported. | 1896. | | 1897. | |
|---|---|---|---|---|
| | Quantity. | Value. | Quantity. | Value. |
| | Cwt. | £ | Cwt. | £ |
| United States | 1,852,706 | 183,154 | 2,178,587 | 196,739 |
| Brazil | 586,975 | 55,637 | 480,529 | 44,538 |
| Australia | 382,169 | 31,837 | 523,004 | 42,434 |
| Japan | 39,837 | 4,149 | 405,173 | 34,217 |
| Chile | 90,225 | 9,054 | 161,643 | 15,281 |
| Cape Colony | 69,168 | 6,107 | 173,367 | 15,172 |
| China | 61,419 | 5,509 | 117,619 | 10,819 |
| Venezuela | 89,158 | 8,184 | 98,801 | 9,505 |
| East Africa | 63,037 | 5,955 | 98,459 | 8,826 |
| West Africa | 30,886 | 2,966 | 60,452 | 5,890 |
| Dutch East Indies | 46,657 | 4,928 | 49,582 | 4,692 |
| Porto Rico | 36,111 | 3,172 | 41,058 | 3,752 |
| Mexico | 24,830 | 2,238 | 41,259 | 4,163 |
| Russia | 53,286 | 4,814 | 107,418 | 9,324 |
| Norway | 86,041 | 8,813 | 102,125 | 10,516 |
| All other countries | 240,707 | 23,965 | 229,843 | 22,098 |
| Total | 3,753,912 | 360,482 | 4,871,919 | 437,966 |

## CHEMICAL TRADE OF STETTIN.

*Foreign Office Annual Series, No.* 2177, *Aug.* 1898.

The Stettin Portland Cement Factory report that they had a good sale for their increasing produce during 1897. It appears that the demand for cement throughout the whole of the German Empire increased to such an extent that at times the efforts of the German factories could not keep pace with the demand. This Company sold off all the cement produced in 1897, and hold considerable orders for 1898. The Company declared a dividend of 20 per cent.

The Portland Cement Factory, "Stern," report that even at increased prices the demand continued so lively that they have no stock on hand left from 1897, and state that the demand from home being so great they had to limit their export abroad as much as possible.

They employed 585 workmen and paid 471,172 marks (23,210*l.* 9*s.*) in wages, that is to say, for each labourer per day an average of 2 marks 69 pf. (2*s.* 8*d.*).

The Stettin-Bredow Portland Cement Factory report that the results in cement as well as in brick-making during 1897 have been very satisfactory; they paid a dividend of 10 per cent., and paid in wages the sum of 252,488 marks (12,137*l.* 17*s.*) for 90,915 working days.

The oil mill owned by A. H. Zander worked 12,200 tons of oil-seed, and produced 4,456 tons of rape-seed oil and 7,320 tons of oil-cake. Wages were paid amounting to 83,030 marks (3,942*l.* 8*s.*).

The oil mill owned by P. J. Stahlberg worked 6,300 tons of rape-seed and colza, and produced 2,210 tons of rape-seed oil with 3,850 tons of oil-cake.

The Pommersche Provincial Sugar Refinery worked the following amounts of raw sugar:—1897, 77,500 tons; 1896, 73,700 tons; 1895, 66,000 tons; 1894, 59,600 tons; and produced in 1897, 75,300 tons, value 32,000,000 marks (1,576,351*l.*); in 1896, 73,250 tons, value 31,800,000 marks (1,566,502*l.*). The company employed 734 persons (492 males and 242 females), and paid in wages 670,000 marks (33,004*l.* 19*s.*). The dividend amounted to 10 per cent.

The import of phosphates to Stettin by sea amounted to 81,646 tons, and of pyrites, 68,618 tons.

The "Union" Chemical Produce Factory report that in consequence of lower prices the demand for artificial manure has considerably increased during 1897. Thus, their sales rose from 5,410,375 marks (266,521*l.*) in 1895–96 to 5,897,902 marks (290,537*l.*) in 1896–97. They sold 68,191 tons of superphosphates, 35,842 tons of Thomas slag; whilst 35,102 tons of sulphuric acid, 5,434 tons of potash, and 1,313 tons of bone manure were produced. The firm employed 588 hands (male and female), with average wages of 1,081 marks (53*l.* 5*s.*) each per annum.

## PEAT BRIQUETTE FACTORY AT LANGENBERG, PROVINCE POMERANIA.

*Foreign Office Annual Series, No.* 2177, *Aug.* 1898.

A briquette factory at Langenberg has only been in existence for two years, but has proved so far a complete success. Proprietors of turf moors in Scotland and Ireland might start factories of a similar nature with profit. Mr. Peters, the owner and manager of this factory, has taken a patent in England for his machinery; it could therefore be employed either by purchase or by royalty.

The turf at Langenberg is brought by water in an undried condition, and is thrown into the first breaker-machine, somewhat in effect like a large turnip-cutter; in this it is broken into small lumps. From the first it passes to the second breaker, and is ground into a fine powder. From here it goes into the drier, a steam cylinder which is filled with the exhaust steam from the engine, and is perforated by tubes, much in the manner of boiler-tubes, but larger. This cylinder revolves, and, being on a gentle slope, the powder passes slowly through the tubes, and by this means becomes thoroughly dry. From the drier it passes to a hopper which feeds the plunger. This plunger receives the power of a 75 horse-power engine, and by pressing the powder in a form against the preceding briquette, pushes them forward each stroke the width of a briquette.

The factory turns out 80 briquettes a minute, or 35 tons per day, with an average output of about 12,775 tons per annum, and the demand is far greater than the supply. The reason for this is that the briquettes are so marvellously cheap, an average price being 6½*d.* per 130 briquettes. In a closed oven, one briquette will remain in a glowing state for 24 hours; in an open grate it burns quicker, but remains for a longer time alight than any coal, giving a good red heat. The cost of working is comparatively light, as but few men are required to attend to the machinery. These men are paid on an average 3 marks (2*s.* 11½*d.*) per day, the engineer getting more. Women are employed to stack the briquettes in sheds. The cost at Langenberg of material and working 1 centner (112 lb.) is 35 pf. (4¼*d.*), and per ton about 7*s.* With a more extended plant, Mr. Peters is of the opinion that this could be reduced to 5*s.* per ton.

## REGULATIONS RESPECTING THE MANUFACTURE OF GLUCOSE, &C. IN BELGIUM.

*Board of Trade J., Sept.* 1898, 325.

Her Majesty's Minister at Brussels sends copies of Royal Decrees bringing into force on and from the 15th August last, the law of the 19th May last, respecting the manufacture of glucose, of syrups and of "invert" sugar.

These decrees define the various points which, in that law, were left to the subsequent decision of the Government.

Copies of the above may be seen at the Commercial Department of the Board of Trade, 7, Whitehall Gardens, S.W., any day between the hours of 11 a.m. and 5 p.m.

### New Coalfields in Bulgaria.

*Board of Trade J., Sept.* 1898, 330.

Her Majesty's Agent and Consul-General at Sofia states that coal has recently been found in the Balkans near Trevna, south of Tirnovo, which is said to be equal in quality to the best Welsh coal.

Several preliminary concessions for exploring the coalfield have been granted, but no concession for actually working it has yet been given.

The district can easily be brought into railway communication with the line now in course of construction between Rustchuk and Tirnovo.

Samples of the coal, and of the coke from it, were forwarded with the above despatch, and have been sent by the Foreign Office to the Cardiff Chamber of Commerce.

### Sulphur and Manganese at Hakodate, Japan.

*Foreign Office Annual Series, No.* 2176, *Aug.* 1898.

The exportation of sulphur touched its highest point in the years 1887–91, when the average annual export amounted to about 17,000 tons. Since that time shipments have gradually decreased, until last year it amounted to only 6,723 tons.

Owing to the excessively high prices asked by holders, stocks here have accumulated to the extent of 6,500 tons during the past two years. This stock the owners have now been shipping on their own account. Inclusive of this accumulation it is estimated that this year's shipments will probably be about 8,000 or 9,000 tons, after which the export is expected to drop to a maximum of 2,000 or 3,000 tons per annum.

With one trifling exception of no value, so far as export business is concerned, all the sulphur mines on this island and the northern mainland of Japan are surface volcanic deposits. The principal mines from which sulphur has been obtained are: Osoresan, in Rikuoku, Nambu, north mainland; Atosanobori, in Kushiro, Hokkaido; Sharesan, in Kitami, Hokkaido.

The two last mines have been exhausted, and from the first-named only 200 or 300 tons per annum are now extracted.

The only mine of any importance now being worked in this district is that of Ponto, situated in Kunashiri, one of the Kurile Islands, on the north-east coast of Hokkaido, from which about 2,000 or 3,000 tons are produced annually.

A few other small surface deposits, yielding from 50 to 500 tons per annum, are being worked in this island and the Kuriles, but they are unimportant, and no information is to hand as to the discovery of any further deposits.

On the other hand, the Japanese home demand for sulphuric acid works and other objects is estimated at 2,000 to 3,000 tons per annum. Although reliable statistics on this point are not available, the demand is certainly increasing year by year in the country, and it would seem that unless further and very extensive deposits are discovered, the foreign exportation of this article from Japan will soon virtually cease to exist.

The exports of sulphur during the year 1897 from this port to various countries were as follows:—

| Destination. | Quantity. | Value. |
|---|---|---|
| | Tons. | £ |
| United States | 4.523 | 16,470 |
| Canada | 1,000 | 3,702 |
| Hawaii | 737 | 2,609 |
| China | 463 | 1,605 |
| Total | 6,723 | 24,386 |

Mining operations of manganese in this island began in 1894, and that year's output is given as 1,300 tons; the following year's, 5,700 tons; 1896, 7,500 tons; and last year's, 5,200 tons; or 19,700 tons for the four years.

The mines are situated at Setanai. Aza Toshibetsu, at the head of Volcano Bay, and the deposits so far appear to be fairly extensive, whilst the ores, both superoxide and metallic, are of high grade. The following are a few of the latest analyses:—

| Peroxide. | Metallic. | Peroxide. | Metallic. |
|---|---|---|---|
| Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| 79·431 | 51·506 | 90·277 | 57·886 |
| 80·022 | 52·810 | 93·062 | 59·436 |
| 83·525 | 54·440 | 93·746 | 59·233 |
| 87·300 | 58·693 | | |

| Metallic. | Phosphorus. | Metallic. | Phosphorus. |
|---|---|---|---|
| Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| 51·091 | 0·117 | 57·094 | 0·131 |
| 55·042 | 0·083 | 57·169 | 0·169 |
| 56·474 | 0·156 | 58 357 | 0·096 |
| 57·135 | 0·109 | | |

The metallic ores show much higher grades of metal, it is stated, than those derived from the principal sources of supply in Europe, South America, and India, the exception being that in some of them the percentage of phosphorus rises over the standard of 0·10 per cent. Should much larger deposits of metallic ores be discovered and worked, those which might be objectionable on that score could doubtless be eliminated.

The ores from this island, as well as those from the northern mainland, are shipped to Yokohama, from which port they are forwarded to Europe and America. The principal mines, however, in the northern mainland do not appear to be as productive as formerly.

### Neapolitan Asphalt.

*Foreign Office Annual Series, No.* 2173, 1898.

Mount Laviano is one of the principal peaks of the Apennines in the province of Salerno. It rises to a height of some 5,500 feet above the sea. It is of limestone, in many parts saturated by asphaltic bitumen, and in some places above the calcareous strata sandstones full of asphalt occur.

The Neapolitan Mining Company secured perpetual mining rights on July 21, 1874, and since then have produced large quantities of asphalt. The railway is only 18 miles off. The quality of the product is of the first class, and capable of being used for any of the numerous purposes for which asphalt is applicable.

The company is not sufficiently strong to carry on work on a scale large enough to develop the resources of the mine, and desires to sell its concession and plant. The matter seems worth looking into by those interested in the trade, and the more so as the presence of petroleum has been ascertained, though no estimate of its quality appears to have been made.

### Neapolitan Sulphur.

*Foreign Office Annual Series, No.* 2173, 1898.

The owners of the mines of Oliveto Citra, in the province of Salerno, invite foreign capital to extend their undertaking; and while mining investments should not be entered upon without the necessary caution, the large amount of sulphur used here in agriculture forms in itself a considerable local market for the company's output.

The mines are the property of the Societa Mineralogica Napoletana, and are situated near the village of Oliveto, about 10 kiloms. (6 miles) from the railway which leads from Eboli to Naples, a line which touches the important ports of Salerno, Torre Annunziata, and Naples.

The mine is rectangular, and lies between the rivers Sele and Piceglio. The ground explored is about 4,000 by

500 metres in extent, and forms part of a large extinct volcano; and, in fact, when trenches are sunk, sulphuretted steam is found to rise. The deep tubular vents existing on the surface are covered with sublimate of sulphur, and some of them, although they have cooled, develop "sulphuric acid" gas in large quantities mixed with carbonic acid gas and sulphuretted hydrogen. Close beneath the cultivated ground burnt volcanic matter is found mixed with sulphur, and the deeper the trench the more sulphur in proportion is laid bare.

Twelve experimental trenches of 50 or 60 metres in length, 12 to 15 metres in breadth, and 5 metres deep have been sunk in sundry parts of this vast superficies of 2,000,000 metres.

By means of these, many banks of sulphurous deposit have come to light, giving an average of 20 per cent. sulphur.

### Cement at Odessa.

*Foreign Office Annual Series, No.* 2171, 1898.

Cement, formerly supplied exclusively by England, is now extensively produced locally by the several large works at Odessa and Novorossisk, which have come into existence within recent years. The demand for this product is considerable, but the home supply is nearly sufficient for local needs, while large shipments have been made from Odessa to the Far East.

### Nitrate of Soda at Odessa.

*Foreign Office Annual Series, No.* 2171, 1898.

The benefits of Chilian nitrate have, during the past couple of years, been receiving the attention of farmers, fruit-growers, viticulturists, and sugar-beetroot growers throughout Russia, articles appearing upon the subject in the newspapers both in North and South Russia, while an active propaganda, by means of circulars and pamphlets, is spreading throughout the country. The valuable assistance of the rural communes and other official bodies has been enlisted in carrying out trials of this nitrate upon grain, sugar-beets, grape vines, &c. Orders in this rapidly growing trade are going almost exclusively to Germany, where Russians find they can buy on three to six months' credit as cheaply as they can from England upon cash against bills of lading. It is to be feared that unless British firms are willing to waive the point, this trade will be carried on exclusively through Hamburg, whereas upon equal terms the Russians prefer to do business with English firms.

### Soap, Soda, and Candles in the Crimea.

*Foreign Office Annual Series, No.* 2171, 1898.

Soap and candles could be profitably manufactured here. The toilette soaps mostly in use come from France, Moscow, and Warsaw, and obtain a high price. The candles come from Moscow at a heavy railway charge. A manufactory of soda, both caustic and crystallised, should be a paying affair. These manufactories are very few and far between in Russia, and the products are dear. An enquiry addressed lately to a firm of Moscow merchants, representing a manufactory in the interior, elicited a reply naming something very high as the price of the sodas, but intimating that no orders could be booked for six months ahead.

### Products of Kwangsi (China).

*Foreign Office Annual Series, No.* 2168, 1898.

The dye plants of Kwangsi are (1) indigo, probably from *Indigofera tinctoria, L.*, and (2) the dye-yarn (Shu-liang), the so-called "false-gambier," the tuberous root of *Dioscorea rhipogonoides, Oliver.* Both are very widely grown. The former is exported in a liquid state in wooden tubs, mostly by junk, to the Canton province; but shipments have been made to Hong-Kong, and a growing trade is expected. Dye-yarns are also largely exported, and they are used in this and the Canton province for tanning and for dyeing native and foreign cottons, grass-cloth, and silk that lustrous brown to dark brown and black waterproof-looking colour so much affected by the Southern Chinese in summer. The natural dye is brown, and if a darker shade is required, alum or nutgalls are added, and in Canton the juice of green or unripe persimmons is frequently applied as a varnish to the outside of the cloth. That it possesses a waterproofing quality may be gathered from the fact that perspiration does not show upon it, and that to remove dirt only superficial washing is necessary, as the cloth does not absorb the water.

Two kinds of grasses are grown in Kwangsi and utilised for perfumery purposes: (1) Lemon grass (*Hsiang mao-ts'ao—Andropogon Schœnanthus, L.*), from which a sweet-scented oil is distilled, and (2) Hsiang-ts'ao (*Lysimachia fœnum græcum, Howe*), which is mostly used, dried, for steeping in and scenting tea-oil, wherewith women in the south of China dress their hair before adding bandoline. The former is exported for distillation.

Moxa is frequently described as crude camphor, but although it is a greyish-white powder, smelling strongly of camphor, it is not derived from *cinnamomum camphora, Fr. Nees*, which is fairly abundant in this province. It is distilled from *Blumea balsamifera, D.C.*, and the centres of export are Nan-ning and Po-sê. It is a valuable product costing as much as 200 taels a picul (133⅓ lb.), and is used in medicine, perfumery, and in Hong-Kong it is stated to be converted into camphor oil.

Nutgalls are derived from *Rhus semialata, Murr.*, and are used in tanning and dyeing. The centres of export are Nan-ning and Lin-chou. They are exported for use in foreign countries, and there would be a considerable saving in freight if they were crushed by machinery before shipment. At least the crushed material would occupy probably less than half the space required by the entire hollow galls.

The two essential and most valuable oils of Kwangsi are aniseed and cassia; but there are other oils produced in the province which are of no mean importance. They are castor (*Ricinus communis, L.*), ground-nut (*Arachis hypogæa, L.*), rape (*Brassica Chinensis, L.*), sesamum (*indicum, L.*), tea (*Camellia sasanqua, Thunb.*), and wood oil (*Aleurites cordata, M. Arg.*). Of these, tea and wood oil are the most important from a commercial point of view, the former being used principally as a hair oil, the latter universally in China, where painting, varnishing, and waterproofing have to be done. A coating of the latter is always applied to junks and native craft generally. Wood oil is now being carried direct from Wuchow to Hong-Kong for distribution in the southern provinces of China, and the West River bids fair to compete with the Yangtze for this trade. Numbers of transit passes have been taken out for this oil, and they point to P'ing-lo Fu, between Wuchow and Kuei-lin, the capital, and Li-chou Fu, farther west, as the chief centres of production and export. It is also brought down from Nan-ning and Po-sê, so that the *Aleurites cordata* is well distributed throughout the province.

The poppy is grown in Kwangsi, but not to an extent to admit of the export of opium. The drug is consumed in the province and is supplemented by opium of superior quality from Yunnan and Ssŭ-ch'uan. Yunnan, Ssŭ-ch'uan, and Kueichow opium is brought here annually to the amount of about 17,000 piculs for export to the Canton province.

The materials for the manufacture of paper in Kwangsi are very plentiful, and there is a considerable export of paper from the province. There are two kinds: (1) Sha-chih or "gauze paper," made from the inner fibrous bark of the paper mulberry (*Broussonetia papyrifera, Vent.*), which may be seen on every hand. This is the P'i-chih (bark paper) of Kuei-chow. There are several qualities manufactured, but even the best is poor compared with the product of the same material in Japan. (2) Ts'ao-chih, or Chu-chih—straw or bamboo paper—from the rotted fibres of bamboo and rice straw, is manufactured in the Wuchow prefecture in the districts of Ts'ên-chi and Jung. This is the ordinary coarse wrapping-paper of China, and its manufacture has been described elsewhere.

Copper exists in the department of Yulin in the south-east, and at Hsiang-wu-chow in the Ssu-ch'êng prefecture in the west of the province.

Tin is found in the Ho and Fu-ch'uan districts of the P'ing-lo prefecture.

Sulphide of antimony is abundant in the Nan-ning, Ssu-ch'êng, Chên-an, and T'ai-p'ing prefectures in the west and south-west of the province, particularly in the district of Hsi-lin near the Yünnan border, where there are whole mountains full of the ore. A small shipment of this ore has recently been sent to Hong-Kong.

### BOSNIAN INDUSTRIES.

*Foreign Office Annual Series, No.* 2167, 1898.

The various industrial establishments in this country have done fairly well during the past 12 months.

The ammonia-soda factory at Lukavica has declared a dividend at the rate of 11 per cent.

The petroleum refinery at Brod has not yet published the year's accounts, but will show a small profit.

The brewery of the "Actien Gesellschaft" at Serajevo brews excellent ale, and continues to be a most lucrative undertaking.

A discovery of asbestos has been made at Halilovac in the north-west of Bosnia.

### CALCIUM CARBIDE AND ACETYLENE IN FRANCE.

*Eng. and Mining J., Sept.* 17, 1898, 333.

In regard to the production of acetylene gas from calcium carbide in France, Consul Thackara, writing from Havre, August 4th, 1898, says that there are no official statistics bearing upon the annual output of calcium carbide, so that the amount furnished yearly by the different manufacturers has to be estimated. These estimates vary from 1,000 to 5,000 tons per year from each factory. There are 10 factories at present engaged in the manufacture of carbide of calcium in France. Two are at La Bathie, and one each at Séchilienne, Froges, Chapareillan, Lancey, Notre Dame de Briançon, St. Béron, Bellegarde, and Crampagna. Four factories are under construction. They will be situated at St. Etienne de Maurienne, Epierre, Serres, and Chute du Giffre. They will be able to produce, when running at their full capacity, from 2,500 to 3,000 tons per year. The wholesale price of carbide of calcium in France is from 350 to 400 frs. (67·55 dols. to 77·20 dols.) per ton, exclusive of cost of packing. The cost of packing is 4·50 frs. (87 c.) per iron drum containing 50 kilos. (110 lb.), 6 frs. (1·19 dols.) per drum of 100 kilos. The carbide of calcium manufactured in France is guaranteed to give 300 cubic litres of gas per kilogramme. The output is constantly increasing, and the supply is fully equal to the demand. The retail price of the article is from 55 to 60 frs. per 100 kilos. (10·21 dols. to 11·58 dols.), not including packing. There are two villages in France completely lighted by acetylene, by the Société Franco-Espagnole du Gaz Acétylène, of No. 81, Rue St Lazare, Paris. They are Alzonne, in the department of Aude, a town of 1,506 inhabitants, and Saurat-par-Tarascon, in the department of Ariege, a place of 3,024 inhabitants.

### BEETROOT-SUGAR INDUSTRY IN RUSSIA SINCE ITS FIRST INTRODUCTION IN 1800.

*Foreign Office Miscellaneous Series, July* 1898, *No.* 471, 1—22.

The first portion of this report is concerned with the history and development of the Russian sugar industry. The statistics bearing upon the home consumption of sugar give an average yearly increase of about 12,903 tons, which works out at $\frac{1}{4}$ lb. per annum, assuming the population to be 128,000,000. Stress is laid on the point that when the consumption of sugar in Russia comes within measurable distance of what it is per head of population in other countries, allowing that 80 lb. per head in Great Britain is a high percentage, instead of 235 factories being sufficient to meet the requirements in Russia, there would be something like 1,000 factories to give a production of 3,709,677 tons. It is thus seen that there is a great future for this industry, and it would be more inducive to a speedy development if the trade were allowed to work out its own salvation, instead of being weakened by artificial means of regulating production or prices. The following table shews the consumption per head, and the quantity of beetroot sugar produced, in other countries for 1897:—

| Country. | Consumption per Head, in Lb. | Estimated Population. | Quantity Produced, in Tons. | Quantity consumed by Population, in Tons. |
|---|---|---|---|---|
| Great Britain... | 86·09 | 39,000,000 | .. | .. |
| America ........ | 62·60 | 70,000,000 | .. | .. |
| Switzerland..... | 44·66 | 2,895,000 | 102,000 | 57,719 |
| Denmark ....... | 45·41 | 2,300,000 | .. | .. |
| Netherlands .... | 31·30 | 4,732,000 | 156,800 | 66,122 |
| France ......... | 30·61 | 38,800,000 | 703,300 | 532,942 |
| Germany ....... | 26·78 | 51,650,000 | 1,821,000 | 621,678 |
| Belgium ........ | 22·50 | 6,325,000 | 280,000 | 63,532 |
| Austria ......... | 19·81 | 43,456,000 | 929,900 | 389,125 |
| | | | Austria-Hungary. | |
| Russia .......... | 8·25 | 128,000,000 | 644,900 | 483,872 |

The most remarkable fact these figures show is that countries granting export bounties or artificially regulating the production and prices, are those who consume less sugar per head. If the bounties given at present by France, Austria, and Germany can be abolished, the step can only be to the ultimate good of the sugar industries of these countries. The abolition of bounties would greatly benefit the sugar industry of Russia, in so far that the price upon the world's market, London, would naturally rise equivalently to the bounty formerly granted, and therefore the manufacturers in Russia would meet their German, Austrian, and French competitors upon even terms.

The world's production of sugar from the four campaigns 1893–94 to 1896–97 was as follows:—

| —— | 1896–97. | 1895–96. | 1894–95. | 1893–94. |
|---|---|---|---|---|
| | Tons. | Tons. | Tons. | Tons. |
| Beetroot sugar: | | | | |
| Germany ............ | 1,800,000 | 1,615,111 | 1,844,586 | 1,381,603 |
| Austria-Hungary ..... | 1,050,000 | 791,405 | 1,055,821 | 841,809 |
| France.............. | 780,000 | 667,583 | 792,511 | 579,111 |
| Russia.............. | 750,000 | 783,489 | 615,058 | 658,070 |
| Belgium ............ | 265,000 | 260,000 | 243,957 | 240,317 |
| Netherlands ......... | 145,000 | 106,829 | 84,597 | 75,015 |
| Other countries....... | 170,000 | 169,120 | 156,000 | 113,610 |
| Total ............ | 4,960,000 | 4,393,537 | 4,792,530 | 3,889,535 |
| Cane sugar............ | 2,747,500 | 2,945,811 | 3,570,413 | 3,552,374 |
| Grand total ...... | 7,707,500 | 7,339,348 | 8,362,943 | 7,441,909 |

During the campaign 1896–97, Germany, Belgium, Austria, Netherlands, France, Russia, had respectively 400, 111, 217, 30, 358, 235 factories engaged in the production of beetroot sugar.

There are no reliable particulars of the quantity of sugar imported into England from Russia, but it is known that in 1896 Russia exported 149,482 tons of sugar to Europe, most of which found its way to London.

There can be no doubt that a great future is open for the sugar industry of Russia, and there is bound to be an immense increase in quantity consumed by her own population. This increase can only take place when the system of taxing the home consumer, and making him pay about three times more for what the same sugar is sold to foreign nations, has been abandoned as being just as detrimental to the interests of the manufacturer as it is to the consumer. Exportation to Europe will also increase, and when the Siberian Railway is opened Russian-made sugar will probably supply the wants of China and Japan.

—J. L. B.

### ERRATUM.

*Minerals and Caoutchouc in New Caledonia.*

(*This Journal, Aug.* 1898, 811.)

In the table giving the quantity of minerals and oils exported in 1897, the last comma in each set of figures should be a decimal point, making, for instance, the total nickel 33,771·302 tons instead of 33,771,302 tons—an impossible amount.

The figures were correctly transcribed; but it is suggested that being derived from French statistics, in which commas are used instead of the decimal points with which we are familiar, the error has thus crept in.

## GENERAL TRADE NOTES.

### THE CARRIAGE OF DANGEROUS GOODS AT SEA

*Chem. Trade J., Aug.* 27, 1898, 130.

*Board of Trade Instructions.*

The Board of Trade have just issued further instructions to their surveyors relative to the carriage of dangerous substances on shipboard. With regard to the carriage of sulphuric acid under deck, the Board state that they have recently had under consideration the conditions under which strong sulphuric acid contained in electrically-welded steel drums—holding about half a ton each—can, with proper regard to safety, be carried on board ship and stowed under deck.

Two steel drums of the following dimensions were tested to destruction:—

| External Diameter. | Length Over All. | Thickness of Body. | Thickness of Ends. | Approximate Weight, Sulphuric and Acid. | Remarks. |
|---|---|---|---|---|---|
| Ft. Ins. | Ft. Ins. | In. | In. | Lb. | |
| 1 5 | 2 2¼ | 0·07 | 0·08 | 410 | Ends unstayed. |
| 2 1½ | 3 4½ | 0·09 | 0·087 | 1,120 | Ends unstayed; body strengthened near the centre part by two I-shaped rolling rings, 1½ ins. deep. |

The ends were flat, and flanged at the periphery, and electrically welded to the body. The results showed that they are strong enough to withstand rough usage when filled with sulphuric acid.

As regards stowage, the Board of Trade are advised that the acid can be carried with safety under decks if the drums are embedded in coal to a depth of not less than three-fourths the diameter of the drum, it being of course understood that the quantity of coal must be sufficient to absorb any leakage from the drums, and for this purpose a depth of one foot of coal for every hundredweight of acid contained in the largest drum in the shipment, but with a minimum depth of six feet, may be deemed sufficient.

In the event of leakage, the Board of Trade are advised that coal, even in a powdered state, will not evolve heat by reason of the presence of sulphuric acid, and that the marketable condition of only so much of the coal as is wetted by the sulphuric acid is affected.

In issuing this notice the Board of Trade think it desirable to indicate the possible results that may be anticipated as regards spontaneous combustion in a cargo of coal on the surface of which drums of sulphuric acid have been embedded.

1. If the sulphuric acid escapes into the coal, spontaneous combustion will not take place within the region of the leakage.

2. If the spontaneous combustion takes place first, then the rupture of the drums, and consequent escape of the sulphuric acid, will retard or extinguish the fire by the production of sulphurous acid vapour, in which combustion cannot be sustained. This gas has a pungent odour and a strongly suffocating effect, so that great care should be taken to keep away from it. Coal wetted with acid should not again be used.

The Board are aware that it is the practice at some ports to stow sulphuric acid in a compartment by itself on a layer of chalk, but the leakage of sulphuric acid on to the chalk would produce carbonic acid gas—a heavy odourless gas, which it is dangerous to inhale, and which gives no warning of its presence. For these reasons the ventilation of a space where this practice is followed should be carefully attended to before any person is allowed to enter the compartment, the down-cast being carried as low as possible.

It is recommended that wherever sulphuric acid is carried under deck, the precautions indicated above should be observed; but it is of course to be understood that drums of sulphuric acid should not be stowed on coal known to be specially liable to spontaneous combustion.

*Stowage of Dangerous Goods.*

The attention of the surveyors is drawn to the case of the steamer *Legislator*, of Liverpool, on board which vessel a serious fire recently occurred, resulting in the loss of the vessel with several lives, owing to the indiscriminate stowage together on deck of "dangerous goods" and chemicals.

With a view to the prevention of similar casualties in future, the surveyors are instructed, when on their rounds of inspection of vessels loading, to use all due diligence in keeping themselves informed as to what vessels are likely to ship "dangerous goods;" and when inspecting the stowage of such goods, to see that any one description of "dangerous goods" shall be shipped apart from all other goods, whether dangerous or not, except in the case of certain combinations which may be approved by the Board of Trade.

This precaution is so obvious in the interest of shipowners, that, if properly explained, it is not anticipated that any difficulty will be experienced; but if the surveyor be unable to get this instruction carried out, the case should be treated as one of "improper loading" within the meaning of the 459th section of the Merchant Shipping Act, 1894.

### AN AMERICAN CONSUL ON THE CHEMICAL TRADE.

*Chem. Trade J., Aug.* 27, 1898, 135.

Mr. James Boyle, the United States Consul at Liverpool, in making his report on the English chemical trade, refers to the new works which the United Alkali Company is now erecting for the manufacture of chemicals at Bay City, Michigan. The question of greatest interest in the trade, he reports, is the possibility of producing bleaching-powder and other chlorine products cheaper than they now can be produced by the Leblanc process. The electrolytic process has been used for some time in Germany, and, to a small extent, in the United States; but in England it is in its experimental stage. The heavy decrease in the export of chemicals from England to Germany and the United States is compelling English manufacturers to find some cheaper way of producing bleaching-powder and other chlorine products. The exports of chemicals from Liverpool to the United States have declined from over eight and a half million dollars in 1891 to less than four millions in 1897.

### DISCOVERY OF FULLERS' EARTH.

*Chem. and Druggist, Aug.* 20, 1898, 337.

A well-known prospector in San Bernardino county, California, claims to have found an immense bed, apparently inexhaustible, of fullers' earth. The new discovery is pronounced to be not only fully equal in quality to that imported from Great Britain, but is in some respects held to be superior. Fullers' earth is used very freely in the American packing-houses, and so pleased are the Cudahy Packing Company, of Los Angeles, with the samples received, that they have contracted to take 24 tons per month. It is reported that the Armour Packing Company use from 500 to 600 tons of fullers' earth per month, all of which is imported from Britain. According to the *San Bernardino Sun*, which announces the discovery, the United States import a million tons per annum.

### THE OCCURRENCE OF CHROMITE IN CANADA.

*Imp. Inst. J., Sept.* 1898, 258.

In the recent issue of *The Canadian Engineer* (June 1898, 31) an account is given of the existence of chromite in Canada, the facts being furnished by a report of Mr. J. Obalski, M.E., Provincial Inspector of Mines for Quebec.

Chromite is a hard, heavy mineral, and consists of chromium sesquioxide associated with ferrous oxide in the proportions indicated by the formula $Cr_2O_3 . FeO$. It forms the chief source of the chromium compounds, many of which are extensively used at present for industrial purposes. Recently, on account of its extreme refractoriness, low-grade chromite has been employed as a lining for furnaces exposed to high temperatures in many smelting operations.

The chief sources of chromite up to the present have been Asia Minor and the United States, where large deposits occur in Maryland, Pennsylvania, and California. More recently it has been found in New South Wales, New Caledonia, and Canada.

The existence of chromite in the serpentines of the eastern townships of Quebec has long been known, and in 1861 about 10 tons of the mineral were obtained in the neighbourhood of Lake Nicolet. Since that time it has been found in various localities, but many of the deposits did not appear rich enough to be worked with profit. In 1894, however, an important discovery of high-grade chromite was made near Black Lake, and this has been followed by other discoveries in the same neighbourhood, especially in the township of Coiraine. These deposits have proved remunerative to work, and the industry promises to become a prosperous one. The district is near the line of the Quebec Central Railway, the mineral is easily won, and labour and fuel are cheap. Up to the present the mining has been carried on by very primitive methods, and only by surface workings, but already more than 10,000 tons, valued at 140,000 dols., have been obtained. The ore furnishes a very large quantity of high grade chromite. Pure chromite contains 68 per cent. of chromium sesquioxide, but the percentage in picked specimens of ore rarely exceeds 56 or 57, and the shipments seldom reach 53. For the manufacture of potassium dichromate the grade used is not less than 48 per cent., below which the ore is classed as of inferior quality. The Black Lake ores have given 49·8 and 50·3 per cent. on shipments, 58 per cent. on picked specimens, and as much as 60·66 per cent. on concentrated specimens. Shipments of ore from the neighbourhood of Breeches Lake have yielded 55 per cent. of chromium sesquioxide.

There is also an important bed of chromite on the west coast of Newfoundland, at Bluff Head, Port-au-port Bay, which is now being worked. In the summer of 1896 about 15,000 tons were obtained, and this also was of good quality, yielding 50 per cent. of chromium sesquioxide.

### SCARCITY OF TIN.

*Scientific American.*

The fact that tin, of all the metals in common use, is only sparingly distributed throughout the world, is again called attention to by an Australian geologist, Mr. B. J. Stertchley, who has published a monograph upon the subject. While the known goldfields of the world cover more than 1,500,000 square miles, the tinfields have an area of less than 12,500 square miles. Thus, for every square mile of tin ground there are 132 square miles of gold-bearing country. There are seven tin districts in Europe, producing about 8,300 tons yearly, of which the Cornish mines yield about 8,000 tons. Asia has two tin areas—Hunan, in China, estimated by some of the best authorities to produce 10,000 to 20,000 tons a year, but proved by official figures to yield less than 2,500 tons; and the Straits Settlements, and adjacent principalities, yielding 58,000 tons yearly—the richest yield in the world. Africa has no known tin mine; North America no payable one; South America only one tin area, Bolivia and Peru, yielding less than 4,000 tons a year; and Australasia, the youngest, contributes about 6,000 tons a year.

### ALKALI-MAKING IN EGYPT.

*Chem. and Druggist, Sept.* 10, 1898, 445.

The deposits of natron at Wady-Natron, in Western Egypt, which have been for a long time unprofitably worked by the Egyptian Government, have now been made over to a private company. The situation of the deposits in the centre of a desert was much against the success of the venture, but the company are trying to overcome that by constructing a railway to the spot, and they have also built a factory with modern appliances, which will give them a better chance of placing soda on the Alexandrian market to compete with British alkali.

### SOAP-MAKING IN EGYPT

*Chem. and Druggist, Sept.* 10, 1898, 445.

This industry has made considerable progress in Egypt of late. Whilst formerly large quantities of soap were imported from Candia and Syria, the Alexandrian manufacture now covers 85 per cent. of the home consumption. The soap is made from the same materials as in Syria, *i.e.*, olive oil, talc, and caustic soda. The olive oil is brought from Syria and mixed in Alexandria with cotton oil and inferior Italian oil obtained from the olive kernels, and talc from Mitylene chiefly, partly also from Austria. There are in Alexandria four large and several small soap factories, mostly of French type. The soap is easily disposed of, the average price being 0·20 fl. per kilo.

### GENERAL NOTES.

*Färber-Zeit.* 1898, **13**, 204—209.

The accompanying table shows the rise, in Germany, of the number of patents, and other information, in the various industries, during the years named:—

| No. of Class. | Name of Material. | Patents applied for and completed. | | | | | | Percentage of Completed Returns. | | | | | Patents abandoned. 1877–1897. | Patents in Work. | Percentage. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1893. | 1894. | 1895. | 1896. | 1897. | 1877–1897. | 1893. | 1894. | 1895. | 1896. | 1897. | | | |
| 8 | Bleached goods, colours.... | 258 | 308 | 265 | 322 | 366 | 4,086 | 46·0 | 46·5 | 48·7 | 43·7 | 39·8 | 1,285 | 495 | 27·8 |
| 8 | Printing stuffs, finished goods. | 131 | 145 | 129 | 117 | 133 | 1,780 | | | | | | | | |
| 22 | Colouring matters, varnishes | 552 | 348 | 354 | 295 | 319 | 4,889 | 45·4 | 49·8 | 54·2 | 57·8 | 50·1 | 924 | 1,269 | 57·9 |
| 22 | Lakes ................ ... | 249 | 224 | 208 | 144 | 133 | 2,193 | | | | | | | | |

These statistics show, generally, that the prosperity of the colour industry is most marked in Germany.—C. M.

### INDIAN COAL.

*W. R. Dunstan. Imp. Inst. (Indian Sect.) Ann. Rep.* 1897–98, 40.

*Production.*—The output in 1896 amounted to 3,848,013 tons as compared with 3,537,820 tons in 1895, and indicates an improvement in the commercial and industrial prosperity of the country, since nearly the whole production is consumed as fuel for motive power. The average price of Bengal coal is Rs. 2 at the pit's mouth, and of picked coal Rs. 3 to 4 per ton.

*Imports and Exports.*—In 1895–96 the imports declined to 746,850 tons, the previous year's shipments having been 808,713 tons, of which more than 75 per cent. was landed at Bombay. England furnishes the major portion of the supply, but Australia and Japan are doing an increasing though relatively very small trade.

India appears to be relying more and more on her own coalfields, the increase in the imports during the past few years being far from commensurate with the enormous advance in the home output.

The export trade increased from 53,565 tons in 1895 to 79,989 tons in 1896, the bulk going to Ceylon to supply the demand for steam coal at Colombo, where Indian coal now replaces European.

Complaints are said to have been made that Bengal coal is too "dirty" for the Ceylon railways, but this difficulty should be easy to overcome, Bengal producing plenty of good locomotive coal.—C. S.

### The German Beet-Sugar Industry.

*The Times, Aug.* 18, 1898.

Official statistics of the production of beet-sugar in Germany in 1896–97 show that during that season 399 beet-sugar factories were in operation, against 397 in the previous season. The 399 factories used 13,721,601 tons of beetroot in 1896–97, compared with 11,672,816 tons in 1895–96, and 14,521,029 tons in 1894–95. The average price per ton of beetroot in 1896–97 was 17*s.* 8½*d.* (against 17*s.* 8½*d.* in 1895–96 and 1*l.* 0*s.* 2½*d.* in 1894–95), and varied between 2*l.* 0*s.* 10*d.* per ton (in Würtemberg) and 17*s.* 1¼*d.* (in Westphalia). The beetroot used was raised in 1896–97 on an area of 1,019,881 acres (against 920,749 acres in 1895–96), which gave an average yield of 13 tons per acre (against 12·67 tons in 1895–96). The consumption of foreign-grown beet was very restricted last year, and was greatly exceeded by the export of home-grown sugar-beet. According to the statistics, only 32,106 tons of fresh sugar-beet were imported, of which 25,920 tons came from Russia while the quantity exported amounted to 52,225 tons, of which 50,481 went to Austria. The production of raw sugar of all descriptions was 1,738,882 tons in 1897, compared with 1,537,522 tons in 1896, and 1,766,805 tons in 1895. The exports of raw sugar from Germany in 1896–97 reached 760,657 tons, against 504,441 tons in 1895–96, and were consequently over 50 per cent. higher last year than in the preceding season. The imports were insignificant—only 665 tons, against 765 tons. In 1887–88 the German exports of raw sugar amounted to 314,711 tons, and the imports to 4,079 tons. The exports to the United States increased by 307,906 tons; to Great Britain, by 14,943 tons. The United States took last year 487,729 tons; Great Britain, 237,786 tons; British North America, 13,922 tons; Holland, 11,821 tons. The home consumption decreased considerably, from 744,367 tons in 1895–96 to 561,882 tons in 1896–97, or by 24¼ per cent. This gives for last year 23·21 lb. per head of the population, against 31·15 lb. in 1895–96. The complete figures for the season of 1897–98 are not yet available; but it is stated that up to the end of May, according to preliminary statistics, 1,636,907 tons of raw sugar of all descriptions had been manufactured from 13,698,281 tons of beetroot. As there was a heavy beet crop in the season 1896–97, it is estimated that the production of raw sugar in the German Empire in 1897 will reach 1,875,000 tons.—J. L. B.

### Chemical Industry in California.

*Eng. and Mining J., Sept.* 3, 1898, 271.

A new and interesting development in the chemical industry of the United States is the utilisation of the sulphuretted hydrogen gas developed as a by-product in the refining of asphaltum at the California Asphaltum Company's works at Ventura, Cal. The gas is burned to sulphurous anhydride in the usual well-known manner, and the product of the combustion is conveyed to the lead chambers. Owing to the method of its production the gas is of a high degree of purity, and the acid produced is also of high grade. At present the output amounts to 10 tons per day, when the works are run at their full capacity.

The Pacific Coast is beginning to have an important chemical industry. Sulphuric acid is made on a rather large scale at one other plant, while the manufacture of bluestone and boric acid, carbonic acid, Epsom salts, magnesia, and numerous other products, is now carried on. The State of California has great natural resources upon which to base an important chemical industry, and its geographical position gives it an advantage in supplying certain markets of the Pacific Slope, so it is probable that a more important and interesting chemical industry will grow up there when the transportation problem is settled.

### Discoveries of Lithia Mica, and other Minerals in the United States.

*Eng. and Mining J., Sept.* 3, 1898, 277.

At the Boston meeting of the American Association for the Advancement of Science, held Aug. 22nd to 27th last, Mr. C. R. Orcutt, San Diego, presented a note, saying that near San Diego, Cal., there is an enormous vein of lepidolite (lithia mica) containing rubellite (pink tourmaline) in large amounts. The bed or vein is 60 ft. or more in width where best exposed, but its length was not stated. As a source of lithia and potash this deposit must soon take first rank commercially. It is now being worked as an open quarry, and 1,500,000 tons estimated to be available. The deposit was discovered in 1889, and some large masses from it were displayed at the Columbian Exposition in 1893. About 20 tons of museum and cabinet specimens have been scattered over the world. Tourmalines of gem quality have not been found until this year, and they are all of the red variety. Green and black tourmalines have also been found, however, and are not to be considered rare at the locality.

G. F. Wright, of Oberlin, Ohio, reported the discovery of a new deposit of celestine (strontium sulphate) at Put-in-Bay, Ohio. Celestine occurs at many places in Europe, but the principal locality in America from which cabinets have been supplied with specimens and the material has come in commercial quantities has been Strontian Island, near Put-in-Bay, Lake Erie. Just as this supply was becoming exhausted, a remarkable cavity was discovered last winter on Put-in-Bay Island, which is completely lined with very large crystals of the mineral. The cavity was broken into at 17 ft. below the surface while digging a well, and it is large enough to receive 10 or 12 people at a time. It is not an ordinary cavern, but apparently is the interior of an immense "geode" lined with celestine. The geological formation in which it occurs is the Water-lime of the Lower Helderberg group. Large deposits of gypsum (calcium sulphate) occur in the vicinity.

### Platinum from Algeria.

*Eng. and Mining J., Aug* 27, 1898, 244.

According to a French exchange, M. Gourdin lately brought from Algeria a new ore of iridium, platinum, &c. (found in the crusts and crystallised nodules of clay beds), analysis of which shows, osmiridium, 64·85; titanate of iron, 12·15; copper, 1·78; platinum, 9·64; and palladium, 0·89.

### Columbite in Ontario.

*Eng. and Mining J., Sept* 10, 1898, 306.

A mineral of the columbite group has been discovered by W. G. Miller in the township of Lyndoch, containing the rare acids niobic and tantalic acids, also the rare earths ceria, didymia, and the yttria group. Its chief economic interest, however, arises from the fact that it contains a small percentage of tin. Columbite and cassiterite are usually associated in the tin deposits of South Dakota, and there as well as in Lyndoch, beryl is found in close proximity.

### The Mineral Resources of Cuba.*

*R. Cabrera. Eng. and Mining J., Sept.* 10, 1898, 308.

The mineral resources of Cuba have always been subordinated to the agricultural, and the island has not been considered a mineral country. In fact the only deposits which have been worked on a commercial scale have been the iron ores. The development of such resources has been hindered by maladministration and excessive taxation, which have prevented the investment of capital in mining works.

The minerals which had been more or less constantly mined in Cuba, up to the outbreak of the present revolution in February 1895, were the following:—

1. Asphalt and mineral oils.
2. Copper.
3. Iron.
4. Manganese.
5. Gold.

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* Abstract of lecture before the Franklin Institute, Philadelphia.

1. The deposits of hydrocarbons, asphalt, and mineral oils, are abundant and of excellent quality, both for fuel and lighting purposes. They are present in various conditions, solid, plastic, and liquid, and several experts have affirmed "the existence at a great depth of very light oils, petroleum or naphtha." Of these, however, no definite investigations have been made, nor scarcely any examinations beyond such as are incidental to a very primitive stage of industry. On the Motembo estate, Province of Santa Clara, at a location named San Juan, borings were made in 1881, which resulted in discovering, at a depth of 95 metres, a deposit of naphtha of extraordinary purity, colourless, transparent as water, inflammable, leaving no residue after combustion, and of a density of 0·754, which congealed at a temperature of 85° C., dissolved asphalt and the resins, and which was so volatile that its vapour at ordinary temperature could be utilised as illuminating gas. Nevertheless this well was abandoned after the workings had reached a depth of 290 metres, although the supply continued at that depth.

A well similar to that of Motembo was discovered at Lagunillas, Province of Matanzas, from which, out of a source at a depth of 25 metres (82 ft.), there overflowed a supply of 70 litres (18½ gallons) per day. This also has never been further explored or worked.

In the same Province, at scarcely a kilometre from the northern coast, there exists a deposit of bituminous shale impregnated with asphalt of a high degree of purity and in great abundance, whose workings, though favoured by facilities of transportation, have also been abandoned. But even at the Bay of Cardenas, in the Province of Havana itself, there has been found a similar deposit; this latter appears, from contemporary molluscs and corals embedded in it, to be of comparatively recent geologic age.

In the rocks underlying a large extent of the bottom of this bay are great masses of asphalt in a liquid state, having in places a thickness of 70 ft., and quantities of this have been carried to market in New York, where it was sold at prices varying from 80 dols. to 120 dols. per ton. The exploitation of these deposits in the layers at shallow depths was begun with considerable success; but the enterprise languished for want of facilities, and eventually failed entirely through legislative interference inevitable in that country.

Asphalt is to be found extensively in the Provinces of Havana and Pinar del Rio. In the former there are being worked the mines of Potosi and Santa Rosa, distant half a league by rail from the harbour of Havana. The deposits exploited are embedded in the cretaceous marls and the serpentines which are met with in the northern districts, the strata running generally N.W. and S.E. in veins of about 6 metres. The material is earthy and impure, but is advantageously utilised as fuel in grates and ordinary stoves. The mineral is worked in a very crude manner, in the open air, and small quantities of the product have been exported annually. In Pinar del Rio are the Canas and Tomasitas mines, close to the bay of Mariel. These mines are notable for their size, the veins being 35 ft. wide, and from 45 to 90 ft. deep. They produce over 1,300 tons annually, destined for the use of near-by plantations, whose owners combine in working the deposits, the operations all being carried on in the open air. Asphalt is very abundant in the Vuelta Abajo, where it commonly occurs spread on the ground as it has been disengaged from the rocks. There are many smaller workings, among them that of Santa Helena, at Bahia Honda, which district contains unworked deposits of great brilliancy and purity.

2. Copper has been mined on a larger scale than any other of the mineral resources of Cuba, particularly near Cobre in the Province of Santiago de Cuba. These mines were discovered about the middle of the sixteenth century, but their working was reserved for itself by the Spanish Government, with the result simply of giving them over to various contractors. Subsequent to 1833, the Consolidado Company discovered some large lodes, and this stimulated competing enterprises, one of which undertook the development of a number of mines, under the ownership of an English syndicate, and named the Santiago Company.

The mines of Cobre, of which the machinery and outfittings were destroyed by the insurgents during the so-called Ten Years' War, and the workings wholly abandoned, were subsequently acquired by the Cobre Railroad Company, which constructed five iron bridges, equipped the workings with tramways, rolling stock, and locomotives, but the enterprise was afterwards practically abandoned, solely because of the obstacles created by the administration. The exploitation of the mines has dwindled to the working of the tailings of the early rich outputs.

Other Minerals.—Sixty kilometres distant from Santiago de Cuba, there is a group of 10 mineral sites spread over an area of some 4,000 acres, the property of Don Julio Aurich, the mineral from which contains 34 per cent. of iron, and 28 per cent. of manganese; these deposits are not worked. Limonite is found in various deposits in Baracoa, where it has been discovered in strata in the ridges bordering the seashore.

Chrome iron is also to be found in the district of Baracoa, where three mining claims have been taken up. The analysis of the mineral from the Valhourat mine, made in the Paris School of Mines, showed the presence of chrome oxide to the extent of nearly 58 per cent. Other mines of this mineral have been located at Mayari and at Sagua de Tanamo, in both of which localities it is very abundant. In Bayamo, in the precinct of Vavecito, there is a tract of 84 hectares (170 acres) of lead ore, and two other sites of this mineral in the same vicinity show veins of some 50 centimetres width. In the district of El Cobre, precinct of Sevilla, there is a deposit of argentiferous lead covering some 150 acres. Of this class of mineral numerous mining claims have been taken up, all of which remain unworked.

Of asbestos, two mining concessions have been granted at Mayari, both covering 120 hectares (300 acres). This mineral also appears in abundance at Holguin, in the Nuevo Potosi mine.

### French Cyanide.

*Chem. and Druggist, Sept.* 24, 1898, 535.

A recent letter of the French Minister of Commerce, addressed to the Paris Syndical Chamber of Chemical Products, draws attention to the important outlets for cyanide in gold-recovery. The sale of cyanide in the Transvaal and in the whole of South Africa exceeds (according to the Minister) 300 or 400 tons yearly, and it is anticipated that, within the next two or three years, the demand will exceed 10,000 tons in the Rand district alone. The cyanide used in mines must be 98 to 100 per cent. and very pale in colour. It is shipped in wooden cases lined with zinc, holding 100 kilos. each. Unfortunately, continues the Minister, the English and Germans have up to now had a monopoly of the stuff, and the French company which manufactures it cannot compete with the English manufacturers' prices. The latter employ an economical process, which obviates the use of ferrocyanide of potassium. The Minister urges scientific chemists to assist French manufacturers to fight against foreign competition.

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 31st August. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,572,652 | 1,754,453 |
| Chemicals and dyestuffs | 360,704 | 377,046 |
| Oils | 594,905 | 787,978 |
| Raw materials for non-textile industries. | 5,635,240 | 6,102,167 |
| Total value of all imports | 33,371,385 | 37,216,527 |

## SUMMARY OF EXPORTS.

| Articles. | Month ending 31st August. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) .... | 2,615,718 | 2,857,528 |
| Chemicals and medicines.......... | 599,190 | 612,241 |
| Miscellaneous articles.............. | 2,324,179 | 2,649,121 |
| Total value of all exports..... | 18,773,907 | 20,186,016 |

## IMPORTS OF METALS FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Copper:— | | | | | |
| Ore .............. | Tons | 13,053 | 11,471 | 58,556 | 41,421 |
| Regulus ........ | ,, | 5,631 | 5,151 | 150,782 | 137,915 |
| Unwrought ..... | ,, | 4,301 | 5,661 | 214,641 | 288,592 |
| Iron:— | | | | | |
| Pig and puddled bar. | ,, | 14,038 | 11,246 | 16,800 | 14,502 |
| Ore.............. | ,, | 491,411 | 507,297 | 379,908 | 382,891 |
| Bolt, bar, &c. ... | ,, | 6,409 | 7,365 | 53,301 | 55,330 |
| Steel, unwrought.. | ,, | 2,178 | 4,403 | 23,327 | 27,419 |
| Lead, pig and sheet | ,, | 11,306 | 13,881 | 136,826 | 179,228 |
| Pyrites ............ | ,, | 48,708 | 58,202 | 79,396 | 94,404 |
| Quicksilver ....... | Lb. | 34,000 | 63,555 | 3,284 | 6,413 |
| Silver ore....... | Value £ | .. | .. | 111,697 | 101,572 |
| Tin ............... | Cwt. | 24,953 | 25,488 | 74,964 | 88,415 |
| Zinc .............. | Tons | 5,256 | 5,617 | 89,305 | 103,722 |
| Other articles ... | Value £ | .. | .. | 149,865 | 199,629 |
| Total value of metals | | .. | .. | 1,572,652 | 1,754,453 |

## IMPORTS OF CHEMICALS AND DYESTUFFS FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Alkali.............. | Cwt. | 18,496 | 22,812 | 8,416 | 12,650 |
| Bark (tanners', &c.) | ,, | 37,797 | 35,989 | 12,512 | 16,614 |
| Borax ............ | ,, | .. | 80 | .. | 104 |
| Brimstone ........ | ,, | 47,668 | 48,152 | 11,353 | 10,470 |
| Chemicals...... | Value £ | .. | .. | 107,834 | 119,009 |
| Cochineal ......... | Cwt. | 411 | 161 | 2,158 | 848 |
| Cutch and gambier | Tons | 2,037 | 1,105 | 29,279 | 19,722 |
| Dyes:— | | | | | |
| Alizarin ....... | Value £ | .. | .. | 16,143 | 16,976 |
| Anilin and other | ,, | .. | .. | 35,460 | 43,496 |
| Indigo ............ | Cwt. | 862 | 852 | 12,021 | 15,314 |
| Nitrate of potash . | ,, | 22,551 | 24,144 | 16,231 | 17,915 |
| Valonia........... | Tons | 2,673 | 1,805 | 27,306 | 20,396 |
| Other articles... | Value £ | .. | .. | 81,691 | 83,532 |
| Total value of chemicals | | .. | .. | 360,704 | 377,046 |

## IMPORTS OF OILS FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Cocoa-nut......... | Cwt. | 11,957 | 31,832 | 12,363 | 34,576 |
| Olive.............. | Tuns | 900 | 599 | 28,543 | 20,729 |
| Palm .............. | Cwt. | 96,573 | 83,619 | 90,116 | 88,798 |
| Petroleum ........ | Gall. | 13,235,414 | 21,764,905 | 249,523 | 373,671 |
| Seed ............... | Tons | 2,307 | 2,372 | 55,668 | 45,014 |
| Train, &c. ........ | Tuns | 1,583 | 3,512 | 27,536 | 57,452 |
| Turpentine ....... | Cwt. | 56,787 | 94,871 | 58,307 | 104,155 |
| Other articles .. | Value £ | .. | .. | 72,849 | 67,280 |
| Total value of oils... | | .. | .. | 594,905 | 787,978 |

## IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Bark, Peruvian .. | Cwt. | 924 | 3,896 | 1,149 | 8,575 |
| Bristles.......... | Lb. | 331,641 | 295,769 | 39,193 | 33,769 |
| Caoutchouc ...... | Cwt. | 21,272 | 30,158 | 227,102 | 400,362 |
| Gum:— | | | | | |
| Arabic......... | ,, | 7,128 | 4,527 | 14,588 | 7,945 |
| Lac, &c. ....... | ,, | 11,477 | 7,961 | 35,822 | 26,670 |
| Gutta-percha .... | ,, | 3,238 | 5,238 | 32,249 | 66,783 |
| Hides, raw:— | | | | | |
| Dry............ | ,, | 40,672 | 36,358 | 104,096 | 90,624 |
| Wet ........... | ,, | 52,249 | 50,390 | 112,778 | 107,016 |
| Ivory ............ | ,, | 707 | 622 | 31,150 | 28,906 |
| Manure:— | | | | | |
| Guano ......... | Tons | .. | 542 | .. | 1,896 |
| Bones ......... | ,, | 3,363 | 2,809 | 11,736 | 11,103 |
| Nitrate of soda... | ,, | 565 | 5,856 | 4,272 | 42,339 |
| Phosphate of lime | ,, | 36,621 | 34,730 | 53,885 | 53,652 |
| Paraffin.......... | Cwt. | 62,720 | 70,104 | 53,489 | 61,346 |
| Linen rags....... | Tons | 2,278 | 2,016 | 23,629 | 18,331 |
| Esparto.......... | ,, | 15,611 | 23,366 | 63,723 | 88,361 |
| Pulp of wood:— | | | | | |
| Chemical ...... | ,, | 35,864 | 18,633 | 172,429 | 117,610 |
| Mechanical .... | ,, | | 20,280 | | 63,264 |
| Rosin............ | Cwt. | 156,159 | 146,213 | 39,407 | 34,132 |
| Tallow and stearin | ,, | 102,177 | 143,474 | 96,916 | 146,749 |
| Tar .............. | Barrels | 34,551 | 25,118 | 21,590 | 19,846 |
| Wood:— | | | | | |
| Hewn ......... | Loads | 332,208 | 269,604 | 762,781 | 610,072 |
| Sawn ......... | ,, | 1,143,801 | 1,183,019 | 2,641,104 | 2,800,178 |
| Staves......... | ,, | 13,156 | 20,669 | 57,607 | 80,154 |
| Mahogany ..... | Tons | 6,711 | 5,733 | 58,831 | 51,202 |
| Other articles.... | Value £ | .. | .. | 975,423 | 1,130,682 |
| Total value ....... | | .. | .. | 5,635,240 | 6,102,167 |

Besides the above, drugs to the value of 67,613*l*. were imported, as against 75,471*l*. in August 1897.

## EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Brass............ | Cwt. | 9,048 | 8,359 | 38,470 | 34,338 |
| Copper:— | | | | | |
| Unwrought.... | ,, | 43,456 | 45,026 | 105,772 | 118,522 |
| Wrought ...... | ,, | 22,713 | 20,750 | 67,994 | 67,988 |
| Mixed metal... | ,, | 15,715 | 21,528 | 37,616 | 54,014 |
| Cutlery ......... | Value £ | .. | .. | 155,043 | 49,910 |
| Hardware ....... | ,, | | | | 108,723 |
| Implements ..... | ,, | .. | .. | 101,299 | 102,658 |
| Iron and steel ... | Tons | 287,719 | 294,142 | 1,853,705 | 1,832,382 |
| Lead ............. | ,, | 2,324 | 3,042 | 30,908 | 43,676 |
| Plated wares ... | Value £ | .. | .. | 29,223 | 41,051 |
| Telegraph wires . | ,, | .. | .. | 93,475 | 265,501 |
| Tin .............. | Cwt. | 8,110 | 9,081 | 26,617 | 33,394 |
| Zinc ............. | ,, | 14,027 | 12,579 | 11,520 | 12,268 |
| Other articles .. | Value £ | .. | .. | 64,076 | 93,103 |
| Total value ....... | | .. | .. | 2,615,718 | 2,857,528 |

## EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST AUGUST.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Alkali............ | Cwt. | 376,186 | 271,245 | 97,185 | 77,507 |
| Bleaching materials | ,, | 87,085 | 76,969 | 26,921 | 20,873 |
| Chemical manures | Tons | 50,496 | 53,122 | 174,799 | 185,564 |
| Copper sulphate .. | ,, | 971 | 482 | 15,069 | 7,792 |
| Medicines....... | Value £ | .. | .. | 81,732 | 94,161 |
| Other articles... | ,, | .. | .. | 200,484 | 226,844 |
| Total value .......... | | .. | .. | 599,190 | 612,241 |

### Exports of Miscellaneous Articles for Month ending 31st August.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 1,031,900 | 1,125,600 | 17,746 | 21,727 |
| Military stores.. Value £ | .. | .. | 95,728 | 164,694 |
| Candles.......... Lb. | 1,831,000 | 1,583,400 | 24,206 | 22,493 |
| Caoutchouc..... Value £ | .. | .. | 94,363 | 102,026 |
| Cement.......... Tons | 36,089 | 23,765 | 60,257 | 46,045 |
| Products of coal Value £ | .. | .. | 97,173 | 93,417 |
| Earthenware ... " | .. | .. | 119,448 | 155,602 |
| Stoneware ...... " | .. | .. | 12,505 | 10,876 |
| Glass:— | | | | |
| Plate.......... Sq. Ft. | 145,955 | 149,982 | 7,820 | 6,945 |
| Flint.......... Cwt. | 7,373 | 7,374 | 15,550 | 17,068 |
| Bottles......... " | 56,162 | 51,047 | 26,527 | 25,400 |
| Other kinds.... " | 20,934 | 21,015 | 14,990 | 18,499 |
| Leather:— | | | | |
| Unwrought .... " | 12,569 | 10,092 | 102,408 | 92,436 |
| Wrought ..... Value £ | .. | .. | 38,553 | 36,911 |
| Seed oil.......... Tons | 3,512 | 2,838 | 60,938 | 51,164 |
| Floorcloth ..... Sq. Yds. | 1,638,900 | 1,754,400 | 71,656 | 75,589 |
| Painters' materials Val. £ | .. | .. | 117,865 | 128,097 |
| Paper .......... Cwt. | 80,248 | 75,516 | 118,382 | 109,621 |
| Rags............ Tons | 4,396 | 5,035 | 22,948 | 24,970 |
| Soap............. Cwt. | 59,442 | 72,267 | 57,836 | 75,533 |
| Total value .......... | .. | .. | 2,394,179 | 2,649,121 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### Applications.

17,611. J. P. Eckersley and G. Eckersley. Improved apparatus for skimming fermented and other liquids. Aug. 16.

17,661. A. Mabille. Automatic apparatus for indicating the pressure of hydrocarbonated or oxycarbonated gases. Aug. 16.

17,722. J. V. Winchester. Improvements in machines for drying brewers' refuse and other materials and substances. Aug. 17.

17,749. J. Taylor. Improvements in furnaces. Aug. 17.

18,033. G. Rusche, jun. Improvements in coolers, scrubbers, or cleaners, for treating liquids. Aug. 22.

18,074. J. Wilson. Improvements in and relating to filter presses and similar filtering apparatus. Aug. 23.

18,234. C. Huber. Improved construction of chambers or receptacles for drying, evaporating, heating, or cooling substances. Aug. 24.

18,345. A. Niel, *née* Jauiot. Improvements in apparatus for regulating the flow and pressure of gas. Aug. 26.

18,442. J. C. Montgomerie. Improvements in filter presses. Aug. 27.

18,562. J. E. Dunn and J. B. Durrant. Improvements in and relating to destructor bagasse and other furnaces. Aug. 30.

18,768. W. Riley and A. Kershaw. *See* Class II.

18,770. W. Riley and A. Kershaw. A new or improved gas holder. Sept. 2.

18,790. W. H. Hyatt. Improvements in drying with apparatus for same. Sept. 2.

18,869. W. W. Wardle. Improvements in drying apparatus. Sept. 3.

18,879. T. H. Cobley. Improvements in the production of colloidal tannates as disincrustants for use in land and marine boilers. Complete Specification. Sept. 3.

18,910. J. Onions. Edge runner grinding mill for powdering marls, bones, cement, slags, &c. Sept. 5.

18,921. J. J. Fries and F. Gerhard. Improvements in furnaces. Complete Specification. Sept. 5.

18,923. E. L. Blosse.—From A. Demanet, Belgium. Improvements in the manufacture of compositions or compounds for preventing or removing incrustation in steam boilers, hot water pipes, and the like. Sept. 5.

18,969. E. I. Braddock. Improvements in the manufacture of composite pipes and in apparatus therefor. Complete Specification. Sept. 6.

18,997. B. Haigh. Improvements in apparatus for carburetting air, and other purposes. Sept. 6.

19,097. W. Scott. Improvements in devices for heating and evaporating liquids. Sept. 7.

19,262. E. Shaw. *See* Class XVI.

19,405. J. Zehra. Apparatus for the production of ice. Complete Specification. Sept. 12.

19,461. J. B. Spurge. Improvements in valves for regulating the flow of gases and liquids under pressure. Sept. 13.

19,529. W. Young, S. Glover, and T. Glover. Improvements in the method of separating from gases suspended particles gases and vapours and for the distillation of liquids holding vapours and gases in solution and in apparatus therefore. Sept. 14.

19,742. E. C. C. Stanford. Apparatus for separating soluble bodies having different degrees of solubility. Complete Specification. Sept. 17.

### Complete Specifications Accepted.*

1897.

15,147. J. H. Bowley. Apparatus for controlling atmospheric conditions. Aug. 31.

22,046. J. Y. Johnson.—From H. K. Mulford, United States. Machines for compressing powdered materials into tablets. Sept. 14.

22,066. J. Dymond. Gas producers. Sept. 14.

22,921. W. Whittaker. Mechanical stokers. Sept. 7.

23,178. A. J. Bell and P. A. G. Bell. Apparatus for the filtration of water sewage and other fluids. Aug. 31.

23,724. J. B. Hilliard. Apparatus and process for treating spirits and other fluids. Sept. 21.

25,654. The Reeves Patent Filters Co., Ltd., and W. Reeves. Filtering apparatus. Aug. 24.

1898.

8017. P. Pessé. Purification and decoloration of liquids and the like, and apparatus therefor. Filed April 4. Date applied for Oct. 1, 1897, being date of application in France. Sept. 7.

13,108. T. Molinard. Refrigerators. Sept. 7.

13,618. S. S. Bromhead.—From Möller and Pfeifer. Drying apparatus. Aug. 24.

## II.—FUEL, GAS, AND LIGHT.

### Applications.

17,531. F. W. Barratt. An improvement in acetylene gas generators. Aug. 15.

17,575. G. Buhlmann. Improvements in self-igniting incandescent mantles for gas-light. Aug. 15.

17,612. E. W. Gillett and C. R. Gillett. Improvements iin acetylene gas generators. Complete Specification. Aug. 16.

17,682. W. P. Thompson.—From N. J. Carey, United States. Improvements in heat generators. Complete Specification. Aug. 16.

17,718. A. Ford-Lloyd and A. E. Mondey. Improvements in acetylene gas apparatus. Aug. 17.

17,734. H. Drivet and J. Bilbie. Improvements in acetylene generators. Aug. 17.

17,751. P. M. A. Drouot. Improved means for atomising and diffusing combustible liquids. Aug. 17.

17,800. J. R. Bell. New or improved apparatus for generating acetylene. Aug. 18.

17,825. G. Little. Improvements in the construction of gas retort settings. Aug. 18.

17,838. A. E. N. Yeadon and S. N. Yeadon. An improved washer or scrubber for purifying gas. Aug. 19.

17,840. T. Thorp. Improvements in and connected with acetylene generators. Aug. 19.

17,892. J. G. Lorrain.—From L. Ward, Cape Colony. Improvements in acetylene gas generating apparatus. Aug. 19.

17,896. S. Pitt.—From The Pharmaceutisches Institute, L. W. Gans, Germany. The production of incandescent materials suitable for use in electric lighting. Aug. 19.

17,922. A. Ford-Lloyd and A. E. Mondey. Improvements in acetylene gas apparatus. Aug. 19.

17,956. J. B. Melhuish and F. Temple. Improvements in the production of acetylene gas. Aug. 20.

18,063. K. G. Gustafson. Improvements in acetylene gas apparatus. Complete Specification. Aug. 22.

18,072. B. Jones An improvement in acetylene gas generators and gasometers. Aug. 23.

18,133. J. Kremer. Improvements in acetylene gas generators. Aug. 23.

18,220. E. Engasser. Improvements in apparatus for generating acetylene gas. Complete Specification. Aug. 24.

18,231. J. F. Bachmann, A. Vogt, C. C. Weiner, A. König, J. Kirchner, and A. Jörg. Electrical heating appliances formed of artificial stone. Complete Specification. Aug. 24.

18,435. D. J. Van Praag. A new or improved acetylene gas generator. Aug. 27.

18,436. G. Matthyssens. Improvements in acetylene producers. Aug. 27.

18,575. F. Edwards and J. Frazer. Improvements in or connected with apparatus for generating and burning acetylene gas. Aug. 30.

18,591. R. Quatannens-Moens and E. Carreer-Dilger. Improvements in and relating to the production of acetylene gas. Complete Specification. Aug. 30.

18,679. T. Clarkson and the Clarkson and Capel Steam Car Syndicate Ltd. Improvements in or relating to the burning of inflammable gas or vapour and in apparatus therefor. Aug. 31.

18,741. W. L. Wise.—From F. C. Wilson, United States. Improvements in apparatus suitable for generating and supplying acetylene gas and the like. Sept. 1.

18,768. W. Hiley and A. Kershaw. Improvements in oxygen gas generators and gas holders. Sept. 2.

18,932. J. Perl. Process for the production of continuous and durable platinum gas-lighting pellets for illuminating gas. Sept. 5.

18,943. R. Söderquist. Improvements in apparatus for the production of acetylene gas. Complete Specification. Sept. 5.

19,086. W. T. Sugg. Improvements in burners for acetylene gas. Sept. 7.

19,087. C. W. S. Crawley. Illumination photometer. Sept. 7.

19,106. G. Hansen and A. Kraelting. A new or improved portable generator of acetylene gas. Sept. 7.

19,131. G. T. Holloway. A new or improved acetylene gas burner. Sept. 8.

19,149. J. S. Goodwin. Improvements in acetylene generators. Sept. 8.

19,180. D. Martini. Improvements in ozone apparatus. Sept. 9.

19,228. E. Evans and J. W. B. Wright. A governor for acetylene and other gases. Sept. 9.

19,323. A. W. Price. Improvements in apparatus for the production of acetylene. Sept. 10.

19,361. W. A. Drake and J. S. Marsland. Improvements appertaining to inclined gas retorts. Sept. 12.

19,369. J. Boss. A new or improved apparatus for generating and utilising gas, such as acetylene. Sept. 12.

19,374. F. Edwards and J. Fraser. Improvements in or connected with apparatus for the storage of acetylene gas. Sept. 12.

19,439. J. Wishart. Improved method and means for congealing and separating solid paraffin from liquid hydrocarbons. Sept. 13.

19,549. A. Browett. An improved device for generating and burning acetylene gas. Sept. 14.

19,674. J. V. Robinson. Improvements in apparatus for the manufacture of acetylene gas. Sept. 16.

19,683. T. R. Waite. Improvements in or relating to acetylene gas generators. Sept. 16.

19,757. A. Landsberger. Improved process of purifying acetylene gas. Complete Specification. Sept. 17.

19,770. A. J. Boult.—From Emile Verbecke, Belgium. Improvements in or relating to metallic mantles for gas-burners. Complete Specification. Sept. 17.

### Complete Specifications Accepted.

### 1897.

19,319. E. Barnard. Apparatus for generating and supplying acetylene gas. Aug. 24.

20,537. F. Windham and E. Fry. Portable lamps for producing gas from carbide of calcium. Sept. 7.

20,572. H. H. Leigh.—From H. Abraham and L. Marmier, France. Generating of ozone, and apparatus therefor. Sept. 7.

21,372. E. C. Chardin. Apparatus for producing acetylene gas. Aug. 24.

21,747. L. Dufrasne. Manufacture of fuel. Aug. 24.

22,327. A. F. B. Gomess. Mantles for use in incandescent gas lighting. Aug. 31.

22,518. G. R. Hislop. Improvements in gas producers. Aug. 24.

23,287. E. Skriwan. Mantles for incandescent lights. Sept. 7.

23,351. O. V. Sigurdsson. Apparatus employed in the manufacture of acetylene gas. Sept. 14.

24,707. C. Guy. Apparatus for the production of acetylene gas. Aug. 31.

27,639. W. J. H. Lazarus. The production of acetylene gas. Aug. 31.

28,003. J. S. Billwiller. Method of and means or apparatus for purifying the products of combustion of acetylene gas flames. Sept. 21.

29,405. V. Maréchal and A. Moreau. Acetylene generators. Aug. 31.

### 1898.

4,352. H. Walker. Process and apparatus for the production and purification of acetylene gas. Aug. 24.

7280A. S. Rosinski and V. E. Ducroix. Composition for automatically lighting gas, &c. Sept. 7.

7427. J. Liddle.—From L. O. Tuebel and D. Whiteis, United States. Apparatus for making gas. Sept. 7.

7671. N. A. Alexanderson. The manufacture of candles. Aug. 31.

11,161. W. W. Strode and G. H. White. The production of acetylene gas. Sept. 14.

12,510. O. Ernst and A. Philips. Apparatus for producing acetylene gas. Aug. 24.

12,701. B. Fröhlich. Briquettes or fuel blocks. Aug. 31.

13,501. A. Koch. Manufacture of durable incandescence bodies. Aug. 31.

13,881. L. von J. Jarocki and Kiczerowski. *See* Class XII.

14,432. The British Acetylene Gas Generator Co. and H. K. Spence. Acetylene gas generators. Sept. 21.

14,729. G. Gastaldi. Apparatus for generating acetylene. Sept. 21.

15,020. E. Henriquez. Acetylene generating apparatus. Sept. 7.

15,212. G. Dillberg. Treatment of calcium carbide to retard its conversion into acetylene gas. Sept. 7.

15,214. G. Dillberg. Means for converting calcium carbide into acetylene gas.

16,090. H. Dant. Acetylene gas generators. Sept. 7.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Applications.

19,321. S. Pitt.—From The Société Anonyme pour la Construction des Appareils Savalle, France. Improvements in the extraction of the benzoles contained in the heavy oils, and in classifying the several hydrocarbons which accompany the benzoles. Complete Specification. Sept. 10.

19,658. G. H. Hosie. Improvements in and relating to the destructive distillation of shales, peat, and other carbonaceous substances. Sept. 16.

## IV.—COLOURING MATTERS and DYES.

### Applications.

17,740. R. Holliday and Sons, Ltd., J. Turner, J. Turner, and H. Dean. Improvements in the manufacture of colouring matters. Aug. 17.

17,912. A. J. Boult.—From F. Fritzsche and Co. Improved manufacture of aldehydes and their derivatives. Aug. 19.

18,477. O. Imray.—From The Firm "Basle Chemical Works," Switzerland. Improvements in the manufacture of dyestuffs of the phthaleine series. Complete Specification. Aug. 29.

18,506. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new substantive azo colouring matters. Aug. 29.

18,572. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new disazo colouring matters. Aug. 30.

18,737. H. E. Newton.— From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of blue dyestuffs for cotton. Sept. 1.

19,008. C. D. Abel. — From E. Beringer, Germany. Extraction of colouring matters from vegetable substances by means of ketone solutions. Sept. 6.

19,009. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of blue dyestuffs of the anthraquinone series. Sept. 6.

19,075. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement Gilliard P. Monnet et Cartier, France. Improvements in the manufacture of azo dyestuffs. Sept. 7.

19,088. C. Rudolph. Manufacture of chlorinated naphthalene sulphonic acids. Sept. 7.

19,327. S. Pitt.—From L. Cassella and Co., Germany. The manufacture of aromatic nitramine monohalides and colouring matters derived therefrom. Sept. 10.

19,698. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of blue to bluish-green dyestuffs of the anthraquinone series. Sept. 16.

### Complete Specifications Accepted.

1897.

18,990. T. R. Shillito.—From J. R. Geigy and Co., Switzerland. Production of paradinitrodibenzyldisulpho acid and of new colouring matters or dyes. Aug. 24.

19,785. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement Gilliard P. Monnet et Cartier. Production of colouring matters and intermediate products. Aug. 24.

19,904. I. Levinstein and Levinstein, Ltd. Production of colouring matters. Aug. 31.

19,994. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Production of new colouring matters related to the rhodamine series. Aug. 24.

20,410. E. S. Wilson and E. Stewart. The production of dyes. Aug. 24.

21,399. A. G. Green and A. R. Wahl. Preparation of colouring matters for cotton. Aug. 24.

21,553. A. G. Green and A. R. Wahl. Production of colouring matters for cotton. Aug. 24.

21,825. A. G. Green, A. R. Wahl, and The Clayton Aniline Company, Ltd. Manufacture of paranitrobenzaldehyde ortho-sulphonic acid, and colouring matters therefrom Aug. 24.

24,287. J. C. Mewburn.—From Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung, Germany. Production of oxyphenylguanidines and their derivatives. Sept. 21.

24,410. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new colouring matters of the safranine series. Sept. 14.

24,644. C. E. Gassmann. Process for obtaining new mordant dyes. Aug. 31.

24,938. S. Pitt.—From L. Cassella and Co., Germany. Producing dyestuffs from amidophenols. Aug. 31.

26,419. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of mordant-dyeing diazo colouring matters. Sept 7.

26,675. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of derivatives of the naphthalene series. Aug. 31.

1898.

6057. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of sulphur trioxide (sulphuric anhydride). Aug. 31.

6533. O. Imray. — From The Farbwerke vormals Meister, Lucius, and Brüning, Germany. Manufacture of mordant dyestuffs from anthraflavine and isoanthraflavine acids. Sept. 14.

12,679. O. Imray.—From The Society of Chemical Industry in Basle, Switzerland. Manufacture of napthacetaldisulphonic acids, and of new colouring matters derived therefrom. Aug. 24.

16,199. A. Goldberg, W. Siepermann, and H. Flemming. Preparation of a yellow sulphur-containing dye from sulpho cyanide salts. Aug. 31.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

17,549. A. Millar. Improvements in producing fabrics or tissues. Aug. 15.

18,217. E. Maertens. Process and apparatus for treating of wool and similar fibres by means of dissolvents. Aug. 24.

18,461. R. Thom. An improvement in linoleum and other floor coverings. Aug. 29.

18,496. The Société Anonyme de Blanchiment Teinture Impression et Apprêt. An improved method of mercerising tissues. Filed Aug. 29. Date applied for April 21, 1898, being date of application in France.

19,211. E. Wendler. Improvements in apparatus for mercerising and imparting lustre to hanks of yarn and the like. Sept. 9.

19,477. H. Krissmanek and F. Auderieth. Apparatus for and in connection with the mercerising of yarns. Sept. 13.

### Complete Specifications Accepted.

1897.

17,901. W. P. Dreaper and H. K. Tompkins. Manufacture of artificial silk from cellulose. Aug. 10.

21,719. M. Rivizza and R. Schönlin.—From J. R. Bantz, Germany. Process for impregnating woollen fabrics intended for keeping the human body at an equal temperature. Sept. 21.

22,351. E. Remy. Relating to the washing and like treatment of printed fabrics, and to apparatus therefor. Sept. 7.

23,670. W. P. Ingham. Manufacture of slag wool. Sept. 21.

24,528. C. Wetherwax. Treating flax and hemp for obtaining textile fibres therefrom. Aug. 31.

26,247. W. G. Heys—From H. David, France. Production of lustrous effects upon vegetable textile materials. Sept. 7.

1898.

12,669. L. S. van Westrum. Mercerising cotton. Sept. 21.

16,729. H. H. Lake. From M. T. Stevens and Sons. Apparatus for drying wool and other textiles. Sept. 14.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

17,558. W. Turner. Improvements in or relating to awking machines for indigo dye vats. Aug. 15.

17,735. A. Keller. An improved process for the production of genuine variously coloured effects upon azo-coloured grounds with basic colours. Aug. 17.

17,797. F. L. Bartelt. A washing and bleaching compound for vegetable fibre or textiles. Aug. 18.

18,218. E. Hungerbühler. Improvements in processes and materials for bleaching. Aug. 24.

### Complete Specifications Accepted.

1897.

17,173. H. H. Lake.—From D. Mattei. Apparatus for dyeing textile fabrics. Aug. 31.

1898.

9484. F. Haber. Mordanting. Aug. 21.

12,379. W. P. Thompson.—From B. Cohnen, Germany. Machines for mordanting, sizing, or mercerising hanks of yarn. Aug. 24.

15,169. M. S. Sharp. Fixing the finish on cotton piece goods. Sept. 7.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

17,852. P. Naef. Method and apparatus for decomposing certain chlorides. Aug. 19.

17,937. W. Galbraith. The extraction or recovery of potash and soda salts and of iodides and bromides from the waste liquors resulting from the extraction of ammonia from blast furnace gases. Aug. 20.

18,110. W. L. Wise.—From The International Patent Company, United States. Improvements in the manufacture of hydrofuge metallic carbides. Complete Specification. Aug. 23.

18,256. J. Farmer. Improvements in or relating to the distilling of acetic acid and in apparatus therefor. Aug. 25.

18,283. C. O. Weber, C. F. Cross, and J. Frankenburg. Improvements in the manufacture of cellulose tetracetate. Aug. 25.

18,376. T. Meyer. Improvement in condensation chambers for the manufacture of sulphuric acid. Aug. 26

19,050. B. Piffard. Improvements in the manufacture of prussiate of potash and Prussian blue. Sept. 7.

19,196. L. G. Hill. An invention for obtaining carbonic acid. Sept. 9.

19,446. H. Blumenberg (junr.). Improvements in the manufacture of peroxides. Sept. 13.

19,470. W. R. Lake.—From La Société Bonnet Ramel, Savigny Girand et Marnas, France. Improvements in the manufacture of dioxide of barium. Sept. 13.

19,528. R. Brunk. Process for manufacturing brick tiles, &c. the refuse of alkali works. Sept. 14.

19,641. E. Donath and E. Ornstein. Improved process for obtaining Prussian blue, prussiate of potash, and alkaline cyanides from used gas-purifying material. Sept. 15.

19,743. B. Piffard. Improvements in the manufacture of chlorine, bleaching liquids, and powders. Sept. 17.

19,752. A. Marshall. Improvements in the manufacture of ammoniacal solution of copper, commonly called Schweizer's Reagent, such as is used in preparing waterproof paper and canvas. Sept. 17.

19,767. J. Raschen and The United Alkali Co., Ltd. Improvements in and apparatus for the manufacture of cyanides and ferro-cyanides from sulpho-cyanides and the recovery of by-products. Sept. 17.

19,786. J. Bilbie and H. Drivet. New or improved process for the treatment of carbide of calcium. Complete Specification. Sept. 17.

### Complete Specifications Accepted.

1898.

14,038. W. Schmeisser. Manufacture of fluoro compounds. Sept. 14.

14,153. J. E. T. Woods and W. A. Byrom. Manufacture of calcium carbide. Sept. 21.

14,780. J. E. T. Woods and W. A. Byrom. Manufacture of magnesium carbide. Sept. 7.

## VIII.—GLASS, POTTERY, and ENAMELS.

### Applications.

17,941. F. Hancock. Improvements relating to the manufacture of pottery from plaster moulds and applied mechanism. Aug. 20.

18,290. C. Kempton, sen. Improvements in glass furnaces. Aug. 25.

18,729. T. Pfister. An improved manufacture of enamelled cloisonné. Complete Specification. Sept. 1.

19,429. J. H. Barratt. An appliance for the decoration of tiles by means of a stream or streams of liquid colour coming into contact with a jet or jets of compressed air. Sept. 13.

19,436. C. C. Hartung. Improvements in machines for rolling wire glass. Complete Specification. Sept. 13.

19,437. E. Walsh, jun. Machine for and method of rolling wired glass. Complete Specification. Sept. 13.

19,528. R. Brunk. Process for manufacturing brick tiles, &c., from the refuse of alkali works. Sept. 14.

### Complete Specifications Accepted.

1897.

24,278. G. L. Siever. Process and apparatus for manufacture of ceramic panels for decorative purposes. Aug. 24.

1898.

5073. E. J. Lutwyche. Ornamental glass. Sept. 7.

12,014. W. M. Edge, W. S. M. Edge, and S. W. Dean. Reproducing engravings upon the surface of pottery and other articles. Sept. 21.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

17,696. J. C. Swan. Improvements in kilns for burning cement - making materials and for similar purposes. Aug. 16.

18,302. G. H. Grey.—From J. W. Hayward, United States. Improvements in the manufacture of artificial asphalt, and in apparatus to be employed therein. Complete Specification. Aug. 25.

18,483. J. Steiger. An improvement in the manufacture of cement. Aug. 29.

18,654. J. Steiger. An improvement in the manufacture of hydraulic cement. Aug. 31.

18,689. S. Grossiord. Improvements in the manufacture of artificial stones. Aug. 31.

18,953. S. Jablónski and S. Niewiarowski. Improvements in and relating to the manufacture of fireproof protecting compositions, and of fireproof articles therewith. Complete Specification. Sept. 5.

19,102. W. L. Wise.—From F. Boas, Canada. New or improved manufacture of material suitable for wall plaster and similar purposes. Sept. 7.

19,136. H. Neil. *See* Class XIX.

19,465. V. Kjeldsen. A furnace or kiln for calcining cement materials. Complete Specification. Sept. 13.

19,579. J. W. Butler. An improved magnesian cement applicable for the manufacture of stone, emery wheels, billiard ball compositions, and for other purposes. Sept. 14.

19,633. G. Collautier. An improved coating material for decorating and building purposes. Sept. 15.

19,684. W. H. James, C. Wallis, and W. MacKinnon. Non - conducting fireproof substance and composition, especially suitable for use in the construction of fireproof buildings or parts of same. Sept. 16.

19,766. The Asbestos and Asbestic Co., Ltd. — From Feodor Boas, Canada. An improved fireproofing composition. Sept. 17.

### Complete Specifications Accepted.

1898.

10,527. P. Wernicke. Process and apparatus for depositing colours for the patterns of cement and other slabs. Aug. 31.

12,902. J. Goodwin, jun. Composition for covering the roofs and walls of buildings. Aug. 24.

13,346. F. Pulda. Fire-proof floors or ceilings. Aug. 24.

## X.—METALLURGY, MINING, Etc.

### Applications.

17,579. F. Ellershausen. Improvements in treating precipitated lead fumes and fine zinciferous lead ores. Aug. 15.

18,032. E. Bohon. Improved method for the extraction of metals from their ores. Aug. 22.

18,110. W. L. Wise.—From The International Patent Company, United States. *See* Class VII.

18,127. A. J. Rossi, J. MacNaughton, and W. D. Edmonds.—Improvements in alloys of titanium and processes for producing same. Complete Specification. Aug. 23.

18,169. D. C. Ripley. Improvements in manufacture of glassware. Complete Specification. Aug. 23.

18,387. A. Gray. Improvement in pickling castings. Aug. 27.

18,424. E. Ferraris. An improved method or process for the direct smelting of sulphide lead ores and the like. Aug. 27.

18,487. R. R. von Berks and J. Renger. Process for the preparation of composite plates of lead or the like materials. Aug. 29.

18,604. G. Wolfram. A process for the production of the light metals (potassium, sodium) and anhydrous oxides and peroxides and cyanides of these metals. Aug. 30.

18,714. G. Hoyermann, A. Harris, and R. Craig. Separating the rough basic slag or phosphate meal from the fine. Sept. 1.

18,788. C. W. Sponsel and W. A. Lorenz. An improved process and apparatus for hardening metals. Complete Specification. Sept. 2.

18,933. F. Ellershausen. Improvements in the treatment of refractory sulphide ores. Sept. 5.

19,012. W. L. Wise.—From J. Burns, United States. Improvement in the art of producing steel. Complete Specification. Sept. 6.

19,029. R. W. Davies. Improvements in apparatus for casting. Filed Sept. 6. Date applied for Feb. 7, 1898, being date of application in United States.

19,051. B. Piffard. Improvements in the manufacture of iron and steel. Sept. 7.

19,168. S. Kurovsky, G. Gschwandtner, and H. Schuster. Improvements relating to the extraction of precious metals from ores, slicks, and the like. Complete Specification. Sept. 8.

19,358. D. Knowles. A new or improved metallic alloy. Sept. 12.

19,383. A. Furno. An alloy for soldering aluminium. Sept. 12.

### Complete Specifications Accepted.

1897.

21,123. T. J. Heskett and H. Jones. Manufacture of steel. Sept. 14.

21,483. C. Wetherwax. Treating gold ores. Sept. 21.

23,883. R. B. Thomas and H. S. Thomas. Treatment of iron or steel plates or sheets to be afterwards coated with tin or terne metal or other coating metal or alloy, or to be otherwise coated or used uncoated, and in apparatus employed in the said treatment. Aug. 31.

25,080. R. Wootton and G. B. Martin. Mode and means of annealing metal. Sept. 14.

1898.

6861. H. Schmidt. Increasing the density of metal alloys in casting. Sept. 14.

7904. A. M. G. Sebillot. Method and apparatus for dressing zinc ores and other volatile metals in blast furnaces. Sept. 14.

11,992. W. P. Thompson. From G. Behr and T. Walfisch, Germany. Process for hardening steel. Aug. 31.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### Applications.

17,583. O. T. Bugg, jun., and D. H. Darling. Improvements in electric batteries. Complete Specification. Aug. 15.

17,588. A. J. Boult.—From V. Busson, France. Improvements in or relating to primary electric batteries. Complete Specification. Aug. 15.

17,741. T. Machin and E. Cake. Improvements in electric batteries. Aug. 17.

17,769. M. Dubrot. Improvements in electric accumulators. Aug. 17.

17,780. J. T. Niblett and M. Sutherland. Improvements in or relating to electric storage batteries. Aug. 18.

17,794. J. G. A. Rhodin. Electro-chemical process for the decomposition of alkaline sulphates or corresponding salts, thereby forming caustic soda sulphuric acid or other products. Aug. 18.

17,828. S. O. Cowper-Coles. Improvements in electrolytic cells. Aug. 18.

17,959. J. C. Chorlier. Improvements in or relating to electrodes for electric batteries. Aug. 20.

18,241. R. N. Lucas and E. Sandford. Improved secondary battery. Aug. 25.

18,522. W. Thomson, Baron Kelvin of Largs. An improved electrolytic apparatus for the production of alkali. Aug. 30.

18,702. A. Schanschieff and A. E. Hodgson. Improvements in electricity accumulators. Sept. 1.

18,713. J. F. L. Moeller and E. A. G. Street. Improvements relating to the extraction of chromium by electrolysis. Sept. 1.

18,847. M. E. Fuld. Improvements in electric batteries. Complete Specification. Sept. 3.

18,958. E. Edser. Improvements relating to the electrolytic decomposition of alkaline salts. Sept. 5.

19,445. H. Blumenberg, jun. Improvements in electric batteries. Sept. 13.

19,536. S. W. Maquay and P. E. Vanderpump. Improvements in and connected with primary batteries. Sept. 14.

19,581. J. Greenwood. Improvements in the decomposition of alkaline salts by electrolysis and in apparatus therefor. Sept. 14.

19,632. W. A. Crowdus. Improvements in and relating to secondary batteries. Sept. 15.

### Complete Specifications Accepted.

1897.

23,352. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Manufacture of electrodes for electrolytic purposes. Sept. 7.

23,489. C. P. Elieson. Electric storage cells. Sept. 14.

25,519. J. Hargreaves. Electrodes. Sept. 14.

26,880. G. W. B. Crees, W. L. Durban, and J. Gore. Electrical furnaces, specially applicable for the production of calcium carbide. Sept. 21.

29,180. P. Brandt. Electrical accumulator. Sept. 14.

1898.

6637. C. E. Acker. Electrolytic production of metallic alloys, and apparatus therefor. Sept. 14.

15,028. H. J. Haddan.—From C. Franke. Manufacture of electrodes. Sept. 21.

15,647. H. Bettermann. Electrically ignited fuses for blasting purposes. Aug. 24.

## XII.—FATS, OILS, and SOAP.

### Applications.

17,842. J. McNeill. Improvements in the manufacture of soap. Aug. 19.

17,977. F. Arledter. Apparatus for manufacturing resinous soap under pressure. Aug. 20.

18,694. J. C. E. Bataille. Process of and apparatus for the extraction and decolorisation of fatty bodies. Aug. 31.

18,730. C. G. Hepburn. Improvements in apparatus for treating and cooling heated liquid fats and oils with air. Complete Specification. Sept. 1.

18,864. A. H. Alderman. New or improved preparation for cleaning and cleansing wall papers, frescoes, water-colour ceilings, and the like. Sept. 3.

19,085. J. N. Harris and H. S. Hendington. Improved mode of treating edible and other oils. Sept. 7.

19,150. G. Bornemann. Process for producing a white soap which, when dissolved in benzene, leaves a clear transparent liquid. Sept. 8.

19,160. A. C. M. Rieck. Improvements in and relating to the manufacture of cacao butter. Sept. 8.

19,294. F. Donati and F. Hornbostel. An improved lubricant. Sept. 10.

### Complete Specifications Accepted.

1897.

21,773. E. Wilczynski. Manufacture of an improved soap. Aug. 31.

25,570. J. Schaschl and H. Hinterberger. Apparatus for purifying oily fluids and for separating the water therefrom. Sept. 14.

1898.

8017. P. Pessé. *See* Class I.

12,508. S. Rosenblum and The Commercial Ozone Syndicate, Ltd. Production of chemical compounds from a certain oil or oils or the fatty acids of the same, and the employment of such compounds in the manufacture of drying oils, varnishes, enamels, lacquers, anti-fouling compositions, and the like. Sept. 7.

13,881. L. von J. Jarocki and T. Kiezcrowski. Purification of Caucasian petroleum. Sept. 14.

17,087. M. Ekenberg. Method of refining oils. Sept. 14.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

18,008. J. Anderson, junior. Improvements in the method of and means for dissolving india-rubber. Aug. 22.

18,179. J. Jones and J. H. Brindlf. Improved rubber substitute. Aug. 24.

18,364. G. Verbereckmoes. A process for the manufacture of artificial india-rubber or caoutchouc. Aug. 26.

19,512. T. H. Leathart. Improvements in the manufacture of red lead and other oxides of lead. Sept. 14.

### Complete Specifications Accepted.

1898.

2887. C. Ives. Plastic compositions. Sept. 14.

12,327. Répin. Treatment of india-rubber, gutta-percha, and other natural elastic substances. Aug. 31.

12,508. S. Rosenblum and the Commercial Ozone Syndicate, Ltd. *See* Class XII.

16,472. E. Schliemann. Manufacture of a wax-like substance. Sept. 7.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Application.

18,429. J. Murgatroyd. Improvements in and relating to machinery for removing hair and wool from skins. Aug. 27.

### Complete Specifications Accepted.

1897.

24,291. A. S. Bury and T. A. Brown. Machines for mixing and agitating size. Sept. 7.

1898.

14,398. F. de Rechter and G. de Rechter. Treatment of skins for use in furriery and tanning operations. Sept. 21.

## XV.—AGRICULTURE AND MANURES.

### Complete Specification Accepted.

1897.

20,660. J. E. J. Johnson. Manufacture of fertilising material. Sept. 14.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

17,650. C. Brueder. The treatment of fecula and other amylaceous matters by means of alkaline chlorites or hypochlorites for the purpose of obtaining a gummy and gelatinous product. Complete Specification. Aug. 16.

18,143. W. P. Thompson.—From J. C. Hoot, Java. Improvements in the purification of syrup, molasses, and the like. Aug. 23.

18,650. J. Evershed. A new adhesive substance or gum, and process for the manufacture thereof. Aug. 31.

18,678. P. Horsin-Déon. A process and apparatus for purifying syrups and other saccharine liquids. Aug. 31.

19,021. A. J. Boult.—From C. M. Higgins, United States. Improvements in or relating to adhesive compounds. Complete Specification. Sept. 6.

19,262. E. Shaw. Improvements in apparatus suitable for preparing syrup for use in the manufacture of sweetmeats, and for condensing, concentrating, and evaporating liquids. Sept. 9.

### Complete Specifications Accepted.

1897.

22,532. G. S. Baker and W. K. Baker. Manufacture of sugar, glucose and the like and apparatus therefor. Sept. 7.

23,875. H. Higgins. Manufacture of lactose or sugar of milk. Sept. 7.

1898.

2287. M. Riegal and J. A. Rose. Production of casein compounds and casein derivatives soluble in water. Sept. 7.

8017. P. Pessé. *See* Class XII.

11,599. J. Welcker. Production of milk-like solutions of albumen. Aug. 24.

11,813. K. Kommerell. Process and apparatus for producing sugar in the form of bars, cubes or other shapes. Sept. 14.

16,292. E. W. Gerbracht and F. G. Wiechmann. Purification of saccharine juices or sugar solutions. Sept. 21.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

17,709. E. J. Boake and A. Boake, Roberts and Co., Ltd. New form of fining for beer and other liquids. Aug. 17.

17,722. J. Y. Winchester. *See* Class I.

18,412. S. White and J. Sleeman. Improvements in the methods of malting, drying, and otherwise treating grain and the like, and in apparatus therefor. Aug. 27.

18,840. W. Wardle. An improved process and apparatus for desiccating barm or yeast and obtaining alcohol therefrom. Sept. 3.

19,574. H. Wegener. An improved process for the utilisation of yeast. Sept. 14.

19,737. C. C. B. Brown and J. Brown (County Mineral Water Co.). An improved beverage. Sept. 17.

### Complete Specifications Accepted.

1897.

23,724. J. B. Hilliard. *See* Class I. Sept. 21.

27,376. W. Jago. Manufacture and treatment of malt preparations. Sept. 14.

29,930. M. Pren and P. Himmel. Saccharometer or device for testing beer worts. Sept. 21.

1898.

15,561. A. Collette, jun., and A. Boidin. Apparatus for manufacture of alcohol. Sept. 21.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

### *A.—Foods.*

18,144. T. Eves, G. Cuddon, and W. R. Bates. An improved process for preserving milk and cream. Aug. 23.

18,145. T. Eves, G. Cuddon, and W. R. Bates. Improvements in apparatus for preserving and storing milk and other liquids. Aug. 23.

18,146. T. Eves, G. Cuddon, and W. R. Bates. An improved process for preserving meat fluids. Aug. 23.

18,500. M. Poppe. Process for the production of margarine or artificial butter. Complete Specification. Aug. 29.

19,705. Société "le Lait." Improved process and apparatus for sterilising and preserving milk. (Date applied for March 10, 1898, being date of application in France.) Sept. 16.

### *B.—Sanitation.*

19,128. A. E. H. Lozé. Improved material for purifying, filtering, clarifying, and sterilising liquids. Complete Specification. Sept. 8.

19,486. W. P. Thompson.—From Valerius D. Anderson, United States. Improvements in driers for treating distillery slops, brewers' grains, slaughter house refuse, and other masses containing a large percentage of moisture, together with solids which are of value for fertilising or other purposes. Complete Specification. Sept. 13.

19,782. Maiche, Ltd.—From Louis Maiche, France. Improvements in apparatus for sterilising water. Sept. 17.

### *C.—Disinfectants.*

18,511. J. L. Wade. Improvements in sulphur candles and in their containing vessels. Aug. 29.

18,751. F. Eiermann. A process for obtaining a mixture which can be used both as a perfume and means for killing insects. Complete Specification. Sept. 1.

19,326. A. Zimmermann. Improvements in antiseptics and deodorisers. Sept. 10.

### Complete Specifications Accepted.

### *A.—Foods.*

1897.

22,112. H. A. Lacroix and A. C. de Candenberg. Process of preserving organic compounds. Aug. 31.

30,877. A. Backhaus. Method for obtaining from milk water-soluble caseïne, preparations and compound caseïne albumen preparations. Sept. 21.

1898.

15,535. J. Wohlgemuth. Production of artificial butter and the like. Aug. 31.

### *B.—Sanitation.*

1897.

17,261. J. B. Petrie. Purifying sewage and foul waters. Aug. 31.

21,982. W. Watson. Method of and apparatus for purifying and softening or similarly treating sewage and other impure water. Aug. 31.

23,428. W. H. L. Galloway.—From J. E. Jordan, South African Republic. Apparatus for treating, softening, and purifying water, sewage, slimes, or other liquids or matters held in solution or suspension. Aug. 24.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

18,970. A. Wiedwald. Improvements in the construction of wooden rollers to be used in the manufacture of paper. Complete Specification. Sept. 6.

19,136. H. Neil. A solution to render paper, strawboard, millboard, wood, and other articles impervious to damp. Sept. 8.

19,324. W. A. Hall. Improvements in the manufacture or treatment of paper. Sept. 10.

19,540. E. Dresen. Improvements in the manufacture of waterproof paper. Sept. 14.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

18,050. E. Kander. An improved method of manufacturing methyl morphine (codeine) and ethyl morphine. Aug. 22.

18,751. F. Eiermann. *See* Class XVIII. C.

19,103. A. J. Boult. From R. Wagnitz, Germany. Manufacture of new chloral combinations. Sept. 7.

19,279. E. R. Blundstone. Improvements in the production of artificial perfumes. Sept. 10.

### Complete Specifications Accepted.

1898.

6238. F. C. Doolittle. A tonic and medicinal preparation or composition applicable also for the manufacture of yeast, bitters, beers, &c. Aug. 24.

13,710. G. Lorenz. Production of a permanent preparation containing the white corpuscles of the blood serum of swine immunised against swine fever. Sept. 21.

13,965. C. A. Steche. Treatment of sandalwood oil and products obtained therefrom. Aug. 24.

15,210. J. C. Mewburn.—From L. D. Huguenin et Cie. and P. Barbier. Manufacture of new perfumes. Aug. 24.

## XXI.—PHOTOGRAPHY.

### Applications.

28,920A. J. W. Bennetto. Improvements in positives for coloured photographs. Complete Specification. Filed Aug. 31. Date claimed Dec. 7, 1897.

18,697. J. C. Chorley. Improvements in the method of and means for producing cellulose films for photographic and other purposes. Sept. 1.

18,799. W. Selke. Improved process and apparatus for the plastic representation of solid bodies by the aid of photography. Complete Specification. Sept. 2.

18,875. W. White. Improvements in apparatus for producing and exhibiting colour photographs. Sept. 3.

### Complete Specification Accepted.

1898.

5721. C. Pietzner. Production of relief photographs. Sept. 7.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

17,577. J. Ross, J. C. Cook, and W. A. Mason. Improvements in explosives. Aug. 15.

17,744. M. Jacobsohn. A new or improved form of match and match receptacle combined. Aug. 17.

17,878. C. W. Curtis and A. H. Durnford. Improvements in the manufacture of gunpowder. Aug. 19.

17,957. P. Ward. A new or improved igniting composition to be employed in the manufacture of matches. Aug. 20.

17,965. P. Ward. A new or improved igniting composition to be employed in the manufacture of matches. Aug. 20.

17,978. P. Ward. A new or improved igniting composition to be employed in the manufacture of matches. Aug. 20.

18,002. C. F. Hengst. An improved smokeless and waterproof explosive compound. Aug. 22.

18,233. A. Luck and C. F. Cross. Improvements in the treatment of nitrocellulose. Aug. 24.

18,570. W. P. Ferguson. Improvements in fulminate caps for dynamite cartridges. Complete Specification. Aug. 30.

18,800. A. Luck. Improvements in the manufacture of explosives. Sept. 2.

18,814. F. J. Gillibrand. Improvements in safety matches. Sept. 3.

18,868. A. Luck and C. F. Cross. An improvement in the treatment of nitro-cellulose. Sept. 3.

18,935. J. Okell.—From H. Pipitz, Austria. Improvements in and connected with the manufacture of explosives. Sept. 5.

19,028. A. von Stubenrauch. Improvements in or relating to the manufacture of explosives. Sept. 6.

19,039. S. A. Rosenthal and S. J. von Romocki. Improvements in the manufacture of matches capable of being struck anywhere without yellow phosphorus. Sept. 7.

19,074. W. H. Cromie. Improvements in the manufacture of explosives. Sept. 7.

19,127. J. C. Hamilton. Improvements in explosives suitable for blasting. Sept. 8.

19,230. C. S. Bailey. Improvements in smokeless powders. Sept. 9.

19,260. W. Friese-Greene. Improvements in the manufacture of explosives. Sept 9.

### Complete Specifications Accepted.

1897.

24,385. G. Benekó. Waterproofing blasting cartridges, and new waterproofing compounds suitable therefor. Aug. 24.

29,202. J. Joscht. Manufacture of matches. Sept. 21.

29,204. J. Bown. Fog signals. Sept. 21.

## PATENTS UNCLASSIFIABLE.

### Applications.

18,340. S. B. Earle.—From P. W. Wierdsma and J. Kuipers, Holland. Improvements in and in the manufacture of a substance or product for use as a substitute for vulcanite, hard woods, or other hard materials, or for any other uses or purposes to which same may be applicable. Aug. 26.

18,809. P. G. Sanford. Improvements in the treatment of blood serum and other albuminous liquids or semi-liquids for use in the manufactures and the arts. Sept. 2.

19,689. G. G. Smith and M. Stuart. Improvements in the manufacture of fluid-proof articles. Sept. 16.

### Complete Specification Accepted.

1898.

15,115. J. Macneil. Improved composition, and process for its manufacture. Aug. 31.

* * *

Printed and Published by Eyre and Spottiswoode, East Harding Street, London, E.C., for the Society of Chemical Industry,

# THE JOURNAL
OF THE
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## A MONTHLY RECORD

FOR ALL INTERESTED IN CHEMICAL MANUFACTURES.

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SESSION 1898-99.

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Watson Smith. "On a Central American Lignite Soluble in Benzol. Exhibit of Miike Coal and Shale, with Fossils, &c."

A. W. Crossley and H. R. Le Sueur. (1.) "A New Method for Determining the Melting-point of Fats and Waxes." (2.) "Indian Edible Oils."

Oscar Guttmann. "Exhibition of New Filling Material for Reaction Towers."

Dr. G. Harris Morris. "The Causes of Stench in Beer."

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SESSION 1898-99.

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SESSION 1898-99.

Monday, Nov. 28th.—W. McD. Mackey. "The Colorimetric Estimation of Oxygen."

## NOTICES.

Foreign and Colonial Members are reminded that the subscription of 25*s.* for 1899, payable on January 1st next, should be sent in good time to the Treasurer (Mr. Samuel Hall), in order to ensure continuity in the receipt of the Society's Journal. Any changes of address to appear in the new List of Members now in course of preparation, should reach the General Secretary not later than January 15th, 1899.

### Collective Index.

In reply to numerous inquiries, the printing of the Collective Index is in active progress, the bulk of the work being already in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer and Committee who have the work of correction in hand.

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## Yorkshire Section.

### GLYCERIN ESTIMATION.

BY F. W. RICHARDSON, F.I.C., AND ADOLF JAFFÉ.

(*This Journal, April* 30, 1898, pp. 330—333.)

Errata.

Page 331, col. 2, under "Estimation of Glycerin in Crude Glycerin," in line 2, for "50 c.c." read "250 c.c."; and in line 11, for "2 grms." read "0·2 grm."

# Obituary.

## HENRY TRIMBLE, Ph.M.

PROFESSOR OF PRACTICAL CHEMISTRY IN THE PHILADELPHIA COLLEGE OF PHARMACY; EDITOR OF THE AMERICAN JOURNAL OF PHARMACY; MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY.

In the decease, at the comparatively early age of 42, of Prof. Henry Trimble, American pharmacists have sustained a severe loss, for besides the Professorship he held in the Philadelphia College, he also acted as editor of the *American Journal of Pharmacy*, and was an authority of high repute as well as a scientific investigator in connection with pharmacology.

He was also an accomplished botanist, and contributed an article on the Coniferæ to Sargeant's monumental work, "Silva of North America."

To chemical technologists he is best known by his work on the "Tannins," of which one volume was published in 1892 and a second in 1894.

Henry Trimble was born near Chester, Pa., U.S.A., on May 22nd, 1853, and was the son of Stephen M. Trimble, a member of the Society of Friends. He was brought up on a farm, going to school in winter and working during the summer. His earlier education was obtained at the Westtown School, in Chester County, and at the age of 19 he was apprenticed to learn the drug and apothecary business. Two years later, in 1871, he entered the Philadelphia College of Pharmacy, the institution in which most of his subsequent active career was to be cast, and here he graduated in 1876.

From the year 1878 to 1883 he was partner in a retail pharmacy; but his love for chemical research induced him to return and devote himself entirely to college work.

After a connection with the Philadelphia College of Pharmacy, as a teacher, from the year 1883, Henry Trimble was appointed assistant to Prof. Sadtler, later on receiving the sole charge of the chemical laboratories as Professor of Practical Chemistry. Henry Trimble was a man of much amiability of character, and he possessed the rare gift of inspiring in others, especially his pupils, the enthusiasm which he himself possessed. More closely in connection with his work as a teacher of chemistry, he published in 1885, a "Handbook of Analytical Chemistry," which has passed through several editions. In conjunction with Prof. Sadtler, he also wrote a "Text-book of Chemistry." A great deal of original research by himself alone, and also in conjunction with his pupils, also stands to his credit. Since he succeeded J. M. Maisch in 1894 as editor of the *American Journal of Pharmacy*, it is reported that Henry Trimble has more than succeeded in sustaining the reputation of that journal for originality, &c. The published articles which appeared under his name in that journal in the years 1875—1898, number fifty-four. A complete list of his published works is given in the J. Franklin Inst. 146, [1], 1893, 311—312.

In a brief monograph by the Committee of our New York Section, occurs the following short but comprehensive eulogium, which may be fittingly used to close this notice:—"His life was marked by unremitting industry, and a devotion to duty almost unexampled."

# Journal and Patent* Literature.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Filters [Sewage or Other], Impts. in the Discharge of Liquids upon.* T. Caink, Worcester. Eng. Pat. 19,153, Aug. 19, 1897.

The liquid is discharged through a perforated iron pipe which is caused to travel over the surface of the filter. In this way the whole of the filter is utilised. The movement is effected by means of an electric or other motor, or preferably by the action of the liquid itself in issuing from the spreader. In the latter case the pipe is divided longitudinally into two chambers, each of which is fed from a valved siphon at one end. These valves alternately and automatically open and close, so as to reverse the direction of the discharge each time the spreader reaches the end in either direction. The spreader runs on wheels, backwards and forwards, delivering the liquid like rain over every part of the filter.

Circular filters are fed by means of spreaders, driven similarly to the apparatus known as Barker's mill.

—N. H. J. M.

*Filtration of Water, Sewage, and other Fluids; Impts. in Apparatus for the.* W. J. Bell and P. A. G. Bell, Manchester. Eng. Pat. 23,178, Oct. 9, 1897.

The filtering materials of a closed, water-tight filter are thoroughly washed from time to time by means of a hollow revolving vertical hydraulic shaft, provided with hollow radial arms to which vertical rakes are attached. To clean the filtering material, water is forced down the shaft through perforations in the arms; at the same time the shaft with the arms is revolved, and the filtering material thus stirred up at the same time that the water is being forced in. The impure water escapes through an outlet pipe in the roof.—N. H. J. M.

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* Any of these specifications may be obtained by post by remitting 8*d*.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Treating, Softening, and Purifying Water, Sewage, Slimes, or other Liquids or Matters held in Solution or Suspension; Impts. in or relating to Machinery or Apparatus for.* W. H. L. Galloway, Johannesburg, South African Republic, and Inverurie, Aberdeenshire. Eng. Pat. 23,428, Oct. 12, 1897.

The liquid, after treatment with chemicals in tanks, passes through a launder, provided with baffle plates, into a pipe connected with a series of tanks. The liquid enters each tank at about the middle, underneath an inverted cone, the object of which is to check the rush of water and to facilitate precipitation. The tanks are also provided with sloping sides, for conveying the sludge to valves at the bottom. Before leaving the last tank, the liquid passes upwards through a screen of matting or metal, &c., and a filter of shavings or carbon.—N. H. J. M.

*Filtering Apparatus, Impts. in or connected with.* The Reeves Patent Filters Co., Ltd., and W. Reeves, Victoria Street, Westminster. Eng. Pat. 25,654, Nov. 4, 1897.

The claims are for a filter, particularly one of large size for towns' use, formed with a conical wall surrounding the filtering material, to prevent the creeping of the liquid. It is provided with "a spindle furnished with arms carrying rotative stirrers, that are adapted to enter and agitate the upper or finer layer of material on the filtering bed when said spindle is revolved." "The main filtering bed is superimposed by a conical wall," which is combined with "a settling chamber and," above that, "a coarse filtering bed," both the latter being arranged between the upper conical wall and the wall of the filter tank. Below the main filtering bed is "a slotted hollow casting and a perforated strainer or grid, formed in segments, which are supported above a funnel-shaped bottom." In a modification, instead of a hollow casting and a strainer, there is "a series of tubes, having perforations or nozzles," which "radiate from a central box or chamber in the conical" wall surrounding the filtering material.—R. S.

*Sewage and other Liquids, Impts. in Apparatus for the Treatment of.* D. Cameron, F. J. Commin, and A. J. Martin, Exeter. Eng. Pat. 27,349, Nov. 22, 1897. (This Journal, 1897, 459 and 753.)

An apparatus for automatically discharging a liquid from one receptacle and delivering it into another, by means of a valve which opens when the liquid reaches a certain height. When more than two chambers are employed, a second valve may be placed on the supply pipe of each, and may be actuated by the discharge of one of the chambers belonging to some other pair. Any chamber will then receive liquid only when both supply valves are open.

The flow of liquid may be checked by means of a valve, which is wholly or partly closed by the rising of a float in a well, connected with the chamber which is filling.
—N. H. J. M.

*Pressure Filter, An Improved.* E. J. Duff, Liverpool. Eng. Pat. 16,014, July 22, 1898.

The filter consists of a metal casing provided with a cover, which can be securely clamped thereon, and to which, on the inner side, a hollow porous filtering stone, fitting into and nearly filling the casing, is securely fixed. The inlet is at the bottom, communicating with the annular space around the porous stone, while the outlet is at the top, communicating with the central hollow space into which the filtered water passes. The claim is for the filter as described.
—R. S.

*Measuring or Indicating Temperatures at a Distance, Apparatus for.* A. J. de la Croix, St. Petersburg. Eng. Pat. 16,471, July 28, 1898.

The apparatus is an air thermometer, which the patentee calls a "telethermometer." It is principally intended for indicating the temperature of grain in stores or produce in silos. By its use the temperatures of several separate localities may be indicated at one station. It consists of a comparatively large hermetically sealed glass vessel placed in the material, the temperature of which is to be observed, connected by a thin lead tube to a second, considerably smaller, vessel, also hermetically closed, and partly filled with mercury. The cover of the second vessel is dome-shaped, and a glass standpipe projects therefrom, its lower end dipping into the mercury. When in use, the vessels are filled with air under a moderate pressure above that of the atmosphere. The first vessel is provided with a liquid seal at its junction with the tube. The claims are for the whole apparatus, the liquid seal, and the second vessel with its accessories.—R. S.

*Drying, Apparatus for.* S. S. Bromhead, Queen Victoria Street. From Möller and Pfeifer, Berlin. Eng. Pat. 13,618, June 18, 1898.

The inventor claims "a device for drying suitable material, characterised by the drying operations taking place in a space void of air, and by means of steam generated from the said material," such "steam being highly heated before it is again employed." "The sides of the steam-heater and then the walls of the drying chamber are brought in contact with the heat of the furnace, with a view to avoid the overheating of the metal surfaces enclosing the drying chamber and of the materials therein." "The escaping steam" is claimed "for further drying, or for any other object, until all the available heat in the steam is entirely exhausted."
—R. S.

## II.—FUEL, GAS, AND LIGHT.

*Carburetted Water-Gas.* W. Leybold. J. für Gasbeleucht. 1898, **41**, [33], 525—528. (See also this Journal, 1898, 831.)

In the United States, native petroleum, or, preferably, a distillate therefrom, is used for producing carburetted water-gas. In England, solar oil, a distillate boiling between 300° and 360° C. derived from Russian petroleum, is employed, in addition to a little Scotch shale oil. The solar oil is delivered from tank-steamers into the buyer's tanks at an inclusive cost of 2¾*d.* to 3*d.* per gallon. But in Germany there is exacted on crude petroleum, petroleum distillates and residues, and shale oil, an import duty of 6 marks per 100 kilos. gross weight. On distillates boiling above 300° C., which are classed as lubricating oils, the duty is 10 marks. No exemption is made in favour of the gas industry. There are, however, three descriptions of native German oils which can be used for gas-making, *viz.*, (1) the product of the brown-coal distilleries of Saxony and Thuringia; (2) the oil from Messel, near Darmstadt; and (3) petroleum from Alsace. The cost of the Alsace petroleum is, however, rather greater than that of imported petroleum on which the duty has been paid.

The oil from the works of Saxony and Thuringia comprises gas-oil of sp. gr. 0·875, heavy oil of sp. gr. 0·92, paraffin residues, &c. The gas-oil has been successfully employed for making carburetted water-gas at Brussels but the oil-gas works absorb nearly the whole production. The heavy oil is used as boiler-fuel in the navy, but trial showed that it was nearly as good as the gas-oil for the production of carburetted water-gas. The Messel works deal with a shale which yields 6 to 10 per cent. of tar, from which gas-oil, heavy oil, and paraffin are obtained. Trials were made at the Brussels gasworks of German oils and American petroleum. The blows (periods of air-blast) were of three minutes' and the runs (periods of gas-making) of six minutes' duration. Clinkering occupied about 15 minutes twice daily. The cost of labour has been computed on the assumption that there are needed for the production of 50,000 cubic metres of gas in 24 hours, one foreman, four gas-makers, two firemen, who will also attend to the coke elevator, two boiler-men, two engine-men, and three labourers. The wages of these men amount to 61·9 marks per diem, or 1·24 marks per 1,000 cubic metres of gas made. Other fixed charges per 1,000 cubic metres are:—boiler-fuel, 0·25; purifying material, 0·21; wages for purification, 0·20; maintenance and sundry expenses, 0·70 mark. These charges and the wages amount to a total of 2·60 marks per 1,000 cubic metres. The Brussels trials, then, give the results shown in the annexed table. If the duty be added to the cost of American petroleum, the cost of 1,000 cubic metres of gas from that oil, becomes increased to 68·18 marks.

| — | American Petroleum (Duty unpaid). | Gas-Oil from Halle. | Heavy Oil from Halle. | Gas-Oil from Messel. |
|---|---|---|---|---|
| Cost of oil (in Hamburg) per 100 kilos. Marks | 6·00 | 7·00 | 7·00 | 8·85 |
| Specific gravity of oil | 0·870 | 0·875 | 0·920 | 0·870 |
| Specific gravity of the gas made. | 0·74 | 0·73 | 0·73 | 0·75 |
| Illuminating power of the gas (English candles per 5 cb. ft.). | 15·64 | 15·32 | 15·98 | 16·70 |
| Coke used per 1,000 cb. m. Kilos. | 596·3 | 555·5 | 643·1 | 562·2 |
| Oil used per 1,000 cb. m. " | 416·8 | 393·2 | 427·6 | 380·6 |
| Make of gas × candle-power candles per 100 kilos. of oil | 3,753 | 3,897 | 3,738 | 4,387 |
| Grammes of sulphur per cubic metre of unpurified gas. | 1·101 | 1·236 | .. | 1·398 |
| Grammes of sulphur per cubic metre of purified gas. | 0·291 | 0·257 | 0·323 | 0·270 |
| Cost per 1,000 cb. m. of gas:— | Marks. | Marks. | Marks. | Marks. |
| For coke | 9·31 | 8·68 | 10·05 | 8·78 |
| For oil | 25·01 | 27·52 | 29·93 | 33·68 |
| For wages and other fixed charges. | 2·60 | 2·60 | 2·60 | 2·60 |
| Total cost per 1,000 cb. m. | 36·92 | 38·80 | 42·58 | 45·06 |
| [Total cost per 1,000 cb. ft. | 12·54*d.* | 13·19*d.* | 14·47*d.* | 15·31*d.*] |

The cost of carburetted water-gas appears from the table to be rather greater than the cost of coal-gas when coal is cheap. In 1896, coal-gas was made at a large German factory for 26·61 marks per 1,000 cubic metres; but in 1891–92 the cost, owing to dear coal, was 50·07 marks. In such circumstances carburetted water-gas would compete with coal-gas. As a rule, however, only 8 to 10 per cent. of the total production of gas would be carburetted water-gas, and its candle-power would be about 17 in order that the mixture might be 16-candle gas. The oil required for the production of this proportion of carburetted water-gas in about five large German gasworks would be equivalent to the present total oil production of the country, and therefore the adoption of that gas on an extensive scale would stimulate the native oil industry, which is at present confined to a few centres.

In England, carburetted water-gas is made solely by the Lowe system. The Dellwik water-gas system (this Journal, 1897, 900; also pages 525 and 431), in which carbonic acid is formed in place of the carbonic oxide of the producer-gas, during the periods of air-blasting, has not been applied to the production of carburetted water-gas, because the producer-gas is at once needed for heating the carburetting and superheating chambers in which the oil is gasified. A non-illuminating water-gas may, however, be mixed with coal-gas, and the requisite illuminating power be given to the mixture by means of benzene. But such a mixture would contain more carbonic oxide than a mixture of coal-gas and carburetted water gas. By the ordinary water-gas process 73 per cent. of the heating value of the coke is expended during the times of the air-blast, and only 27 per cent. is utilised for gas production; in the Dellwik process the proportions are reversed, for 74 per cent. of the heating-value of the coke is actually applied to gas making. The mean composition of two samples of Dellwik water-gas, made at Warstein in the author's presence, was as follows:—

| | Vols. Per Cent. |
|---|---|
| Hydrogen | 48·3 |
| Carbonic oxide | 40·4 |
| Methane | 0·5 |
| Carbonic acid and sulphuretted hydrogen | 5·2 |
| Oxygen | 0·1 |
| Nitrogen (by difference) | 5·5 |

The bed of fuel in the Dellwik generator is only 1·2 metres deep, whereas about 3 metres is the customary depth in the ordinary generator.—J. A. B.

*Explosive Mixtures of Gases in Thin Layers, Inflammability of.* F. Emich. Monatsh. für Chem. 1898, **19**, [6], 299—320.

Explosive mixtures of gases are allowed to flow slowly between two small glass plates, through which very fine wires are fused so as to be just level with the surfaces of the plates. The distance between them can be varied and accurately measured. When this distance is smaller than a certain value, a spark passed between the wires fails to ignite the gas. This distance may therefore be taken as a measure of the inflammability of the mixture. In this way it is found that the most inflammable mixtures of the gases investigated, have the following compositions:—

Hydrogen, 1 vol. + oxygen, 1 vol.
Hydrogen, 1 vol. + chlorine, 2 vols.
Methane, 1 vol. + oxygen, 3 vols.
Carbon monoxide, 2 vols. + oxygen, 1 vol. (moist).

The inflammability increases nearly in proportion to the pressure of the gas. In the case of carbon monoxide and oxygen, the inflammability increases rapidly with the amount of aqueous vapour present (up to 5 per cent. by volume), and thereafter more slowly.—T. E.

*Furnace for Burning Sugar Cane* [*Megasse*]. L. Colson. Le Génie Civil, Nov. 6, 1897, 12; Proc. Civil Eng. **132**, [2], 54.

The author found by experiments that for each ton of cane crushed per hour, there should be in the furnace, 40—50 sq. m. (431—539 sq. ft.) of heating surface; 0·2 sq. m. (2·15 sq. ft.) of chimney area for a height of 25—30 m. (82—98 ft.); and 0·5 sq. m. (5·5 sq. ft.) of grate area. Tubular boilers are preferred on account of their evaporating power and economy of fuel; and the difficulty of their being unsuited for a variable consumption of steam, is surmounted by placing a large reservoir of steam and water over each boiler.

The following conditions are given as leading to the best results:—The furnace should have as large an arch as possible, and also a very strong draught. The gases and flames should be well combined before they reach the boilers. A separate supplementary furnace should be used for burning wood or straw.—A. S.

*Silicon, Electrical Resistance of Crystallised.* Le Roy. L'Electricien, 1898, **15**, 101.

The author has found that crystallised graphitic silicon, which can easily be compressed into the form of rods, is the best substitute for the long, thin metallic resistances usually employed in the construction of electrical heating apparatus. Its specific resistance has not yet been accurately determined, but it seems to depend to a very large extent on the degree of fineness of the powdered silicon and on the pressure and temperature employed whilst it is being manufactured into rods. By varying these conditions, rods, having a cross section of 40 mm. and a length of 70 cm., can be made, the resistance of which varies between 25 and 200 ohms, whilst the resistance of similar rods of carbon and German silver, is about 0·15 and 0·00085 ohm.—J. S.

*Incandescence Mantles of Commerce, Examination of.* E. Hintz. Zeits. Anal. Chem. 1898, **37**, [8], 504—524.

In a previous paper (this Journal, 1898, 378) it was shown that the illuminating power of mantles made of 99 parts of $ThO_2$ and 1 part of $CeO_2$ was not influenced by the addition of up to 1 per cent. of $Nd_2O_3$, $La_2O_3$, or $Yt_2O_3$, or from 0·2 to 1 per cent. of $ZrO_2$, while the influence of less than 0·2 per cent. of $ZrO_2$ was doubtful. Continuing these experiments exhaustively, the author quotes a large number of results, which bear out the following conclusions, (In each case "thoria-ceria" implies the above mixture of 99 : 1):—(1.) The addition of less than 0·2 per cent. of $ZrO_2$ to the thoria-ceria does not influence the illuminating power (which brings $ZrO_2$ into line with the other oxides). (2.) When up to 2 per cent. of $Nd_2O_3$,

$La_2O_3$, or $Yt_2O_3$ is admixed with the thoria-ceria, the illuminating power is not injured, but about 2 per cent. of $ZrO_2$ lowers it slightly. (3.) In presence of greater proportions of the added oxides, the illuminating power is almost always lower, a high proportion of $Nd_2O_3$ or $Yt_2O_3$ being more injurious than a similar proportion of $ZrO_2$ or $La_2O_3$. The following is an abstract of the results obtained at this point, the italicised lines giving the British equivalents in *candle-power, hourly consumption of gas in cubic feet, and candle-power per cubic foot :—*

| Composition of Mantle. | Maximum Illuminating Power: Hefner Units. | Hourly Gas Consumption in Litres. | |
|---|---|---|---|
| | | Total. | Per Hefner Unit. |
| Thoria-ceria ..... 95<br>Zirconia ......... 5 | 43<br>*37·8* | 123<br>*4·3* | 2·9<br>*8·7* |
| Thoria-ceria ..... 80<br>Zirconia ......... 20 | 50<br>*44·0* | 105<br>*3·7* | 2·1<br>*11·9* |
| Thoria-ceria ..... 95<br>Neodymia ....... 5 | 66<br>*58·1* | 120<br>*4·2* | 1·8<br>*13·8* |
| Thoria-ceria ..... 80<br>Neodymia ....... 20 | 40<br>*35·2* | 143<br>*5·0* | 3·6<br>*7·0* |
| Thoria-ceria ..... 95<br>Lanthana........ 5 | 73<br>*64·2* | 135<br>*4·7* | 1·9<br>*13·6* |
| Thoria-ceria ..... 80<br>Lanthana........ 20 | 54<br>*47·5* | 124<br>*4·4* | 2·3<br>*10·8* |
| Thoria-ceria ..... 95<br>Yttria ........... 5 | 62<br>*54·5* | 126<br>*4·4* | 2·0<br>*12·3* |
| Thoria-ceria ..... 80<br>Yttria ........... 20 | 38<br>*33·4* | 121<br>*4·2* | 3·2<br>*7·9* |

*Effect of Varying the Proportions of Thoria and Ceria.*—To prepare pure $ThO_2$, thorite was decomposed by HCl, and the solution was treated with $H_2S$ and oxalic acid as usual. The precipitated oxalates were digested with ammonium oxalate; the solution was diluted largely, allowed to stand some time, and filtered; the oxalates were reprecipitated by acidifying the filtrate with HCl, and were converted successively into the oxides, sulphates, hydrates, and chlorides. The diluted, weakly-acid solution of the chlorides was precipitated with thiosulphate, the precipitate was redissolved in HCl and reprecipitated, after which it was redissolved in HCl; the hydrate of thorium was precipitated by ammonia and converted into nitrate solution containing 14·5 per cent. of $ThO_2$. Pure cerous nitrate solution was also prepared, and from these solutions, mixed in various proportions, mantles were made. The mantles were burned 25—50 minutes before testing. The following is an abstract of several tables of results, the British equivalents being interpolated as in the foregoing table :—

| Composition of Mantle. | Illuminating Power: Hefner Units. | Hourly Gas Consumption in Litres. | |
|---|---|---|---|
| | | Total. | Per Hefner Unit. |
| Pure thoria.......... | 2·6<br>*2·3* | 130<br>*4·6* | 50<br>*0·5* |
| Pure ceria ........... | 2·0<br>*1·7* | 125<br>*4·4* | 61<br>*0·4* |
| Thoria......... 99·9<br>Ceria .......... 0·1 | 17<br>*15* | 115<br>*4·0* | 6·7<br>*3·7* |
| Thoria......... 99·8<br>Ceria .......... 0·2 | 41<br>*36·1* | 129<br>*4·5* | 3·1<br>*8·0* |
| Thoria......... 99·5<br>Ceria .......... 0·5 | 62<br>*54·6* | 133<br>*4·7* | 2·1<br>*11·6* |
| Thoria......... 99<br>Ceria .......... 1 | 71<br>*62·5* | 101<br>*3·5* | 1·4<br>*17·8* |
| Thoria......... 98<br>Ceria .......... 2 | 61<br>*53·7* | 101<br>*3·5* | 1·7<br>*15·3* |
| Thoria......... 97<br>Ceria .......... 3 | 59<br>*51·9* | 101<br>*3·5* | 1·7<br>*14·8* |
| Thoria......... 95<br>Ceria .......... 5 | 40<br>*35·2* | 101<br>*3·5* | 2·5<br>*10·0* |
| Thoria......... 90<br>Ceria .......... 10 | 11<br>*9·7* | 101<br>*3·5* | 9·5<br>*2·7* |
| Thoria......... 80<br>Ceria .......... 20 | 8·4<br>*7·4* | 101<br>*3·5* | 12<br>*2·1* |
| Thoria......... 60<br>Ceria .......... 40 | 3·3<br>*2·9* | 101<br>*3·5* | 31<br>*0·8* |
| Thoria......... 30<br>Ceria .......... 70 | 2·2<br>*1·9* | 103<br>*3·6* | 48<br>*0·5* |

Of many experiments on durability the following may be quoted. A mantle containing 99·9 parts of $ThO_2$ and 0·1 part of $CeO_2$, and giving at the beginning, 17 Hefner units with a consumption of 115 litres of gas per hour (15 candles with 4 cb. ft.), gave, after 400 hours' burning, 9·3 units with 116 litres per hour; and after 800 hours, 7·5 units with 114 litres. A mantle of 99·5 parts of $ThO_2$ and 0·5 part of $CeO_2$, giving at the outset 40 Hefner units with 115 litres per hour (35·2 candles with 4 cb. ft.), gave, after 400 hours, 27 units with 116 litres per hour; and after 800 hours, 22 units with 113 litres per hour. A mantle of 99 parts of $ThO_2$ and 1 part of $CeO_2$ gave, at these three periods, 54 units with 115 litres, 38 units with 116 litres, and 32 units with 114 litres.

Where the amount of ceria in a mantle is very small, the percentage may be approximately estimated by a photometric test.—H. B.

*Incandescent Spirit Lamps.* J. für Gasbeleucht. 1898, **41**, [37], 595—597.

An incandescent lamp is described, which, when consuming spirit of 86 volumes per cent. strength, is said to give a very powerful light, *viz.*, on the average, equal to 71·3 Hefner lights (about 62·5 English standard candles). The consumption of spirit averages 19·6 c.c. per 10 Hefner light-hours. The lamp is said to be specially suitable for lighting large spaces; it has no wick, is easy of manipulation, and is so constructed that an explosion is said to be impossible.

—J. A. B.

*Phosphorescent Sulphides of Strontium, Composition of.* J. R. Mourelo. Comptes Rend. **127**, [8], 372.

*See under* XXIV., *page* 961.

*Blast-Furnace Gas, "Simplex" Motor Using.* M. A. Witz. Iron and Steel Inst., Autumn Meeting, 1898.

*See under* X., *page* 926.

*Carburetted Water-Gas, The Chemical Composition and Technical Analysis of.* E. H. Earnshaw. J. Franklin Inst. 1898, **146**, [3], 161.

*See under* XXIII., *page* 955.

*Acetylene, Toxicological Detection of.* D. Vitali. Boll. Chim. Farm. **37**, 440; Chem. Centr. 1898, **2**, [9], 586.

*See under* XXIII., *page* 954.

## PATENTS.

*Furnaces and Apparatus for, and in the Recovery and Purifying of Iron, Metals, Slags, Cinder Tap Ores, and other kinds of Waste Products, and for other Purposes; Impts. in.* W. J. Clapp, Birmingham. Eng. Pat. 13,332, June 15, 1898.

The claims, four in number, are for "improvements in furnaces and apparatus for and in the recovery and purifying of iron, metal, slag, and cinder tap ores, and other kinds of waste products." Also "in the combination in a furnace (having two fire bridges, also claimed) of an arrangement for charging or injecting into the flame a limited quantity of lime or other purifying agents by means of a steam jet or jets," and "in the arrangement for charging or operating on the flame as it enters the stack, with a jet or spray of steam and a jet or spray of water, whereby the recovery of various by-products [slag-wool, &c.] from the flame is effected, and explosions in the stack prevented."—W. G. M.

*Hydrocarbon Engines, Improved Process for Attaining a very High Ignition Temperature in.* G. C. Dimond, Liverpool. From A. Meyer, Cannstatt, Würtemberg, Germany. Eng. Pat. 19,642, Aug. 25, 1897.

The inventor claims "a process for attaining a high ignition temperature—*i.e.*, great economy—in hydrocarbon engines, consisting in the residual products of combustion being left in the cylinder by a premature closing of the discharge valve and compressed to 20—30 atmospheres (admission pressure), thereby producing an ignition temperature of 1,600° C. for the purpose of igniting a mixture of air and hydrocarbon highly compressed, and previously heated by the waste gases."—C. S.

*Oil or Similar Burners, Impts. in and relating to.* T. Clarkson and The Clarkson and Capel Steam Car Syndicate, Ltd., London. Eng. Pat. 22,364, Sept. 29, 1897.

The chief combinations claimed are: a vaporised fuel jet and supplementary jet of steam or air, with an "inducing tube" or mixing chamber; a regulator for automatically controlling the supply of fuel and steam or air; a concentric jet of vapour and steam or air, to deaden the reverberations of the inducing tube; a regulating needle, fitting in the vapour-jet nozzle and shaped so as to be easily removed for cleaning; the arrangement of the burner and inducing tube in such a manner that any condensed liquid trickles down into a collecting trough; an easily removable vaporiser, provided with gills to increase the heating surface, and heated up by a burner of special shape and arrangement; a shield to prevent the flame from striking back to the fuel supply of the burner; special forms of vaporiser to be worked by superheated steam; an arrangement for "regulating the temperature of the fuel after it has been vaporised by passing it through the steam generator heated by the burner."—C. S.

*Coke, Impts. in the Manufacture and Treatment of.* L. Dufrasne, The Moulinets, near Nant, France. Eng. Pat. 21,747, Sept. 22, 1897.

To increase the combustibility of coke and prevent bad smell, the inventor employs "a small quantity of a carbonate, such as, for example, carbonate of soda or of potash, or of carbonate of lime, with or without addition of carbonate of soda or common salt," either mixed with the coal previous to coking, or else dissolved in the water used for quenching the hot coke or for steeping the already cold coke.—C. S.

*Gas Producers, Impts. in.* G. R. Hislop, Paisley. Eng. Pat. 22,518, Oct. 1, 1897.

The improved gas producer consists of a brickwork chamber having a central narrow solid hearth with inclined gratings, which extend to the side walls and are supported on bearing brackets at any suitable angle, the ash and water-pan chambers, underneath the said gratings, being in communication—by means of adjustable grids in their doors—with a blower for forcing air or steam through the gratings.—C. S.

*Gas, Impts. in Apparatuses for Making.* J. Liddle, Glasgow. From L. O. Triebel and D. Whiteis, Oconto, Wis., U.S.A. Eng. Pat. 7427, March 28, 1898.

The essential features are the combination of initial and terminal gas-holders, in connection with a supply tank and regulating valve, governing the rate of admission of gas-forming material (gasoline), and a means of lifting the initial gas-holder, thus causing a vacuum, in consequence of which, air is drawn in through a perforated pipe, and becomes impregnated with the oil vapour. This lifting is effected by water pressure in a central vertical pump cylinder within the initial gas-holder, the pressure raising a piston attached to the head of the said holder. To facilitate the impregnation of the air the gasoline is fed to an annular chamber situated within the initial gas-holder. The bell of the terminal gas-holder descends as the gas is drawn off for consumption, and, in so doing, causes a ball valve to open, whereby communication is established with the initial holder, the bell of which descends by its own weight—the water pressure having been released automatically—and discharges the gas into the terminal holder until the ball valve is automatically closed by the re-ascent of the latter, the descent of the initial holder in turn reopening the valve and admitting the pressure water to actuate the pump cylinder anew. A steam pipe maintains an even temperature in the water seal of the initial gas-holder.—C. S.

*Gas, Impts. in the Manufacture of.* W. L. Wise, London. From A. Piatti and Co., Rome, Italy. Eng. Pat. 8818, April 15, 1898.

The inventor claims the substitution of bitumen or bituminous substances for colophony in the specification Eng. Pat. 2129 of 1897, in order to render the process cheaper. —C. S.

*Carbonic Acid, An Improved Process for the Manufacture of, and Apparatus therefor.* H. S. Elworthy, London. Eng. Pat. 25,007, Oct. 28, 1897

A cupola generating furnace, charged with some form of carbon, is connected below to a pipe communicating by valved passages to a series of three hot-blast stoves of the regenerative type, the central stove being a superheater. The generating furnace is also connected from its upper part to two oxidising stoves on the other side, filled with refractory material coated with a metallic oxide, such as copper oxide, the two stoves being used alternately. As the carbonic acid is to be free from nitrogen or other non-condensable gases, the apparatus, after the regenerative chambers have been highly heated and the carbon in the furnace well ignited, is swept out by a current of steam or of steam and carbonic acid. Superheated steam is then passed from the regenerators through the ignited carbon, and the mixture of carbon monoxide and hydrogen is passed into one of the two highly heated oxidising stoves, where the carbon monoxide is converted into carbonic acid, and the hydrogen into water. When the first oxidising stove loses its efficiency, the gases are passed into the second stove, and the metallic oxide in the first stove is reoxidised by passage of air through, the production of carbonic acid going on thus continuously.—E. S.

*Automatically Lighting Gas, A New Composition of Matter for, and for other Purposes.* S. Rosinski and Madame Veuve E. Ducruix (*née* M. M. Pierquin), Paris. Eng. Pat. 7280a, March 25, 1898.

Gelatinous alumina is steeped in a solution of platinum tetrachloride in four parts of alcohol, and is then dried at about 120° C. This composition becomes incandescent under the action of a jet of gas, which it ignites. Besides the application to automatic gas lighting, the composition may replace "the spongy platinum employed in apparatus for purifying the air for hygienic purposes."—E. S.

*Carbide of Magnesium, Impts. in the Manufacture of.* J. E. T. Woods and W. A. Byron, both of London. Eng. Pat. 14,780, July 5, 1898.

Carbide of magnesium is produced in a crucible or furnace fed with a mixture of fine charcoal and magnesian limestone, and supplied with gas from a gas-holder and air. The apparatus used is described in Eng. Pat. 14,453 of 1898. The gas is produced by passing crude petroleum vapour over hydrated oxide of iron heated in retorts to 1,000° F., whereby the oxide is reduced to spongy iron, which is again converted to oxide by passing steam over it. The gas formed in the reducing process consists chiefly of acetylene and marsh gas; that formed in the oxidising process, of acetylene, marsh gas, and hydrogen. All the gas formed is conveyed to the same gas-holder and used for the production of the carbide.—J. A. B.

*Calcium Carbide, Impts. in and connected with the Preparation of, for Use in the Production of Acetylene Gas.* W. J. H. Lazarus, Duren, Germany. Eng. Pat. 27,639, Nov. 24, 1897.

Calcium carbide is impregnated with paraffin or other indifferent cementing material, and is then pulverised, and subsequently compressed into cartridges. The carbide in this form is stated to be impervious to atmospheric moisture, but to be attacked when placed in water.—J. A. B.

*Calcium Carbide, Impts. in the Treatment of, whereby the Conversion of same into Acetylene Gas may be retarded without the Aid of any Mechanical Devices.* G. Dillberg, Sydney, N.S.W. Eng. Pat. 15,212, July 11, 1898.

Granulated carbide is stirred into a molten or liquid plastic composition, such as a mixture of naphthalene and resin, and the composition is then placed in a mould and compressed. Hard cakes are thus formed, and may be dropped into water; the generation of gas will then be retarded according to the quality and quantity of the retarding mixture added to the carbide.—J. A. B.

*Acetylene Generating Apparatus, Impts. in.* E. Henriquez, Brussels. Eng. Pat. 15,020, July 8, 1898.

An annular water tank is divided into two superposed compartments; a pipe provided with a valve connects the bottom of the upper compartment with the lower part of the lower compartment. The central well is closed at the top, and forms the generator, in which a basket containing carbide is placed. Water flows from the lower compartment to a pipe extending upwards in the generator and overflows on to the carbide. The parts are so proportioned that the water overflows only when the additional head of the water in the upper compartment is, by opening the valve, added to that of the water in the lower compartment. The gas generated, if not wholly consumed, forces water back into the upper compartment, and stops further production. —J. A. B.

*Acetylene Gas Generators, Impts. in.* H. Dant, Nüremberg, Germany. Eng. Pat. 16,090, July 23, 1898.

In a cistern of water is placed a reservoir closed at the top, and having near its base perforations which permit the access of water from the cistern. Attached to the top of the reservoir are several cylinders containing vessels charged with calcium carbide. The inlet pipes to the cylinders are at different levels, so that the water floods one cylinder before it enters a second. A cooler, in communication with the reservoir, is placed above the latter.—J. A. B.

*Acetylene or other Gases, An Improved Apparatus for Generating and Supplying.* E. Barnard, Christchurch, Hants. Eng. Pat. 19,319, Aug. 20, 1897.

A reservoir of carbide is supported by the crown of the bell of a gas-holder. A rod, passing through the bottom of the reservoir, strikes the bottom of the gas-holder tank when the bell descends, and causes carbide to be expelled from the reservoir into a basket, which, at the position in which the bell is, when the carbide is expelled, is slightly above the level of the water in the tank. The basket is supported by the bell, and the continued descent of the latter causes it to enter the water, whereupon the carbide within it generates acetylene, which causes the bell to rise. When the bell again descends owing to consumption of the acetylene, the expulsion of carbide from the reservoir and generation of acetylene are repeated.—J. A. B.

*[Acetylene] Gas from Carbide of Calcium, Portable Apparatus for Producing.* F. Windham and E. Fry, both of London. Eng. Pat. 20,537, Sept. 7, 1897.

A tube having at its lower end a gas-tight plug, through which the gas-exit pipe passes, is divided into two chambers by a removable diaphragm, through which passes a narrower tube terminating below in a needle valve. The lower chamber contains carbide; the upper, water, which passes to the lower chamber through the needle valve, which is operated by means of a screwed cap at the upper end of the tube. In another construction the diaphragm is fixed, and access of water to the needle valve is prevented when the pressure of the generated gas causes the flexible face of a small vessel—into the interior of which the gas enters—to bulge outwards.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Producing.* E. C. Chardin, Paris. Eng. Pat. 21,372, Sept. 17, 1897.

A tubular water receiver encloses a smaller carbide receiver, from which a gas-exit pipe passes through the upper end of the water receiver. In the upper end of the latter there is an air-inlet valve, which admits air to replace the water used. The action of the apparatus is stopped by closing this air-inlet valve. The water passes through a perforation in the base of the carbide receiver, and its passage is stopped automatically when the pressure of gas in that receiver rises. The apparatus is intended for use on vehicles.—J. A. B.

*Acetylene Gas, Improved Apparatus for the Generation of.* C. J. Bailey and J. H. Nicklin, both of Manchester. Eng. Pat. 22,918, Oct. 6, 1897.

A water container, to the base of which is screwed a carbide container, feeds the latter with water through a narrow pipe, the upper orifice of which is provided with a valve operated by an adjusting screw outside the container. The lower orifice of the pipe is surrounded by a perforated cylinder, and in one form of the apparatus the water passes first into a vessel within this cylinder and reaches the carbide as it overflows the vessel. The generated gas passes direct from the top of the carbide container to a burner.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* C. Guy, Ivry-la-Bataille, France. Eng. Pat. 24,707, Oct. 25, 1897.

A receptacle containing pulverulent carbide is placed above a chamber containing water. The gas which is evolved in this chamber when carbide falls into the water, inflates a flexible pouch with which the chamber is connected. The inflation of the pouch is caused, through suitable levers, to close a slide valve between the carbide receptacle and the chamber, and thereby to stop the passage of carbide to the latter. The valve is opened when the pressure of the gas diminishes, and the pouch becomes partially deflated. Safety devices and purifiers are provided.—J. A. B.

*Acetylene Gas Generators, Impts. relating to.* V. Maréchal, A. Moreau, and L. Garcin, all of Paris. Eng. Pat. 29,405, Dec. 11, 1897. (Under Internat. Convention.)

A capillary tube, through which water passes from a water receiver to a carbide receiver, is traversed by a wire which protrudes below the lower outwardly-flared extremity of the capillary tube, and prevents lime becoming deposited within and choking the latter. The water drips from the wire, and carries with it any deposit at the extremity of the tube.—J. A. B.

*Acetylene Gas, An Improved Process and Apparatus for the Production and Purification of.* H. Walker, Dublin. Eng. Pat. 1352, Feb. 22, 1898.

A MIXTURE of carbide and a solid acid, such as oxalic acid, is placed, with or without addition of chalk, in a lead-lined vessel, in which are paddles attached to a spindle, which is rotated by means of an external handle. Water is introduced through a pipe which enters near the top of the vessel. Another pipe conveys the evolved gas to the lower part of a vessel containing water, above which are supported crystals of sulphate of iron, with or without copper turnings. The gas, after passing through these crystals, is led to the bottom of a vessel containing water, through which it passes, and is then conveyed to a gasometer. The use of a solid acid in place of a fluid one, or compressed carbonic acid, is said to be less dangerous to those using the apparatus.—J. A. B.

*Acetylene Lamp, Improved.* W. P. Thompson, London. From H. Sez, St. Denis (Seine), France. Eng. Pat. 8016, April 4, 1898.

WITHIN a water reservoir is placed a vessel the upper part of which is packed with carbide and is closed at the top by a lid, through which a gas-exit pipe passes. Through the partition between the upper and lower parts of the carbide vessel passes a tube filled by a wick, and also a narrow tube terminating near the upper part of the carbide chamber. The lower part of the vessel is open at the bottom, and water from the reservoir reaches the wick, ascends the same, and attacks the carbide until the gas evolved is in excess of the demand, when the surplus gas passes down the narrow tube and drives the water away from the wick. Thereupon the evolution of gas ceases until the surplus gas has been consumed.—J. A. B.

*Acetylene Gas Generators, Impts. in.* F. A. Kieffer, Paris. Eng. Pat. 11,261, May 17, 1898. (Under Internat. Convention.)

SET in the crown of the bell of a gasholder is a hopper containing carbide, which is fed into the water in the tank of the gasholder through a conical valve operated by a rod attached to an annular float. The valve is opened when, owing to the descent of the bell, the float is supported by the water, and closed, when, owing to the rise of the bell, the float is carried by the rod.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Producing.* O. Ernst and A. Philips, both of Höchst-on-the-Maine, Germany. Eng. Pat. 12,510, June 3, 1898.

A BELL gasholder floats in the annular space between the tank walls, and a gas-collecting vessel immersed in the tank. Within the gas-collecting vessel is a generating vessel, of which the bottom is removable and accessible from the space below the bottom of the tank. When a box containing carbide is placed in the generating vessel, and the bottom is secured to the latter, a valve in a pipe at the top of the generating vessel is automatically raised, and allows water to flow from the gas-collecting vessel to the carbide. The generated gas passes through a pipe leading from the gas-collecting vessel to the gasholder bell. A valve in this pipe is closed automatically when the bell has risen nearly to its full height; the gas then accumulates in the collecting vessel until it depresses the level of the water therein, below the inlet to the generating vessel, and thus interrupts the supply of water and the generation of gas.
—J. A. B.

*Acetylene, Impts. in Apparatus for Producing.* W. P. Thompson, London. From H. Sez, St. Denis, France. Eng. Pat. 14,197, June 27, 1898.

Two generators are attached to the outside of the bell of a gasholder and move with it. Water is supplied from the gasholder tank to the generators, either separately or simultaneously, through a pipe provided with a three-way cock. The carbide is contained in the generators in superposed boxes with lateral perforations, and the water enters at the base when the bell of the gasholder descends so that the generators come below the level of the water in the gasholder tank.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* H. H. Lake, London. From La Compagnie Continentale d'Incandescence et de Chauffage, Brussels. Eng. Pat. 14,713, July 4, 1898.

A TAPPET attached to the bell of a gasholder strikes against a lever when the bell descends, and effects the discharge of the carbide contained in a small bucket into a shoot leading into a generating vessel containing water. A train of the small buckets is revolved so that each bucket is brought in turn over the shoot, and thus continuous operation of the apparatus in accordance with the movement of the bell of the gasholder is secured.—J. A. B.

*Bunsen Gas Burners [Incandescence], An Improvement in.* J. Moeller, Westminster. From O. Kern, Paris. Eng. Pat. 30,831, Dec. 30, 1897.

AN improvement on Kern's Eng. Pat. 291 of 1897 (this Journal, 1898, 566), which showed a burner-tube in the form of two truncated cones tapering towards each other. "It has been found that the efficiency of a tube is greatly enhanced by making its taper not uniform," but gently curved, "following the outline of a hyperbola, so that the interior of the tube is in the form of a hyperbolic spindle or solid of revolution formed by the revolution of a hyperbola round the axis of a tube." In this improved burner the inner perforated cylinder is surmounted by a button "of good conducting metal," instead of by a conical cup, and on the top of the button there is fixed a disc, a little smaller in diameter than the mouth of the outer cylinder, and having its edge serrated, resembling a toothed wheel. The copper loop-support for the mantle is replaced by the ordinary crutch-support. The two concentric cylinders may alternatively taper towards the top. The claims are:—"1. An incandescence gas-burner tube made in the form of a hyperbolic spindle substantially as described. 2. The combination of a tube such as is above referred to with a burner comprising no outer casing, an inner perforated screen, a button thereon, and a disc having a serrated edge . . . ."—H. B.

*Burners for Incandescent Gas Lighting, Impts. in.* H. H. Lake, London. From A. Armellini and E. Kerbs, Milan. Eng. Pat. 10,140, May 3, 1898.

IN the Bunsen burner claimed, the annular aperture at the top, at which combustion takes place, is perfectly free, instead of being covered with a grating or gauze. There is only one air-entrance, both for the interior of the gas-burner and for the space between the mantle and chimney. The air does not enter horizontally, but vertically, through an annular space between the gas-supply nozzle and the outer tube. The burner tube consists of two concentric tubes. The inner one, which is the Bunsen burner proper, rises from a point just above the gas nozzle, and the gas, on entering it, draws in one portion of the air-supply from the annular space, the second portion rising outside it to the exterior of the mantle. As to the dimensions of the tubes, "the air-conduits are so arranged, and are of such sections, that a maximum draught of air may be produced outside the incandescence mantle, without creating a difference of pressure between the interior and the exterior" of the mantle, and the "burner is constructed in such a manner as to enable the pressure produced by the current of air to be directly utilised upon the combustible mixture."—H. B.

*Burners for Gas Lighting by Incandescence, Impts. in.* F. Fritz and The Daylight Incandescent Mantle Co., Ltd., London. Eng. Pat. 12,772, June 7, 1898.

THE base of the burner, whereby it is secured to the gas-supply pipe, is provided, as usual, with a conical nozzle, having at its upper end the usual perforated plate, through which the combustible gas passes. Surrounding the nozzle is a short tube, contracted above to a hollow truncated cone and provided with small lateral air-inlets. An outer cone or bell surrounds and surmounts the upper part of the truncated cone, "so as to leave an annular space of suitable dimensions" between the latter and the lower part of the former. The outer cone is surmounted by the usual burner-tube. By this arrangement the air is admitted to the gas

in two instalments—through the air-inlets and through the annular space. There are claimed the use of the hollow truncated cone and the Bunsen burner as described.
—H. B.

*Incandescent Gas Burners, Impts. in or relating to.* A. J. Boult, London. From G. Deliu, Brussels. Eng. Pat. 14,454, June 30, 1898.

The burner is intended for use without a chimney. It consists of a Bunsen tube or mixing chamber, having adjustable air-openings near its base, and closed at the bottom by means of an arched plate. The gas-supply jet does not enter the tube, but is fixed concentrically a short distance below the arched bottom, and the latter has a central perforation of greater diameter than that of the gas jet. The gas, on entering the mixing chamber, is stated to produce a rotary motion, and hence a thorough mixing of the gas and air. At about two-thirds of the distance up from the bottom of the mixing chamber there is fixed a horizontal screen of gauze or perforated metal, and a similar screen covers the mouth of the burner. The mantle, suspended from a wire, rests on a funnel-shaped ring surrounding the burner mouth. The claims are for the complete burner and the arrangement of the gas jet relatively to the mixing chamber as described.
—H. B.

*Mantles for Use in Incandescent Gas Lighting, Impts. in or relating to.* A. F. B. Gomess, South Kensington. Eng. Pat. 22,327, Sept. 29, 1897.

A process for "stiffening" mantles. According to one way of carrying out the invention, the burnt-off mantle is coated with a weak solution of gutta-percha, india-rubber, or similar gums, and over this coating collodion is applied. According to the second, and more preferable method, the finished mantle (*i.e.*, after it has been stiffened as at present) is treated by applying the aforesaid coating over the collodion. The use is disclaimed of gutta-percha alone or of collodion alone (see Eng. Pat. 11,195 of 1887, this Journal, 1888, 617), the use of the combination only being claimed.—H. B.

*Mantles for Incandescent Lights, Impts. in and connected with.* E. Skriwan, Vienna. Eng. Pat. 23,287, Oct. 11, 1897.

Claim :—"In and connected with mantles for incandescent lights, forming the thread by which the mantle is suspended with a metal core [metal wire, such as iron, nickel, copper, silver, and their alloys] covered with asbestos."—H. B.

*Incandescence Bodies for Illuminating Purposes, Improved Manufacture of.* A. J. Boult, London. From Chemische Fabrik von Max Jasper, Bernau, near Berlin. Eng. Pat. 30,145, Dec. 20, 1897.

The claims cover the manufacture of an incandescence body by "immersing an incandescence body, either in a burnt or unburnt condition, in a solution containing an alcoholic and an ether organic compound of silicon, and subjecting the body, after immersion, to heat, so as to form silicon dioxide." For the organic silicon compound "compounds with esters and alkyls are preferable. Alkyls of silicon are, however, less suitable than silicic esters produced by the action of silico-chloroform ($SiHCl_3$) on alcohols, or by the treatment of methyl, ethyl, amyl, or other similar alcohols with silicon chloride ($SiCl_4$)." -H. B.

*Incandescent Illuminating Bodies, Process for Manufacturing Durable.* A. Koch, Bernau, Prussia. Eng. Pat. 13,504, June 17, 1898.

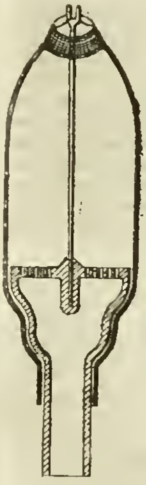
There is claimed a process for manufacturing incandescence bodies by combining with the method described in Eng. Pat. 30,145 of 1897 (see previous abstract) a "preparatory process," consisting of making a thoria mantle. This mantle, either before or after burning off, is then treated, according to the patent referred to, with organic compounds of silicon and an alcoholic cerium solution. There is also claimed, "in connection with the manufacture of incandescent bodies in general, a preparatory process," consisting of making a thoria mantle.—H. B.

*Mantles for Incandescence Gas Lights, Process for the Manufacture of Self-igniting.* L. Stoecklin, Mülhausen, Alsace. Eng. Pat. 6860, March 21, 1898.

The impregnated mantle, before burning off, is dipped into an alkaline bath (*e.g.*, ammonia, caustic soda), and is then washed till free from the alkaline salts produced, and dried. The head of the mantle is then dipped into a concentrated platinum or iridium solution, but only so far that the solution is absorbed up to the seam, and it is at once dipped into ammonia to precipitate the Pt or Ir and prevent its soaking up too far. After washing and drying, the head of the mantle may be slightly impregnated with paraffin to prevent the loss of Pt or Ir, or the mantle may be burned off and stiffened as usual. The process is claimed.—H. B.

*Incandescent Bodies for Gas Lighting, An Improved Method of, and Apparatus for Forming.* W. H. A. Sieverts, Hamburg-Uhlenhorst. Eng. Pat. 12,356, June 1, 1898.

"The object of this improved method is, in the first place, to form a firm and foldless shield or ring at the apex of the incandescent body." The burner, carrying the mantle to be treated (which has been burned off), is rotated while a jet of oxy-coal gas- or other suitable hot flame is applied externally to the apex of the mantle. In this way a strong ring is formed and the folds produced by the asbestos thread disappear. The shaping is best effected when the burner carrying the mantle is itself burning. The method is also used for treating the lower edge of the mantle ; the inwardly directed jet flame causes the mantle to soften and contract, so that it clings closely to the burner head, and then appears as in the illustration.



The method of applying the jet flame to the rotating body is claimed, and there is described and claimed an apparatus for rotating the mantle, consisting of an upright fixed Bunsen tube, surrounded by a socket which may be rotated by a rope pulley or toothed-wheel gearings. The burner carrying the mantle is slipped down over the Bunsen tube until it rests upon a plate at the top of the socket, so that when the socket rotates the burner turns with it.
—H. B.

*Incandescence Oil Lamp, Improved.* K. Reichel, St. Petersburg. Eng. Pat. 11,444, May 19, 1898.

In a circular-wick lamp, provided with the usual three air-supplies, there is placed within the annular space formed by the outer and inner wick-tubes a long sliding tube, secured to the wick at its upper end by means of projections, so that as the sliding tube is raised or lowered, the wick accompanies it. The deflector or baffle is attached to a long spindle passing axially down the inner wick tube and connected with a ratchet and pinion, so that the baffle may be raised or lowered. By means of a nut at the lower end of the spindle and a plate projecting from the above-mentioned sliding tube, the latter is so connected with the spindle that it necessarily follows the upward motion of the same, whereas when the spindle is racked downwards, the sliding tube does not accompany it, but can only be moved down by pressure upon a separate rod. The baffle, which serves as mixing chamber, consists of "an adjustable broad ring and of an adjustable slightly curved plate placed over the said ring, leaving an annular aperture between the two."

When the lamp is to be lit, the pinion wheel is turned so as to screw up the baffle some distance above the burner top, at the same time raising the wick. The plate which projects from the sliding tube, stops the ascent of the wick at just the correct level for use. The wick is lighted, the baffle is screwed down so that it may get heated, and is ultimately screwed down till close to the wick, when the luminous flame changes to the Bunsen flame, and, burning within the baffle, issues through the annular aperture against the mantle. The above arrangement of parts and the complete lamp are claimed.—H. B.

*Incandescent Oil Lamps, Impts. in.* L. R. A. Morel, Paris. Eng. Pat. 21,202, Sept. 15, 1897. Under Internat. Conv., July 28, 1897.

A PATENT referring to modified constructions of burner tops, of which four are illustrated. The claim is for "an incandescent hydrocarbon lamp burner provided with means whereby air is admitted or restored to the flame in such manner as to prevent the flame firing back to the wick, and in particular, with means consisting essentially of two or more deflectors [*i.e.*, caps surrounding the wick-tubes], and, intermediate annular air spaces (with or without intermediate admission of air thereto), so disposed (concentrically) as to direct air currents on to the outer surface and upper edge of the wick, one of the air currents being vertical and the other sensibly horizontal."—H. B.

*Electric Furnaces [Carbides, &c.]; Impts. in and relating to.* G. L. Palmer and E. Williams, Trowbridge, Wilts. Eng. Pat. 16,029, July 22, 1898.

CLAIM 1 is for "the combination with an electric furnace" (shown in box form, mounted on wheels running on rails) "of a feed box or vessel containing the charge" (which can be lowered on to the open furnace below, and so receive heat therefrom), "adapted to be heated by the heat generated in the furnace, during the treatment of a previous charge," &c. "3. The improved process for manufacturing calcium carbide, or for smelting ores, reducing oxides, and like purposes by electricity, consisting essentially in utilising the heat generated in the furnace, during one final heating operation for giving preliminary heat to the charge, which is about to be introduced into the furnace for the next final heating operation, by means of apparatus, such as shown by the drawings."—J. C. R.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Coke Ovens, and Ammonia as By-Product.* B. Terne. J. Franklin. Inst. **146**, [2], 127—137.

THE production of coke with the recovery of by-products has been instituted by two large companies in the United States. The first ovens of the Otto-Hoffman system were constructed in 1895, in the coke department of the Cambria Iron Company, Johnstown, Pa., and very satisfactory results have been reported. The Semet-Solvay system has taken a firm foothold in the United States; there being in operation at the present time 25 ovens at Syracuse, N.Y.; 25 at Sharon, Pa., and 50 at Dunbar, Pa.; in course of erection, 120 ovens at Birmingham, Ala., 120 at Wheeling, W. Va., and 10 ovens at Halifax, N.S. In reality, the Solvay Company has been the pioneer of this industry, the first ovens having been built in 1892. There are about 45,000 beehive ovens in existence in the United States, and although their abolition means the annihilation of a very large capital, it is simply a matter of calculation, whether the interest on the capital required for building new ovens, is greater or less than the profits corresponding to the increased yield of coke, and represented by the value of the by-products. The following table, taken from Mineral Industry, 1895, Vol. IV., 242, gives the comparative costs between the old and new processes:—

| Items. | | Kind of Oven. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Not recovering By-Products. | | | | Recovering By-Products. | | | | | |
| | | Beehive. | Belgian. | Bernard. | Coppée. | Otto-Hoffman. | Simon-carvès. | Huessner | Semet-Solvay. | Festner-Hoffman. | Siebel. |
| Cost per oven | Dols. | 300 | 900 | 1,000 | 1,000 | 4,100 | 2,400 | 2,750 | 3,000 | 3,000 | 2,500 |
| Output of coke per day per oven | Net Tons. | 1·75 | 2·40 | 2·25 | 2·20 | 3·34 | 2·2 | 2·9 | 4·0 | 2·94 | 3·10 |
| Yield of coke | Per cent. | 65 | 68 | 75 | 72 | 75 | 75 | 75 | 75 | 75 | 75 |
| Number of ovens required to produce 100 tons of coke per day. | | 58 | 42 | 44 | 46 | 30 | 46 | 35 | 25 | 34 | 33 |
| Cost of plant to produce 100 tons of coke per day | Dols. | 17,400 | 37,800 | 44,000 | 46,000 | 121,800 | 110,400 | 96,250 | 75,000 | 102,000 | 82,500 |
| Repairs and depreciation per ton of coke | " | 0·047 | 0·102 | 0·119 | 0·124 | 0·337 | 0·298 | 0·261 | 0·203 | 0·275 | 0·228 |
| Labour on coke and by-products per ton of coke | " | 0·320 | 0·270 | 0·250 | 0·270 | 0·390 | 0·390 | 0·390 | 0·390 | 0·390 | 0·390 |
| Total cost per ton of coke | " | 0·367 | 0·372 | 0·369 | 0·394 | 0·727 | 0·688 | 0·654 | 0·593 | 0·665 | 0·613 |
| Value of by-products per ton of coke | " | .. | .. | .. | .. | 0·530 | 0·530 | 0·530 | 0·530 | 0·530 | 0·530 |
| Net cost per ton of coke | " | 0·367 | 0·372 | 0·369 | 0·394 | 0·197 | 0·158 | 0·124 | 0·063 | 0·135 | 0·083 |

From the yields of coke from the different ovens shown in above table, it will appear that the beehive requires 1·54 tons of coal to make a ton of coke, whilst the ovens saving by-products require 1·33 tons. The value of disposable gas is not included in the figures given, depending upon local conditions, but under any circumstances it should not be less than 20 cents per ton of coke. Adding this value to that of the other by-products, it is safe to say that the cost of coke in the by-product oven does not exceed the cost of the coal from which it is made.

The 45,000 beehive ovens have a capacity of 78,750 tons per day, at a cost of 28,350 dols. per day, whilst the same quantity in the improved Otto oven would cost 10,237 dols. per day, or in the Semet-Solvay system, 4,725 dols. The conclusion of the officials of the Cambria Iron Works, respecting the comparative working of different retort ovens is, that the Semet-Solvay system is 30 per cent. quicker in operation than any of its competitors. The Semet-Solvay Company of Syracuse asserts that a single oven will produce 2,000 tons of coke from Pocohontas coal, or 1,600 tons from Connellsville coal, in one year. The Pittsburg coal has given an average of from 18 to 22 lb. of sulphate of ammonia per ton of coal. Taking an average of 20 lb., the daily loss by the use of the existing beehive ovens represents over 2,000 tons of sulphate of ammonia.

A most important step for another source of ammonia has been made by Mond, who found that nearly 100 lb. of sulphate of ammonia could be recovered per ton of fuel from the producer gases, by introducing together with the superheated air required to burn the fuel in the producer, $2\frac{1}{2}$ tons of steam to every ton of fuel consumed. The Solvay Process Company of Syracuse has erected a plant of eight Mond producers at its works, replacing the use of coal in the steam plant and elsewhere. The production of fuel gas with the recovery of ammonia, is said to yield a return of 25 per cent. profit on the capital invested.

A comparatively new source for ammonia, at present exclusively for the benefit of the farmer, has been created by the utilisation of garbage, producing a fertiliser. The processes may in the main be classified as three distinct methods:—(1) The extraction of the grease by hydrocarbons or the naphtha process. (2) The acid process. Dissolving the crude garbage in sulphuric acid, and, after the removal of the grease, transforming the whole mass at once into "superphosphate." (3) The steam-rendering process, commonly known as the Arnold process. The extraction process by naphtha has proved to be unprofitable, owing to the high price of the material and the unavoidable losses. The cities of Buffalo, Detroit, Milwaukee, and St. Louis are working under the Merz naphtha system, but their methods have been so modified that in reality they constitute a steam-rendering system. The most extensive plant is that at the works of the New York Company situated at Barren Island, in New York Bay. The daily accumulation of garbage of the two cities is varying form 1,000 to 1,500 tons, depending upon the season of the year. In New York the gathering is done by the street-cleaning department; in Brooklyn, by contract. In both places the material is loaded on scows, which are towed down to the island. The material is taken by a conveyor to the top of the digester room, where it is distributed to the digesters by means of swivel spouts. A digester is filled with a 9-ton charge in five minutes. Powerful condensers have been erected for the escaping gases from the digesters and the dryers. The liquors from the digesters, which were formerly allowed to go to waste, are now passed through a 150,000-gallon triple-effect evaporator, built by the Sugar Apparatus Company of Philadelphia, under the Lillie patents. Utilising the garbage of the two cities, 2,400,000 lb. of ammonia are added annually to the gain of ammonia in form of fertiliser available to the soil.—D. B.

*Petroleum Industry of Galicia, The.* —Kouindjy. Bull. Soc. Enc. **3**, [7], 870—879.

The article is a *résumé* of a paper by N. J. Pantukoff, published in the Zap. imp. russ. techn. obschtsch. 1898, [2], 1. Two of the largest refineries—those of Dzieditz and Mariampol—have recently erected plant for continuous distillation, the Groling and Estlander system being employed at the last-named works. In this apparatus the heat of the eliminated fractions is utilised for warming up the crude oil, thus facilitating the recovery of petroleum spirit in the first stages of the process. The steam superheater, being situated in the distilling apparatus, enables the temperature of the steam to be raised by 20°. The fractions between 0·750 and 0·880 are classified as petroleum, and the remainder on redistillation yields 10 per cent. of petroleum spirit, 10 per cent. of petroleum, 30 per cent. of merchantable oil (burning oil), and 50 per cent. of lubricating oil.

The commercial brands of oil are: Salon oil, sp. gr. 0·800—0·805; standard oil, 0·805—0·815; burning oil, 0·820—0·830; and seven marks of lubricating oil, ranging in viscosity between 4·4 and 30, flashing point 137°—200°, and sp. gr. 0·8335—0·915.

Working with the Zaloziecki method, the author (Pantukoff) determined the percentage of paraffin in petroleum "goudron" from Romaninsk, and obtained the following results:—

| Ethyl-amyl Alcohol Mixture. | Paraffin. | |
|---|---|---|
| Volume. | Per Cent. | Melting Point. |
| 10 : 10 | 14·3 | 30°—35° C. |
| 7·5 : 10 | 4·9 | 35°—40° C. |
| 5 : 10 | 2·1 | 45°—50° C. |

At Mariampol the paraffin in the residues is recovered by artificial refrigeration; but in other works the residues are stored in barrels embedded in earth until the winter season, when the necessary cooling is effected by the natural temperature of the air. At Lipinsky the residue is placed in iron tanks, the paraffin being skimmed off as it solidifies on the surface.—C. S.

*Petroleum Oils, Polarisation Values of.* P. Soltsien. Zeits. für öffentl. Chem. **4**, 464; Chem. Centr. 1898, **2**, [6], 455. (See also this Journal, 1898, 569.)

Of two samples of vaselin oil from a naphtha of Romany (Baku), one, a yellowish oil with blue fluorescence, sp. gr. at 15° C., 0·8675, showed a rotation of +1° 1′ in a 200-mm. tube; whilst the second, a white oil with blue fluorescence, sp. gr. at 15° C., 0·862, gave a rotation of +1° 6′.—A. S.

*Phenols, Action of Bromine upon, in the Presence of Aluminium Bromide.* F. Bodrouse. Bull. Soc. Chim. 1898, **19**, [16—17], 756.

*See under* XXIV., *page* 963.

*Mineral Oils: Use of Acetylene in Flashing-Point Tests.* P. Wolff. Chem. Rev. Fett- u. Harz-Ind. **5**, [7], 139.

*See under* XXIII., *page* 949.

## IV.—COLOURING MATTERS AND DYES.

*Myrticolorin: The Yellow Dye of Eucalyptus Leaves.* Henry G. Smith. J. Chem. Soc. 1898, **73**, 697—701.

The leaves of *Eucalyptus macrorhyncha*, the "red stringy bark" of New South Wales, and the ordinary "string bark tree" of Victoria (Australia), contain, under favourable conditions, 10 per cent. of a yellow colouring matter, to which the author gives the name Myrticolorin, and which is readily extracted. When purified by recrystallisation and extraction with ether, it is of a pale yellow colour, almost insoluble in cold, and not readily in hot water. It dissolves in alkalis with a yellow colour, and gives yellow and green colorations with lead acetate and ferric chloride respectively. It softens at 179°, and melts at 185°. When dried at 130°, it possesses the formula $C_{27}H_{28}O_{16}$. When boiled with dilute sulphuric acid, it is hydrolysed, quercetin and a glucose being produced. The latter was a syrup which reduced Fehling's solution, was readily decomposed by yeast and yielded an osazone which softened at 170° and melted with slight decomposition at 180° when slowly heated, but which melted with rapid decomposition at 190° when quickly heated. This behaviour is the same as that of the osazone of galactose. Myrticolorin is therefore a new glucoside of quercetin, intermediate between osyritrin ($C_{27}H_{30}O_{17}$) and viola quercitrin, $C_{27}H_{26}O_{15}$.

In colour, melting point, reactions with reagents, and dyeing power, no difference could be detected between Osyritrin and Myrticolorin.—T. E.

*Dimethylaniline, Formation of, by the Action of Brom- or Iod-Benzene and Phenol on Dimethylamine.* B. N. Menschutkin. J. russ. phys.-chem. Ges. **30**, 243—251; Chem. Centr. 1898, **2**, [7], 478.

1. *Action of Brom- and Iod-Benzene on Dimethylamine.*—As a consequence of the reaction discovered by Lellman (this Journal, 1887, 605) between iod- or brom-benzene and piperidine, the author sought to carry out Hoffmann's amine synthesis with aromatic compounds. He worked with dimethylamine, because, like piperidine, it is one of the strongest organic bases. The following method was most successful:—1 part of iodide or bromide and 2—3 parts of dimethylamine were heated in a short closed tube in L. Meyer's air bath for 5—6 days at 250°—260° C., about 15 per cent. of the iodide entering into the reaction. In the end-product, after removal of the dimethylamine and separation of the iodide or bromide by water, &c., the dimethylaniline salt was identified by its melting point, solidifying point, iodine or bromine content, and by analysis and qualitative reactions.

2. *Action of Phenol on Dimethylamine.*—Dimethylaniline was formed by heating one part of phenol with one part of dimethylamine for 60—84 hours at 250° C. It was observed that after expelling at 60° C. the dimethylamine from the products of reaction, a further quantity could be separated by the addition of caustic soda. This is ascribed to the formation of a hitherto unknown phenolate of dimethylamine, $(CH_3)_2NH.C_6H_5OH$, which the author proposes to investigate more closely.

In view of the high price of dimethylamine, the author recommends the following improved method of preparation:—A solution of 100 grms. of caustic soda in 4 litres of water is heated nearly to boiling in a 6—7 litre flask, and 75 grms. of pulverised nitrosodimethylaniline chloride added, together with some zinc shavings. The flask is provided with an inverted condenser, at the end of which is fitted a Péligot's U-tube containing 75—100 c.c. of concentrated hydrochloric acid. The amine formed is set free by the caustic soda, and is then absorbed by the acid. The yield of the dimethylamine salt is nearly theoretical. The salt is mixed with powdered quicklime, the mixture being followed by pieces of quicklime, in a tube 60 cm. long, closed at one end, which is then heated in an ordinary combustion furnace, the dimethylamine being collected in a receiver cooled by ice.—A. S.

*Benzidine Dyestuffs in the Printing Industry, Application of, during the last Fifteen Years.* G. Stein. Zeits. angew. Chem. 1898, [39], 897.

*See under* VI., *page* 920.

*Coal-Tar Dyestuffs, Detection of Arsenic in; Use of the Microbiological Reaction for the.* G. Morpurgo and A. Brunner. Oesterr. Chem. Zeit. **1**, 167; Chem. Centr. 1898, **2**, [7], 505.

*See under* XXIII., *page* 954.

## PATENTS.

*Colouring Matters, New [Yellow, Orange, and Brown]; Production of, from Paradinitrodibenzyldisulpho Acid.* T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 18,990, Aug. 17, 1897.

*p*-Dinitrodibenzyl (dinitrodiphenylethane) disulphonic acid, or the dyestuff formed from it by the action of caustic soda solution at 80° C., reacts in caustic soda solution with the following compounds:—*o*-, *m*-, and *p*-phenylene- and tolylene diamines, benzidine, methylbenzidine, tolidine, ethoxybenzidine, dianisidine, diamidostilbene, diamidostilbene disulphonic acid, 1.4-naphthylenediamine, *o*-, *m*-, and *p*- amidophenol and -cresol, *p*-amidosalicylic acid, dehydrothio-*p*-toluidine sulphonic acid, Primulin, dehydrothio-*m*-xylidine sulphonic acid, dehydrothiopseudocumidine sulphonic acid, the sulphonic acids of the thiobenzenylic bases, which are obtained by the action of sulphur upon mixtures of aniline, *p*-toluidine, *m*-xylidine or pseudocumidine, the sulphonic acid of amidobenzenyl-*o*-amidothiophenol, and aniline and its homologues, as *o*-, *m*-, and *p*-toluidine, xylidine, cumidine, and their nitro compounds.

For example, 24 kilos. of sodium *p*-dinitrodibenzyl disulphonate and 11 kilos. of *p*-phenylenediamine are dissolved in 300 litres of boiling water and are treated with 50 kilos. of caustic soda solution at 50° B. A thick brown paste is thus formed, which is neutralised with hydrochloric acid, and the precipitate is filter-pressed and dried.

The compounds produced, dye unmordanted cotton yellow, orange and brown. On oxidation in alkaline solution, they are changed into dyestuffs of more yellow or greenish-yellow shades. A similar result is obtained also, in the case of the dyestuff formed by the action of caustic soda at 80° C. on sodium dinitrodibenzyl disulphonate.

The compounds derived from *p*-amidophenol give, on alkylation or benzylation, dyestuffs which are fast to alkalis.—E. B.

*Colouring Matters [Blue, Bluish-Green], Production of.* I. Levinstein and Levinstein Ltd., Manchester. Eng. Pat. 19,904, Aug. 30, 1897.

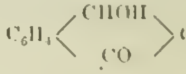
This is supplementary to Eng. Pat. 18,376 of 1897 (this Journal, 1898, 754), and describes the preparation of *p*-nitrobenzaldehyde-*o*-sulphonic acid by oxidising *p*-dinitrostilbene disulphonic acid, and the formation of dyestuffs from the former. The oxidation of the dinitrostilbene disulphonic acid is carried out by dissolving 12 kilos. of the sodium salt in 250 litres of water, and running in a solution of 5 kilos. of potassium permanganate with vigorous agitation at about 5—10° C. The liquid is then boiled, filtered from manganese dioxide, and the filtrate neutralised with acid and evaporated to dryness. The sodium *p*-nitrobenzaldehyde *o*-sulphonate forms a yellow crystalline powder very soluble in water. For most technical purposes, however, it is not necessary to separate the substance in a solid state, as the solution may be directly condensed with alkylated aromatic amines. For the preparation of a dyestuff, 5 kilos. of the sodium salt, dissolved in 50 litres of water and 11·5 kilos. of ethylbenzylaniline sulphonic acid, slightly acidified with sulphuric acid, are cohobated for 12 hours. The mixture is then diluted with several times its volume of water, sulphuric acid is added, and the leuco base is oxidised with the theoretical quantity of lead peroxide. After filtering from lead sulphate, the filtrate is made alkaline with ammonia and evaporated until the dyestuff separates out, which is then filter-pressed and dried. It forms a bluish-green powder, with a coppery lustre, is easily soluble in water, and dyes animal fibres from an acid bath a bluish-green shade fast to alkalis.

—T. A. L.

*Colouring Matters (Rhodamines) and Intermediate Products; Impts. in and relating to the Production of.* G. B. Ellis, London. From La Société Chimique des Usines du Rhône, anciennement Gilliard P. Monnet et Cartier, Lyons, France. Eng. Pat. 19,785, Aug. 27, 1897.

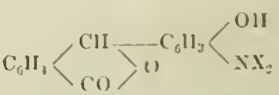
Phthalaldehydic acid condenses, in most cases unaided by condensing agents, with *m*-amidophenols and their alkyl derivatives.

At a low temperature the aldehyde reacts in its labile form—



$$C_6H_4\left\langle\begin{matrix}CHOH\\ CO\end{matrix}\right\rangle O$$

yielding compounds of the formula—



$$C_6H_4\left\langle\begin{matrix}CH - C_6H_3\left\langle\begin{matrix}OH\\ NX_2\end{matrix}\right.\\ CO\end{matrix}\right\rangle O$$

which, at higher temperatures, condense with phenols and amines, giving leuco compounds of colouring matters.

A trimethyl-Rhodamine dyestuff is thus obtained:—4·6 kilos. of methyl-*m*-amidophenol are dissolved in ether, 5 kilos. of phthalaldehydic acid are added, and the mixture is boiled under a reflux condenser for some hours. After evaporating off the ether, the phthalide is obtained as a bright reddish mass, and may be purified by washing with warm water and crystallising from ether. It melts at about 184 C. 9 kilos. of this compound are heated with 20 kilos. of dimethyl-*m*-amidophenol at 110° C. The product is then oxidised and purified by known methods.—E. B.

*Colouring Matters, New, related to the Rhodamine Series; The Manufacture and Production of.* J. Y. Johnson, London. From The Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 19,994, Aug. 31, 1897.

Benzyl derivatives of benzo-rhodamine dyestuffs, containing at least one sulphonic acid group in the benzyl group or groups, are produced by condensing benzaldehyde (or its chloro-, amido, methyl-, hydroxy-, alkoxy-, or sulphonic acid derivatives) with benzylated *m*-amidophenols, converting the products by dehydration into pyrone derivatives, and oxidising, and if necessary, sulphonating. The following is an example of the process:—About 208 parts of sodium benzaldehyde *m*-sulphonate are dissolved in about 2,000 parts of dilute sulphuric acid (10 per cent.) and mixed with about 160 parts of ethylbenzyl-*m*-amidophenol dissolved in about 1,000 parts of spirit. The mixture is heated for 10—12 hours on the water-bath. The product, on cooling, separates out, and is purified by recrystallisation from alcohol; 10 parts of it are heated with 30 parts of concentrated sulphuric acid at 130° C. The melt is poured on ice, and the precipitated leuco compound filtered, pressed, dissolved in dilute sodium carbonate solution, and oxidised by ferric chloride and hydrochloric acid. The colouring matter so produced is then converted into its sodium salt. It dyes wool and silk from an acid bath bluish-red.

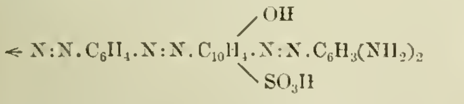
Colouring matters derived from the mixed leuco compounds resulting from the condensation of 1 mol. of an aldehyde derivative, 1 mol. of a benzylated and 1 mol. of an unbenzylated *m*-amidophenol, are also claimed.—E. B.

*Polyazo Colouring Matters or Dyes [Black], Manufacture of New.* T. R. Shillito, London. From J. R. Geigy and Co., Basle, Switzerland. Eng. Pat. 20,278, Sept. 3, 1897.

The dyestuffs referred to are obtained by combining a tetrazo-azo compound of the general formula—

$$\leftarrow N{:}N.P.N{:}N.R.N{:}N \rightarrow$$

in an acid solution, with one molecular proportion of a *m*-diamine, *m*-amidophenol, resorcinol, *m*-phenylene or *m*-tolylene diamine sulphonic acid, or a chrysoidine, and further combining the intermediate compound so formed with an amine, phenol, or derivative thereof. In the above formula P represents benzidine, methylbenzidine, tolidine, ethoxybenzidine, diamidodiphenol ether, diamidostilbene, *p*-phenylene or *p*-tolylenediamine, *p*-diamidodiphenylamine, *p*-diamidophenyltolylamine, *p*-diamidoditolylamine, 1.4 or 1.5-naphthylene diamine or their sulphonic acids, whilst R signifies α-naphthylamine, 1.3′- or 1.2′-naphthylamine sulphonic acid, 1.2-amidonaphthol ether, 1.2.3′- or 1.2.2′-amidonaphthol ether sulphonic acid, 2.1.3′- or 2.4′.2′-amidonaphthol sulphonic acid, 2.1′.3.3′- or 1.1′.3.3′-amidonaphthol disulphonic acid, *p*-xylidine, or amidocresol ether. For example, 15 kilos. of *p*-amido-acetanilide are diazotised with hydrochloric acid and 7 kilos of sodium nitrite, and combined in an alkaline solution with 24 kilos. of 2.4′.3′-amidonaphthol sulphonic acid. The dyestuff is filtered off, dissolved in 500 litres of boiling water, 150 kilos. of caustic soda (40 B.) added, and the whole boiled for about 2 hours. When cold, 500 kilos. of hydrochloric acid (22 per cent.) are added, together with 500 kilos. of ice, and the whole is diazotised with 14 kilos. of sodium nitrite. The tetrazo-azo compound thus formed is mixed with a solution of 11 kilos. of *m*-phenylene diamine. The resulting black disazo-disazo compound has the formula—



$$\leftarrow N{:}N.C_6H_4.N{:}N.C_{10}H_4\begin{matrix}\diagup OH\\ \\ \diagdown SO_3H\end{matrix}.N{:}N.C_6H_3(NH_2)_2$$

This is then added to a solution of 24 kilos. of 2.1′.3′-amidonaphthol sulphonic acid dissolved in 500 litres of water together with 80 kilos. of sodium carbonate. After standing some hours, the whole is heated to about 80° C., when the dyestuff is filtered off and dried It gives deep bluish-black shades on unmordanted cotton.—T. A. L.

*Dyes (Oxazines), Impts. in the Production of.* E. S. Wilson, Strood, and E. Stewart, London. Eng. Pat. 20,410, Sept. 6, 1897.

The invention consists essentially in heating the colouring matter contained in crude cotton-seed oil with nitroso-dimethyl or -diethylaniline and an alkali. Various ways of doing this are described. Brown or red-brown colouring matters are obtained.—E. B.

*Colouring Matters (Yellow, Orange, Red, Brown, and Black) for Cotton, Impts. in the Preparation of.* A. G. Green, Heaton Moor, and A. R. Wahl, Clayton. Eng. Pat. 21,399, Sept. 17, 1897.

Dinitrodiphenylethylene disulphonic acid—

$$[NO_2{:}SO_3H{:}CH = 4:2:1]$$

(Eng. Pat. 5351 of 1897, this Journal, 1898, 148) yields colouring matters, some of which are new, and some possibly identical with colouring matters already obtained in other ways: (*a*) when it is acted upon in dilute alkaline solution by weak reducing agents (zinc dust, ferrous hydrate, sodium stannite, glucose, &c.); (*b*) when condensed with phenols or amines; (*c*) when the compounds obtained according to the latter method (*b*) are treated with weak reducing agents, as in the first method (*a*) (the operations of condensation and reduction may be performed simultaneously); and (*d*) when such of the products as contain free amido groups are diazotised, either on the fibre or in substance, and combined with phenols or amines.

As an example of the first method, 50 lb. of sodium dinitrodiphenylethylene disulphonate are dissolved in 25 galls. of water, 5 galls. of caustic soda solution (30 per cent. NaOH), and 15 galls. of a 10 per cent. solution of glucose are added, and the mixture is heated at 70° C. for 3—4 hours. The excess of alkali is neutralised and the precipitated dyestuff is filtered off and dried.

As an example of the second method, a mixture of 100 lb. of sodium dinitrodiphenylethylene disulphonate, 70 lb. of sodium dehydrothiotoluidine sulphonate, 8 galls. of caustic soda (30 per cent. NaOH), and 100 galls. of water, is heated at 90°—100° C. for about 12 hours. The colouring matter separates out and is filtered off and dried. It dyes a bright yellow on unmordanted cotton.—E. B.

*Colouring Matters [Yellow, Orange, Red, &c.] for Cotton, Impts. in the Production of.* A. G. Green, Heaton Moor, and A. R. Wahl, Clayton. Eng. Pat. 21,553, Sept. 20, 1897.

Dinitrodiphenylethane disulphonic acid—

$$[NO_2 : SO_3H : CH_2 = 4 : 2 : 1]$$

(Eng. Pat. 5351 of 1897; this Journal, 1898, 148), like dinitrodiphenylethylene disulphonic acid (Eng. Pat. 21,399 of 1897; see preceding abstract), yields colouring matters when reduced in the presence of alkalis, condensed, &c.

As an example of one method (*a*):—50 lb. of sodium dinitrodiphenylethane disulphonate, dissolved in 100 galls. of water, are heated at 80° C. for several hours with 18 galls. of a 10 per cent. solution of glucose and 5 galls. of caustic soda solution containing 30 per cent. of NaOH. The excess of soda is neutralised and the colouring matter filtered off and dried. It dyes cotton yellow-orange. By employing a larger quantity of glucose than that given, colouring matters are formed which dye cotton red-orange.

As an example of another method (*b*):—47 lb. of sodium dinitrodiphenylethane disulphonate, 15 lb. of α-naphthol, 5 galls. of caustic soda solution (30 per cent. of NaOH), and 100 galls. of water, are boiled together several hours. The product dyes cotton brown-orange. It may be diazotised and combined with the usual azo-dyestuff components, giving, on combination, compounds of the following colours:—with β-naphthol, red; resorcinol, orange; *m*-tolylenediamine, brown; β-naphthol disulphonic acid R, maroon; α-naphthylamine, dark purple.—E. B.

*Paranitrobenzaldehyde Orthosulphonic Acid and Colouring Matters [Triphenylmethane] therefrom, The Manufacture or Production of.* A. G. Green, Heaton Moor, and A. R. Wahl, Clayton. Eng. Pat. 21,825, Sept. 23, 1897.

Under the influence of alkaline oxidising agents, *p*-nitrotoluene *o*-sulphonic acid is converted successively into dinitrodiphenylethane disulphonic acid (Eng. Pat 5351, 1897; this Journal, 1898, 148), and dinitrodiphenylethylene disulphonic acid. The latter, on further oxidation, yields *p*-nitrobenzaldehyde *o*-sulphonic acid. For example, 20 lb. of sodium dinitrodiphenylethylene disulphonate are dissolved in 25 galls. of water. The solution is cooled to 0°—5° C. and, while it is energetically stirred, a solution of 8¾ lb. of potassium permanganate in 15 galls. of water is slowly run into it. The mixture is then gently heated and filtered from the precipitated manganese oxide. The filtrate is neutralised with hydrochloric acid and concentrated until, on cooling, the sodium salt of *p*-nitrobenzaldehyde *o*-sulphonic acid crystallises out.

*p*-Nitrobenzaldehyde *o*-sulphonic acid condenses readily with secondary and tertiary amines of the benzene series, giving leuco compounds of blue and green colouring matters of the triphenylmethane group. It also condenses with alkylated and alphylated *m*-amidophenols and -cresols giving compounds which, when treated with dehydrating agents, are converted into leuco compounds of colouring matters of the pyrone group. Thus, 12½ lb. of sodium *p*-nitrobenzaldehyde *o*-sulphonate, dissolved in 17½ galls.

of water, are mixed with 8 lb. of sulphuric acid and 16½ lb. of *m*-dimethyl amidophenol, and the mixture is boiled for several hours. The leuco compound which separates out is dried. 10 lb. of it are dissolved in 40 lb. of concentrated sulphuric acid, and heated for about half an hour at 135°—140° C., the oxidation of the leuco compound being thus effected simultaneously with its dehydration. The solution is cooled and poured into 40 galls. of cold water. Part of the colouring matter formed separates out, and the remainder is precipitated by the addition of salt. It dyes silk a bluish-red.—E. B.

*Blue Basic Safranine Azo-Dyestuffs, Manufacture of.* O. Imray, London. From The Farbwerke Meister, Lucius und Brüning, Hoechst a/M., Germany. Eng. Pat. 22,242, Sept. 28 1897.

Pure blue dyestuffs may be obtained by working according to the preceding specification (No. 21,451), but employing α-naphthylamine in place of β-naphthylamine. The dyestuffs formed are suitable for mordanted or unmordanted vegetable fibres as well as for animal fibres.—T. A. L.

*Blue to Green Dyestuffs Soluble in Water, Manufacture of.* O. Imray, London. From The Farbwerke Meister, Lucius und Brüning, Hoechst a/M., Germany. Eng. Pat. 21,451, Sept. 18, 1897.

A bluish-green dyestuff for unmordanted cotton, and for cotton mordanted with tannin and tartar emetic, is obtained by the action of the diazo compound of an asymmetric dialkyl-phenosafranine on β-naphthylamine, of which the following is a typical example:—8 kilos. of diethylphenosafranine (by oxidising 1 mol. of *p*-amidodiethylaniline and 2 mols. of aniline) are dissolved in 500 litres of water and diazotised with 8 kilos. of hydrochloric acid (33 per cent.) and 1·4 kilos. of sodium nitrite, the blue diazo solution being run into 250 litres of water containing 2·8 kilos. of β-naphthylamine and 2·3 kilos. of hydrochloric acid. After some hours the dyestuff is salted out, filter-pressed, and dried. It gives bluish-green shades on unmordanted cotton, and on cotton mordanted with tannin and tartar emetic.
—T. A. L.

*Sulphinic Acids of the Aromatic Series, The Manufacture or Production of.* H. E. Newton, London. From The Farbenfabriken vorm. F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 23,047, Oct. 7, 1897.

This is supplementary to Eng. Pat. 26,139 of 1896 (this Journal, 1897, 907) in which a process was described for producing sulphinic acids of the aromatic series by acting on diazotised aromatic amines with finely-divided copper in presence of sulphurous acid. The patentees now find that, instead of metallic copper, cuprous oxide ($Cu_2O$), cuprous hydrate, the so-called red cuprous sulphite or other copper compounds containing cuprous sulphite, may be used in the above process. The diazo solution from 9·3 kilos. of aniline and 250 kilos. of 20 per cent. sulphuric acid has added to it 15 kilos. of sulphurous acid, and subsequently 15 kilos. of cuprous oxide as a paste, in small quantities, the temperature being kept below 5° C. When the evolution of nitrogen has ceased and diazobenzene is no longer to be detected, the benzene sulphinic acid is extracted from the solution with ether.—T. A. L.

*Black Disazo-colouring Matters, Manufacture of.* C. D. Abel, London. From The Actien Gesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 24,527, Oct. 22, 1897.

Valuable black diazo dyestuffs are obtained by combining the diazo derivative of a mono- or di-sulphonic acid of *p*-amidodiphenylamine or of its homologues with α-naphthylamine, rediazotising and combining with another dyestuff component, such as a mono- or di-sulphonic acid of α- or β-naphthol, or with β-naphthol when a disulphonic acid is used to commence with. The dark yellow diazo solution from 34·4 kilos. of *p*-amidodiphenylamine disulphonic acid, 6·9 kilos. of sodium nitrite and a suitable quantity of hydrochloric acid, is run into a solution of 14·3 kilos. of α-naphthylamine in hydrochloric acid, together with sufficient sodium acetate to neutralise the mineral acid. The combination is complete in a short time, and 49·8 kilos. of the intermediate product thus obtained are dissolved in sodium carbonate solution, 6·9 kilos. of sodium nitrite are added, and the mixture is run into hydrochloric acid. The reddish-violet diazo solution so obtained is added to a solution of 14·1 kilos. of β-naphthol in an equivalent quantity of soda lye, and kept alkaline with sodium carbonate. A black precipitate of the dyestuff is formed which gives a green powder with a metallic lustre, dyeing wool bluish-black from an acid bath.
—T. A. L.

*Mordant Dyes [Yellow, Orange], A Process for Obtaining New.* C. E. Gassmann, Mulhouse, Alsace, Germany. Eng. Pat. 24,644, Oct. 25, 1897.

The patentee combines diazotised nitro-*o*-toluidine—

$$(CH_3 : NH_2 : NO_2 = 1 : 2 : 5)$$

or nitro-*p*-xylidine $(CH_3 : CH_3 : NH_2 : NO_2 = 1 : 4 : 2 : 5)$ with 1·2-hydroxynaphthoic acid, with resorcylic acid—

$$(OH : CO_2H : OH = 1 : 2 : 3 \text{ or } 1 : 2 : 5),$$

pyrogallic acid, or with β-oxynaphthoic acid melting at 216° C., and obtains yellow and orange dyestuffs which dye chrome mordanted wool, or they may be printed with chromium acetate. When the dyes dissolve with difficulty, they may be rendered soluble by sulphonation with fuming sulphuric acid. The resulting products dye mordanted wool the same shades as the original unsulphonated substances.—T. A. L.

*Dyestuffs [Black], from Amidophenols, Producing.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 24,938, Oct. 27, 1897.

By acting with chloride of sulphur ($S_2Cl_2$) on the amidophenols, dyestuffs are obtained having a pronounced phenolic character, dissolving in caustic alkalis and dyeing vegetable fibres from such solutions. They also possess basic properties and dissolve in strong acids. The reaction takes place in two stages, which may be distinguished if the process be carried out in a solvent such as carbon tetrachloride. 50 kilos. of *p*-amidophenol and 150 kilos. of chloride of sulphur are heated to 70° C. under an inverted condenser for about five hours, when the temperature is raised to 190°—200° C. The melt, after cooling, is powdered, and boiled with water to remove any unaltered amidophenol hydrochloride. The residue is treated with soda lye and the resulting solution is evaporated. The product is easily soluble in water, and dyes unmordanted cotton from a bath containing salt. In a similar manner brownish-black dyestuffs are obtained from o- and *m*-amidophenol, black dyestuffs from *p*-amido-*o*-cresol, and brownish-violet from *p*-oxydiphenylamine.—T. A. L.

*Mordant Dyeing Disazo-Colouring Matters [Black], The Manufacture of.* C. D. Abel, London. From The Actien Gesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 26,419, Nov. 12, 1897.

These dyestuffs are derivatives of 1.1′.4-amidonaphthol sulphonic acid, and are obtained by combining one molecular proportion of this substance with two molecular proportions of the diazo compound of a chloro-, nitro-, or chloro-nitro derivative of *o*-amidophenol, or with one molecular proportion of one of these diazo compounds and one molecular proportion of the diazo derivative of another amido product. For example, 21·5 kilos. of 4.6-dichloro-o-amidophenol dissolved in 400 litres of water, and 22 kilos. of hydrochloric acid, are diazotised with 7 kilos. of sodium nitrite. The diazo compound, which separates for the greater part in brownish crystals, is combined with the monazo dyestuff, from 14·5 kilos. of diazotised α-naphthylamine, and 26·1 kilos. of the sodium salt of 1.1′.4-amidonaphthol sulphonic acid, the whole being kept alkaline with sodium carbonate. When all the diazo compound has been taken up, the mixture is heated to 60° C., and the dyestuff is salted out. It forms a bronzy-blackish-brown powder, soluble in water, with a violet-blue colour which turns blue on the addition of caustic soda lye. It gives violet-black shades on wool from an acid bath, and on subsequent treatment with chrome mordants, deep black shades fast to acids, alkalis, and milling.—T. A. L.

*Derivatives* [*α-Naphthoquinone*] *of the Naphthalene Series, Impts. in the Manufacture of.* C. D. Abel, London. From The Actien Gesellschaft für Anilin Fabrikation, Berlin, Germany. Eng. Pat. 26,675, Nov. 15, 1897.

The patentees find that β-naphthoquinone derivatives are converted into α-naphthoquinone derivatives by the action of concentrated sulphuric acid, and with this process may be combined the sulphonation of or the introduction of a nitro group into the α-naphthoquinone derivative. Further, it is not necessary to start with a pure β-naphthoquinone derivative, its production from 1.2.4-amidonaphthol sulphonic acid may be combined, for example, with its transformation into an α-naphthoquinone derivative and the introduction of a nitro group, as follows:—10 kilos. of 1.2.4-amidonaphthol sulphonic acid are stirred at a temperature of 15°—20° C. into 80 kilos. of a mixture of equal volumes of concentrated sulphuric and nitric acids, the temperature being kept below 50° C. for four hours. The melt is then poured on to ice, when nitro-oxy-α-naphthoquinone separates out.—T. A. L.

*Naphthacetoldisulphonic Acids, and of New Colouring Matters* [*Blue, Violet*] *derived therefrom, Manufacture of.* O. Imray, London. From The Society of Chemical Industry in Basle, Switzerland. Eng. Pat. 12,679, June 6, 1898.

As Witt and Dedichen (Ber. **29**, 2947; this Journal, 1897, 134) have pointed out, amidonaphthol, when acetylated, loses the properties of an amidonaphthol and assumes almost entirely the characteristics of a naphthol; and, according to the present specification, the same is true of certain acetylated amidonaphtholdisulphonic acids. Employing Witt's nomenclature, the naphthacetoldisulphonic acids which technically are of value are the 1.1′.3.3-′, the 1.1′.2.4-, the 1.1′.4.3′-, and the 1.4′.3.2′-naphthacetoldisulphonic acids, whilst those acids containing the acetamido group in the β position are only of secondary importance. For example, 3 kilos. of dry powdered 1.1′.3.3′-amidonaphthol sodium disulphonate, 1·5 kilos. of fused sodium acetate, 2·5 kilos. of acetic anhydride, and 2 kilos. of glacial acetic acid are cohobated until a sample dissolved in water no longer gives a colour when treated with acid, sodium nitrite, and subsequently sodium carbonate. Any diacetamidonaphtholdisulphonic acid formed simultaneously can be converted into naphthacetoldisulphonic acid by heating with alkalis or dilute acids, or even prolonged boiling with water. In order to effect this practically, the melt as above obtained, after distilling off the acetic acid, is heated for a short time to 80°—90° C. with a 5 per cent. solution of sodium carbonate. The naphthacetoldisulphonic acids so formed are very easily soluble in water, can only be separated with difficulty, and are therefore employed in solution. They combine with one molecular proportion of a diazo compound, and hence equimolecular proportions of a naphthacetoldisulphonic acid and a tetrazo derivative yield an intermediate compound capable of combining with another dyestuff component. If this latter contains a diazotisable group, the finished dyestuff can be diazotised on the fibre and further combined. It is noteworthy that the change of shade which occurs here is not towards black, as in the case of the Diamine colouring matters, but becomes a darker shade of blue. The monazo dyestuffs from the naphthacetoldisulphonic acids dye wool bluish-red to violet shades, whilst the diazo dyestuffs give violet to blue shades on unmordanted cotton, which are brighter than the analogous dyestuffs from the amidonaphthol disulphonic acids. They are fast to acids, and are not affected by dilute solutions of nitrous acid.—T. A. L.

*Yellow Sulphur-containing Dye from Sulphocyanide Salts, A Process for the Preparation of a.* A. Goldberg, Chemnitz, W. Siepermann, Elberfeld, and H. Flemming Kalk, Cologne, Germany. Eng. Pat. 16,199, July 25, 1898.

By passing chlorine, bromine, or sulphuric anhydride over a sulphocyanide salt, heated in a ball-mill to 160°—180° C., a reddish-brown product is obtained which is converted into its sodium salt, or may be decomposed with acid and forms a brownish-yellow powder which dyes cotton, linen, and paper, and is fast to light and soap. 8 kilos. of sodium sulphocyanide are heated in a ball-mill to 160°—180° C., and 3·5 kilos. of dry chlorine gas are passed in. The product of the reaction is washed with water to remove sodium chloride, and the residue is heated with 5 kilos. of caustic soda, in 50—100 litres of water, to 60° C. After cooling, the solution is filtered, and the residue is washed with 1—2 per cent. soda solution, and is the sodium compound of the yellow dye; this, on decomposition with an acid and washing, yields a brownish-yellow precipitate, which becomes darker on drying.—T. A. L.

[*Writing*] *Ink, Impts. in the Manufacture of.* H. H. Lake, London. From A. H. Spencer, Boston, U.S.A. Eng. Pat. 21,830, Sept. 23, 1897.

This specification describes the manufacture of solid tabloids that yield a writing ink on solution in water; the novelty consisting in the addition to the ordinary ingredients of an acid and a carbonate, which cause the substance to effervesce, thus dissolving more rapidly and completely, producing a homogeneous solution without the necessity for agitation, &c. A suitable formula is: "Nigrosine," 60 grains; sodium bicarbonate, 15 grains; tartaric acid, 16 grains; gum-arabic, 12 grains—to make 12 tabloids.—F. H. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Oxycellulose.* Léo Vignon. Bull. Soc. Chim. 1898, **19**, [16—17], 790—793.

Cotton wool was purified by successive treatment with boiling 1 per cent. sodium carbonate for 30 mins., boiling 1 per cent. caustic soda for 30 mins., 1 per cent. hydrochloric acid in the cold, and, finally, with cold sodium carbonate, then washed with boiling water and alcohol. The loss by this treatment was about 10 per cent. Oxycellulose was obtained from the purified cotton in the following manner:—30 grms. of cotton were immersed in a hot solution of 150 grms. of potassium chlorate in 3 litres of water, and 125 c.c. of hydrochloric acid were gradually (5 mins.) added. After heating for 1 hour, the liquid was poured off and the residue washed by decantation till all acidity was removed, then with alcohol, and, finally, it was air-dried. Oxycellulose, thus obtained, consists of very short filaments, turns yellow at 100°, and is insoluble in neutral reagents. It contains 87·6 per cent. soluble in fused potash at 180° C., whilst cellulose only contains 12 per cent. Immersed for 30 mins. in boiling solutions of Safranine and Methylene Blue, oxycellulose absorbs 0·007 and 0·006 grm. per grm., whilst cellulose absorbs 0·000 and 0·002 grm. respectively. Treated with aqueous potash, oxycellulose yields a yellow solution, which reduces Fehling's solution, and gives a white precipitate on acidifying. Schiff's reagent gives a violet coloration with oxycellulose.
—A. C. W.

*Mercerised Cotton, Microscopical Appearance of.* H. Lange. Färber-Zeit. 1898, **9**, [15], 234—235. (See this Journal, 1898, 839.)

Many of the cotton fibres from long-staple tightly-spun and woven cotton goods, mercerised under tension, present under the microscope a different appearance to cotton which has been mercerised without tension.

On the other hand, fibres from short-staple loosely-spun and woven goods, mercerised with or without tension, show the same microscopical characteristics.

This is due to the fact that, when mercerised under tension, in the first cited case the fibres cannot change their position, and are consequently forced to alter their length and structure, while in the latter case the fibres, being free to move, slide over one another and escape the strain, changing merely their position but not their length or structure. Hence it is impossible to impart to goods made from short-staple and loosely-spun yarns a silk-like gloss.
—H. I.

*Silk Weighting.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [9], 393. (See this Journal, 1898, 840.)

*Weighting with Tin.*—Stannic chloride ($SnCl_4$) has nowadays replaced the old "tin salts" (stannous chloride), because it acts less injuriously on the fibre. The silk is treated for 1 hour in a bath containing stannic chloride solution of 25°—30° B., wrung out, washed, and then worked for 30—40 minutes in a lukewarm bath of sodium carbonate. This weighting (15—20 per cent.) is fast to soap and colourless, but the goods acquire an objectionable feel. The weighting may be increased to 100—120 per cent. by passing the goods through a bath of sodium phosphate (3°—5° B.) at 30° C. Alkaline silicates and tungstates alone, or mixed, may be used instead of the phosphate. The silk, after washing, is worked in a warm solution of stannic chloride (15° B.) or aluminium sulphate. As the deposit is soluble in acids the baths should be neutralised with ammonia. A lustrous white and strong silk may be thus obtained.

Salts of other heavy metals may be used to fix the phosphoric acid. Puller recommends the following series of baths: Stannic chloride, sodium phosphate, aluminium sulphate, sodium silicate. If tannic acid be used to fix the tin a weighting may be obtained (40—50 per cent.), which has the advantage of acting as a mordant in subsequent dyeing operations.

*Weighting with Iron* is largely used for blacks, and is often applied after treatment of the silk with sodium phosphate. Sometimes it is combined with the production of Prussian blue. The commonest method for obtaining heavily weighted blacks is to combine a weighting of iron with tin, and to fix with catechu.—H. I.

*Artificial Silk Industry.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [9], 399—400.

A correspondent of "La Nature" reports that a large factory for the manufacture of this article has been started at Besançon. Artificial silk is finding increasing application for the manufacture of ribbons, dress, and "fancy" goods, theatrical costumes, and the like. According to the report the product resembles silk so closely that only experts can detect any difference; it is, however, slightly less firm to the feel.

The method there employed is that of de Chardonnet. Nitrocellulose, prepared, as usual, from either cotton or wood pulp, is dissolved under a pressure of 40—50 atmospheres in ether-alcohol, and the solution, which has a tough consistency, thicker than photographic collodion, is carefully filtered from unchanged cellulose. It is then led in pipes, still under pressure, to the spinning room, where it is distributed in smaller tubes running along the spinning frames. These narrow tubes are provided with a number of small reservoirs, each of which end in glass jets $\frac{1}{100}$ mm. bore, through which whole system of pipes the liquid is forced. Formerly, the nitrocellulose thread passed direct from the jet into water, which coagulated the cellulose by dissolving out the alcohol, but better results are now obtained by causing the solvent to rapidly evaporate (dry spinning). In order to form a thread, three, four, or more such fibres are spun together. The further treatment of the product is similar to natural silk, only, before weaving, the threads must be denitrated in order to render them non-explosive and less inflammable. The spun thread or yarn is passed through a bath containing ammonium sulphide solution, which, by removing the oxides of nitrogen, converts the material into cellulose. None of the valuable qualities are said to be lost by this process, and the product is rendered less inflammable than ordinary spun cotton. Such artificial silk can be dyed to any shade, and may be produced at 50 per cent. less cost than the natural fibre.—H. I.

*Wool, Crude and Carbonised, Working up for Hatmaking.* Deutsche Färber-Zeit.; Deutsche Hutmacher-Zeit. **29**, [10].

Among the disadvantages of wool carbonised in the fleece may be mentioned decreased felting power, in consequence of which the wool cannot be satisfactorily fulled in the machine, and yields a felt which stretches when beaten. It is also difficult to match colours when dyeing carbonised wool with dyewoods, sumac, &c. to be darkened with iron salts, though this difficulty is less apparent when chrome mordants are used.

All things considered, a short-staple uncarbonised wool is generally preferable to a long-staple carbonised wool, unless the latter be strong in fibre; and carbonising should only be resorted to when the presence of impurities, which would spoil the appearance of the finished goods, renders this course unnecessary.—C. S.

*Wool Hats, Stiffening.* Deutsche Färber-Zeit.; Deutsche Hutmacher-Zeit. **29**, [38].

For slight stiffenings, aqueous solutions of gum tragacanth, Iceland moss, lichen gum, or cherry gum are employed, the first named being qualified with a little glycerin or Iceland moss to counteract the harshness of the unmixed gum.

A higher degree of stiffness is obtained by using so-called "patent" stiffenings prepared by dissolving, for example, 1 part of borax and 1 of soda in 70 parts of boiling water, and adding thereto 3 parts of ordinary rosin and 2¼ of gum elemi. When these have been dissolved by boiling and stirring, ¼ part of turpentine oil or linseed oil, and 20 of good orange shellac, are stirred in and boiled until the ingredients are thoroughly incorporated together.

This preparation is applied uniformly over the surface of the hat body by brushing, after which the hat is steamed, dried, steeped for an hour in a bath of weak hydrochloric acid—the temperature being raised from 104 to 140° F.—and finally brushed in cold water, rinsed, drained, and dried.

In the case of colours unaffected by weak acids, sulphuric acid is used, hydrochloric acid being advisable for very delicate colours, and pyroligneous acid for all others.

For very stiff hats, shellac dissolved in spirit is employed, cold-prepared solutions being used for stiffening the brim, on account of their greater penetrating power, whilst the crown is stiffened with hot-made solutions.

Elastic crown-stiffening for light coloured hats:—Orange shellac, 15 kilos.; white lac, 7 kilos.; elemi, 3 kilos.; spirit, 50 litres.

Hard stiffening for light hats:—Orange shellac, 11½ kilos.; white lac, 6½ kilos.; elemi, 1½ kilos.; rosin, 3½ kilos.; spirit, 50 litres.

Elastic stiffening for dark hats:—Orange shellac, 10 kilos.; blood lac, 10 kilos.; elemi, 3 kilos.; spirit, 50 litres.

Hard stiffening for dark hats:—Orange shellac, 10 kilos.; blood lac, 11 kilos.; elemi, 1½ kilos.; rosin, 1½ kilos.; spirit, 50 litres.

Brim stiffening for very light hats:—Bleached shellac, 20 kilos.; rosin, 1½ kilos.; gallipot, 1½ kilos.; elemi, 1½ kilos.; spirit, 50 litres.

Wash for cleaning the hats after stiffening:—Soft, boiling water, 50 litres; borax, 1½ kilos.; soda, 1½ kilos. Used boiling hot.—C. S.

*Semi-Woollen Textile Fabrics, Examination of.* S. Kapff. M. Text. 1898, 193; Analyst, 1898, **23**, [272], 234.

*See under* XXIII., *page* 956.

## PATENTS.

*Woven Fabrics. Impts. in the Production of certain Figured.* George Douglas, Bowling, Bradford, Yorkshire. Eng. Pat. 23,268, Oct. 11, 1897.

The figured fabrics, to which this invention relates, are made by introducing animal and vegetable yarns in the form of weft from the same bobbins or shuttles into a woven fabric, and then mercerising the cloth produced. The vegetable yarn is contracted, and the animal fibres, by virtue of their greater length, stand out in relief, forming a pattern or design on the surface.—H. I.

*Hose or Tubing: A New or Improved Composition for Treating Fabrics for the Manufacture of.* J. L. Jones, Dublin. Eng. Pat. 23,634, Oct. 14, 1897.

Textile fabrics used in the manufacture of hose or tubing known as "wire-bound," are rendered waterproof and resistant to the action of animal, vegetable, and mineral oils, by coating them with a composition consisting for ordinary purposes of 162 lb. of best boiled linseed oil, 21 lb. of lamp-black, 25 lb. of "dryers," 4 oz. of "antimony," and ½ a pint of "terebine." Instead of the "antimony," graphite, American soapstone, &c., may be employed.—H. I.

*Flax and Hemp for Obtaining Textile Fibres therefrom, An Improved Process of Treating.* C. Wetherwax, Best, New York, U.S.A. Eng. Pat. 24,528, Oct. 22, 1897.

According to this invention, the material, after breaking, is placed in divisional baskets contained in a cage placed in a digester, and subjected to the action of a boiling solution of silicate of soda, or "water-glass" (½ oz. to the gallon), under a steam pressure of about 10 lb., or at the atmospheric pressure. By means of suitable arrangements, the solution is kept in constant circulation through the digester, whereby the retting is effected in a much shorter time than ordinarily required, and better results with regard to the colour and strength of the fibre are obtained.—H. I.

*Dressing or Coating of Cloth or Canvas for Making Waterproof Covers of all kinds, such as Tarpaulins, Van Covers and Sheets, Railway Sheets, Drivers' Aprons, Horse Loin Cloths, and other Articles; Impts. in the.* T. P. Coubro, Limehouse, Middlesex. Eng. Pat. 24,920, Oct. 27, 1897.

This invention consists in mixing with the patent dressing some non-drying or non-oxidising oil, and applying the same to the material. The non-drying oil permeates the cloth or canvas, and thereby renders it soft and pliable, not apt to break or crack when doubled up; while the surface is quite dry, and fit to receive the usual top coat of dressing. —H. I.

*Textile Materials, Impts. in and connected with the Production of Lustrous Effects upon Vegetable.* W. G. Heys, Manchester A communication from H. David, Paris. Eng. Pat. 26,247, Nov. 11, 1897.

In this invention the stretched fabric is passed over a number of boxes, covered by perforated lids, which are partially evacuated by means of a suitable pump. The mercerising, washing, or neutralising liquid is placed on one surface of the fabric, and is withdrawn through the fabric into the boxes by the suction of the pump. Or the material may be mercerised as usual, and then, while in a wet condition, passed on to the machine, where it is stretched, neutralised, and washed.—H. I.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Mordanting Wool, Some New Observations on the Employment of Lactic Acid in.* C. Dreher. Rev. Gén. des Mat. Col. 1898, **2**, [20], 303—306.

Contradictory opinions exist among dyers with regard to the value of lactic acid as an assistant to the bichrome mordant, some being especially favourable to it on account of the exhaustion of the mordanting bath, and therefore its less expense, others condemning it because it does not give a level and penetrating mordant.

This state of affairs is due to the unreasonable way in which the manufacturers of lactic acid at first placed it upon the market. The lactic acid first sold was almost like a black glue, having an odour of putrid starch, and containing only 20 per cent. of the pure acid. The ill effects of the lactic acid, such as sticking together and hardening of the goods, were due to the impurities contained in it, and not to any inherent property of the acid itself, which, in its pure state, is a viscous liquid resembling glycerin.

Again, it was recommended to use the lactic acid in the same way as cream of tartar as an assistant to the chrome mordant. Thus the trials were almost altogether disappointing, the result being that the sale of lactic acid nearly came to a standstill.

The author was now requested by the firm of Boehringer to make a thorough study of this body with regard to its applications. Fortunately, the first experiments, made with pure lactic acid, proved that it could be used advantageously with textile fibres. Its manufacture was then investigated, the fermentation was better controlled, and more care brought to bear on its purification, with the result that now a 50 per cent. lactic acid is produced, of a yellow colour, and containing mere traces of glutinous matter. Indeed, lactic acid is now not only used when mordanting wool, but also, in the form of the double salt of antimony and calcium, as a mordant for cotton; in the printing of fabrics it replaces tartaric acid and glycerin, and is used also to dissolve colouring matters insoluble in water. When wool is mordanted with a mixture of lactic and chromic acids (neither of them being in large excess), a yellow-brown precipitate of chromium chromate is deposited unevenly and in a superficial condition on the wool. This occurs even in the cold, and very rapidly at the boil. However, by mordanting wool with 1·5 per cent. of potassium bichromate and 4·5 per cent. of lactic acid of 50 per cent. strength, the wool is mordanted green, and on dyeing gives shades of double the intensity of those obtained by dyeing fibre mordanted with 3 per cent. of potassium bichromate and 2·5 per cent. of tartar.

The above amounts of potassium bichromate and lactic acid are too dear for practical use, but the same effect is obtained by mordanting with 1·5 per cent. of potassium bichromate, 3 per cent. of lactic acid, and 1 per cent. of sulphuric acid. The wool, after being well wetted out, is entered into the lukewarm bath; by raising very slowly to the boil, the goods are evenly mordanted and well penetrated. The price is much less than with tartar, the bath is exhausted, and the work becomes more regular; moreover, the wool is mordanted green. These proportions of mordant found by experimental trial are in agreement with theory.

Nevertheless, even this method is impracticable for thick cloths, hard stuffs, greasy wools, &c., which require a long and continued boiling for the mordant to penetrate the fibre. However, the author has devised a method of mordanting with lactic acid where the goods can be entered at the boil without any danger of uneven and superficial penetration. This is done by adding ammonium acetate to the mordanting bath containing potassium bichromate, lactic acid, and sulphuric acid; or, more cheaply still, by replacing the sulphuric acid by ammonium sulphate, which decomposes slowly in the boiling bath into ammonia, which evaporates off, and sulphuric acid, which thus acts gradually. By employing 1·5 per cent. of potassium bichromate, 3 per cent. of lactic acid, and 1 per cent. of ammonium sulphate, the wool is slowly mordanted and the bath quite exhausted at the end of two hours' boiling.

The operation can be hastened at will by adding sulphuric acid.

In order to mordant the wool yellowish-green instead of pure green, a little more potassium bichromate may be used, say 2 per cent. This is generally to be recommended for dyeing blacks with logwood, which gives a deeper black with an excess of unaltered chromate. When dyeing with alizarins, the pure green mordanted wool should be used.

A logwood black obtained in single bath with lactic acid, is redder than with tartar. This difference disappears if the wool be oxidised with bleaching powder or any other oxidising agent; also, if the wool be boiled with the potassium bichromate alone for some little time before adding the lactic acid.

To sum up, the author thinks that lactic acid can always be used for mordanting wool, and that it is only a question of a little experience in order to select the most advantageous method for each case.—J. E. H.

*Irichromatine: A New Process of Colouring without Material Colour.* L. Lefévre. Rev. Gén. des Mat. Col. 1898, **2**, [20], 289—292.

The wonderfully dazzling and pure colours which we see in the wings of the butterfly, in the feathers of the peacock, or in a soap bubble, are due to interference phenomena, produced by the action of light rays. Their purely physical origin would seem to exclude the possibility of producing similar effects upon textile and other fabrics, and fixing them there. However, this problem has been solved by

Charles Henry, whose patented process, on account of its simplicity and cheapness, is now being worked by a company.

Interference colours, being spectrum colours, are of great brilliancy and purity. They are produced whenever light meets an extremely thin film, indeed of similar dimensions to those of the wave-lengths of the spectrum colours, that is to say, something approaching to millionths of a millimetre. They are produced under the most diverse conditions; for example, with glycerin films, collodion, by electrolytic deposits, known as Nobili's rings, and, finally, by dropping a small quantity of an oily substance upon water, chiefly olive oil, petroleum, essence of turpentine, &c.

This last fact Henry has utilised in forming thin films of great resistance and perfect continuity, and fixing them fast upon material such as paper, textile fabrics, glass, &c.

A film produced by olive oil upon water soon evaporates; moreover, it is impossible to fix such films upon paper, as they would be at once absorbed. However, by dissolving certain resins or pitches, which have the property of becoming hard on exposure to the combined influence of air and light, in a suitable solvent, we can produce a suitable film by the evaporation of the solvent. It was a much more difficult matter to obtain a perfectly continuous pellicle, but this was ultimately accomplished by employing a solvent like benzene, which is nearly insoluble in water. By dissolving Damar gum, or mineral pitch, in benzene, a solution known as *Trichromatine* is obtained, by means of which a pellicle suitable in every respect is produced. In order to fix and protect the film from friction, the paper which is to be irisated is previously coated with gelatin which has been rendered insoluble by formic aldehyde. The method of producing the film on the large scale is carried out by allowing *Trichromatine* to fall drop by drop upon water contained in a shallow tub. A knife is attached, which stretches the edge of the film and thus causes the thickness of the film to vary; in this way the formation of the colours is modified.

The paper to be irisated is run upon a roller, and penetrates the surface of the water at a very small angle, and by its very slow motion drags with it the pellicle which has just been formed. After coming out of the tub the paper is gently dried, so as not to cause any cracks in the film, and again wound on rollers.

The variety of colours and designs is infinite, and the same effect can never be produced twice. Under proper management, one tub allows of more than 1,000 metres of paper being irisated at a very small cost.

The base upon which the pellicle is deposited plays an important part; black grounds producing intense colorations; light grounds, delicate colorations.

In order to increase the decorative effect, the paper is finally submitted to the operation of embossing (goffering).

Pigment colours can be associated with the colours produced by *Trichromatine*, and resists can also be made upon textile fabrics, especially on half-silk; beautiful effects are obtained by means of *Trichromatine*, but practical difficulties have to be surmounted before they can be produced on a large scale.—J. E. H.

*Benzidine Dyestuffs, in the Printing Industry, Application of, during the last 15 Years.* G. Stein. Zeits. angew. Chem. 1898 [39], 897—900.

Many benzidine dyestuffs (such as Geranine, or Brilliant Geranine for rose colours; Benzo Blue, or Brilliant Benzo Blue 6 B for sky-blues; Chloramine Yellow, Chrysophenine, Chrysamine, &c. for yellows; Benzo Green for olives) are used for obtaining light-coloured grounds on cotton. The desired designs are first printed (with Aniline Black, Alizarin Red, basic aniline colours, &c.) on the fabric, which is then drawn through an aqueous solution of the benzidine dyestuff, and the colour is taken up by the ground. The benzidine dyestuffs are almost without influence on the printed colours. Large blotches may also be printed with Aniline Black, &c., leaving only small white figures which take up the benzidine dyestuff when the cloth is drawn through an aqueous solution of it.

The padding method is better than dyeing in the vat or the jigger, for light and medium shades.

The benzidine dyestuffs are not so good for direct printing on cotton as the mordant dyestuffs; they are, however, being much used where no great fastness is required. By using albumin-thickening for the printing mixture, greater fastness is obtained.

They act as mordants for basic colours, and thus "topped" and "cover-print" effects may be produced.

Most of them are poly-azo compounds, and as such they are decomposed by reducing agents. The reducing agents commonly employed are stannous salts. Zinc dust is to be avoided, since it is difficult to obtain it fine enough, and it is apt to scratch the copper rollers.

In order to destroy the benzidine dyes locally, zinc dust is to be recommended, as it produces a purer white.

In the trade, many other classes of dyestuffs are grouped under the general name of benzidine colours. Many of these are not affected by reducing agents, so that by printing with a combination of those which are and those which are not and then reducing, good "conversion" effects are obtained.

Oxidising agents discharge benzidine dyestuffs, and this process (using potassium ferricyanide, or sodium chlorate) is made use of for the production of variegated prints.

To the class of benzidine dyestuffs a number of dyes belong which entirely or only partly resist the action of oxidising agents, hence by using these in conjunction with Alizarin chrome lakes (which are discharged by oxidising agents) excellent coloured prints can be obtained.

Benzidine dyestuffs, which are not attacked by alkali can be used on mercerised cotton. A paste of the dyestuff is made with the caustic alkali and printed, then dried and washed, or the goods are printed with the dyestuff and gum and then drawn through the alkali. Locally mercerised cotton, when printed with these dyestuffs, gives deeper colours on the mercerised parts, and thus a two-colour effect is produced with one dyestuff and in one operation.

The following four classes of benzidine dyestuffs are distinguished:—

1. Those which dye cotton and wool equally: Benzo-Purpurin 4 B, 10 B; Benzo Orange R; Benzo Green G; Benzo Azurine 3 G, G; Benzo Blue 2 B, 3 B, B X, R W; Benzochrome Brown B, G, R, 3 R; Direct Blue Black B, N; Direct Deep Black G, R; &c.

2. Those which dye wool more strongly than, but of the same tint as, cotton: Brilliant Geranine 3 B; Chrysamine G, R; Benzo Dark Green; Benzo Red Blue; Congo Ruby; &c.

3. Those which dye cotton more strongly than, but of the same tint as, wool: Chloramine Orange; Curcumin S; Brilliant Azurine B; Benzochrome Black-Blue B; Diazo Black B H N.

4. Those which give entirely different tints on the cotton and wool: Benzo Olive; Benzo Black Blue G, R; Benzo Brown N B R; &c.

A somewhat similar classification can be made with regard to their comparative behaviour in dyeing towards silk and cotton.

If mercerised cotton be used instead of ordinary cotton in making half-wool goods, a very prettily dyed cloth can be obtained on using benzidine dyestuffs. These are now beginning to be used in linen and jute printing.

Some other applications of these dyestuffs are given in the paper, which purports to be a review of their employment during the last 15 years.—J. McC.

*Diamine Colours, White Discharge for.* H. Pomeranz. Färber-Zeit. 1898, 9, [15], 230—231.

At present two kinds of tin discharges are employed for these colours: the acid discharge, consisting of stannous acetate; and the alkaline discharge, consisting of a mixture of stannous hydrate and ammonium chloride. The former requires long steaming and gives soiled whites; the alkaline discharge, depending for its action on the gradual formation of stannous chloride and the simultaneous liberation of ammonia, can be steamed for a longer time without "yellowing" the whites. Unfortunately, it is almost impossible to obtain sharp outlines with this latter.

The author recommends a discharge which is really a combination of both the above, and which is stated to be without their defects. It has the following composition :—White starch, 90 parts; British gum, 30 parts; stannous acetate solution, 30° B., 500 parts; ammonium chloride, 110 parts; ammonium citrate, 100 parts; acetic acid, 8° B., 120 parts, and Glauber's salts, 50 parts.

The stannous acetate solution is made by precipitating 1 kilo. of stannous chloride dissolved in water (2½ litres) with 475 grms. of sodium carbonate dissolved in 2½ litres of water, and, after filtering, dissolving the precipitate in 2 litres of acetic acid of 8° B.

Ammonium citrate is made by neutralising a solution of citric acid (30° B.) with ammonia, when the solution should mark 24° B.

Such a discharge may be steamed for 1½ hours; the stannous acetate is gradually decomposed and the stannous hydrate attacks the ammonium chloride.—H. I.

### *Black Oxidation Dyestuffs; Employment of Alcohol in Mordanting.* Marot and Bounet. Rev. Prod. Chim. **1**, [8], 116—117.

The authors find that by an addition of alcohol to the mordanting bath, in dyeing with oxidation blacks, any excess of acid present is eliminated, and a certain equilibrium is established between the active ingredients. This adjustment takes place in the oxidising chamber, where it is favoured by the prevailing temperature. The oxidation then proceeds in a normal and reliable manner without the strength of the fibre being diminished.

Any alcohol may be used, and any mordanting and dyeing ingredients, so long as the same are not precipitable by alcohol. The influence of the alcohol varies directly with the quantity, but there is no advantage to be derived from the use of more than 25 per cent.; and the minimum limit of utility is 5 per cent.—C. S.

### *Printing Tungstate of Barium on Fabrics, so as to Produce Opaque and Dull Effects; Imitating Embossed Articles.* A. Scheurer. Rev. Gén. des Mat. Col. 1898, **2**, [20], 306—307.

C. Koechlin obtained these effects by printing a concentrated lead salt, *e.g.*, acetate-nitrate, on the fabric, drying, ageing, and passing through sulphuric acid or sodium sulphate. The disadvantage which lead salts possess, of turning brown in the presence of the slightest traces of sulphuretted hydrogen, led the author to experiment with the insoluble tungstates and molybdates, the opacity of which is very great.

Although lead tungstate was the most opaque precipitate which the author obtained, it was preferable to use barium tungstate. The fabric was printed with a colour composed of sodium tungstate and gum tragacanth, dried, steamed two minutes to cause the tungstate to penetrate the fibre better, passed into a cold solution of barium chloride (50 grms. per litre), washed and dried. The steaming is not absolutely necessary. The white thus obtained can be coloured with pigments, like vermilion, chrome yellow, Guignet's green, and ultramarine blue. These colours are not so fast as when fixed with albumin, but, on the other hand, the fabric is not stiffened, and the colours appear very fresh, and withstand a lukewarm soap solution.

Barium tungstate can be employed very well along with colours fixed with albumin. In this case the goods are steamed before passing into the solution of barium salt, but too long steaming must be avoided, or the sodium tungstate has a tendency to run. The addition of a trace of caustic soda to hasten coagulation is very effective. Barium tungstate can also be used for the grounds of fabrics, since it communicates no hardness to them, but it will not work with steam-colours, or colours on chrome mordant, which require soaping, since the barium tungstate would be thereby stained.

By printing sodium tungstate upon fabrics previously printed with ground colours, and afterwards passing into a solution of barium salt, very marked effects of contrast are obtained. The portions covered with barium tungstate are quite dull, while the portions not printed with tungstate appear quite brilliant by contrast, and have the appearance of silk.

Other effects of contrast are obtained by printing on a rather thin fabric, the tungstate coloured with chrome yellow, minium, &c. Those portions which have not been printed, possess the complementary colour (but weakened by contrast) to those which have been printed, and which are opaque.

Patents for these various processes have been taken out in different countries by the author.—J. E. H.

### *Mordanting Wool, Observations on the Use of Lactic Acid in.* C. Dreher. Färber-Zeit. 1898, **9**, [16], 248—251.

The quantities given by the author for mordanting wool, *viz.*, 3 per cent. of lactic acid (50 per cent.), 1·5 per cent. of potassium bichromate, and 1 per cent. of sulphuric acid, have been found satisfactory in practice. The mordant is especially suitable for dyeing with Alizarin colouring matters, and with Logwood, when with the latter the production of a good black is the main object. When the cost of dyeing is taken into consideration, it is found that the green chrome-mordanted wool, owing to its incapability of exerting any oxidising action on the dyestuff, requires more of this, and that dyeing with it is, in consequence, more expensive.

It is also found that tightly-spun yarns and tissues of thick texture are not penetrated so well or mordanted so evenly by means of the above mixture, as they are with potassium bichromate and tartar. Sometimes this is due to the presence of fatty substances in the wool. In the case of such materials, mordanting must be so performed that little chromium becomes fixed during the first half-hour of boiling. This is accomplished by the use of a mixture of potassium bichromate (1·5 per cent.), lactic acid (3 per cent. of a 50 per cent. solution), and ammonium sulphate (1 per cent.). The last compound becomes gradually decomposed during boiling, into ammonia, which escapes, and sulphuric acid, which, in conjunction with the lactic acid, acts upon the potassium bichromate and effects the mordanting of the wool. If any fatty matters should be present in the wool, these become acted upon by the ammonia prior to its being volatilised, and are thus removed from the wool.

An oxidising mordant, which is to be recommended for dyeing compound shades from Alizarin dyestuffs and Logwood, is obtained from a mixture of potassium bichromate (2 per cent.), and lactic acid, and ammonium sulphate (in the proportions given above), the operation of mordanting being interrupted when the bath has a pale yellow colour.

This mordant gives colours which closely resemble those produced on a mordant obtained from potassium bichromate and tartar.

A slight redness of shade is to be seen in the Logwood Blacks dyed upon mordants fixed by the agency of lactic acid. This is not to be ascribed to the fact that the mordant is in the condition of chromic hydroxide, since the dyes fixed upon wool which has been mordanted with potassium bichromate and tartar, and afterwards treated with sodium bisulphite, do not exhibit any such shade. It is probably due to the absence of oxidising action on the wool on the part of the mordanting mixture. Such an action may be brought about in the case of lactic acid by performing the mordanting in two operations, the wool being treated first with potassium bichromate, and afterwards in a separate bath with a mixture of lactic and sulphuric acids. The first of these baths should be kept and used again after the necessary quantity of potassium bichromate (or chromic acid) has been added to it. The wool mordanted in this manner dyes the same as wool mordanted with potassium bichromate and tartar.—E. B.

### *Uneven Dyeing, Correction of.* G. Robrecht. Färber-Zeit. 1898, **9**, [16], 251—252.

C. Otto has asserted (this Journal, 1898, 843) that the fault of cloudiness of colour in dyed hats, can be rectified by boiling the latter with equal parts (1 per cent.) of sodium bisulphate and acetic acid.

In connection with this the author remarks that when the defect is due to imperfect scouring or to the presence of dirt of any kind in the dyed material, it is impossible to rectify it without dyeing a deeper shade. It is, consequently, difficult to find a remedy when the fault is discovered after the material has been dyed fully to the shade desired.

When, however, the irregularity makes its appearance shortly after dyeing has commenced, an addition to the bath of Glauber's salt (5—8 per cent.) may be useful, the salt having a retarding action on the dyeing.

The treatment is ineffective when acetic acid is present in the bath, as dyeing is not in such a case prevented from taking place rapidly. For pale shades, therefore, it is advisable to add to the dyebath, ammonium acetate in place of acetic acid.

Sodium bisulphate can be employed to reduce or remove only such colours as are not fast to acid. After it has been used, the woollen materials should be re-dyed with direct acid dyestuffs.

Colours which are loose to alkalis, are best removed by treatment with ammonia.

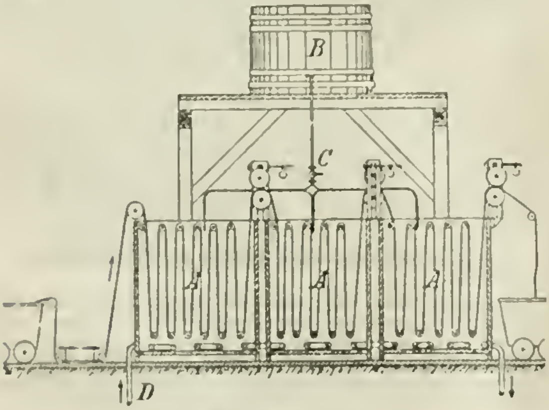
Uneven colours dyed from "Aniline Dyestuffs," unless the material be dirty or the dyestuff at fault, may be rendered passable by boiling well, whilst working about evenly, and, after some time, adding to the bath sodium sulphate, sodium bisulphate, or sulphuric acid.—E. B.

*Diaminogene Black on Mercerised Cotton Tissues, Continuous Process for Dyeing.* A. Kertész. Färber-Zeit. 1898, **9**, [16], 216—218.

By means of the arrangement of apparatus described and illustrated below, mercerised cotton tissues are dyed Diaminogene Black, the process of dyeing, diazotising, and developing being continuous.

The apparatus employed in dyeing consists of three tanks A A A, placed together as shown in the figure.

Fig. 1.



Continuous-Dyeing Apparatus.

These are fitted with tension and draw rollers. Above them is fixed a wooden vessel B, containing a concentrated solution of the dyestuff used. Each tank has in it a steam coil, provided with a three-way tap, the office of which is to shut off the steam, to cause it to pass directly into the bath, or through the coil, as may be desired.

The tanks are approximately 120 cm. in height, 80 cm. in length, and of a width which varies according to the width of the tissues to be dyed. The draw rollers are about 15 cm. in diameter, and are driven at a speed of about 45 revolutions per minute, corresponding with an output of about 1,000 metres of dyed tissue per hour. If a greater speed than this should be desired, a fourth tank must be added to the set.

At the beginning of the dyeing operation, the tanks are each half filled with water. This is heated, and there is then added to each tank, for tissues of ordinary width (*viz.*, about 80 cm.) 200 grms. of sodium carbonate, 2 kilos. of Diaminogene B, 0·75 kilo. of Turkey-red oil, and 2 kilos. of calcined sodium sulphate (salt-cake), double these quantities being taken in setting the tanks for tissues 140—160 cm. wide. Slight variations in these quantities may require to be made for the tissues of different textures, and to suit the speed of running of the machine, if this be different from that given.

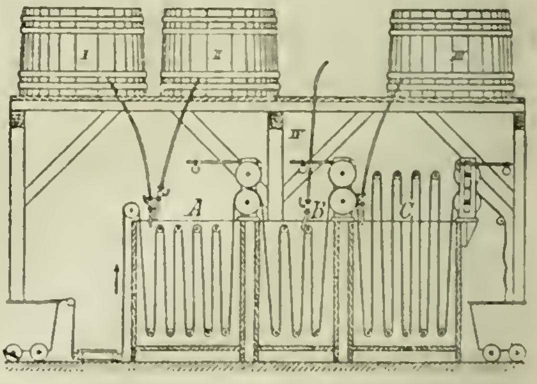
The amounts of the dyeing materials to be added during the passage of the tissues are regulated solely by the weight of the latter, and are, per 10 kilos.:—50 grms. of sodium carbonate, 350—400 grms. of Diaminogene B, 50 grms. of Turkey-red oil, and 350—400 grms. of calcined sodium sulphate. They may be added either at intervals or continuously during the passage of the tissues. The result is not affected if more of them be added to one tank than to another.

The tissues to be dyed must be thoroughly wetted prior to entering the tanks, and the temperature of the baths must be at or near the boil.

A bluer shade of black than is obtained with Diaminogene B alone, is produced with a mixture of equal parts of that dyestuff and of Diaminogene extra.

The tissues, after being dyed, are rinsed in cold water, and are then diazotised and developed. The machine on which these operations are accomplished is shown in the accompanying figure.

Fig. 2.



Continuous-Dyeing Apparatus.

A is the diazotising tank, which is fed with solutions of sodium nitrite and of hydrochloric acid from vats I. and II. respectively. B is a rinsing tank, through which a stream of water, to which acid is added from time to time, is caused to flow. The developing tank C is supplied with the requisite solution from vat III.

Tank A is prepared for use by half filling it with a solution of sodium nitrite (1½ grms. per litre) acidified with hydrochloric acid (5 grms. per litre). Vats I. and II. contain solutions of sodium nitrite and hydrochloric acid, which are added to the tank as the tissues pass, at such a rate that 1 per cent. of the weight of the tissues of the salt and 3 per cent. of the acid are introduced.

For developing, phenylenediamine is used, either alone, to give a jet black, or in admixture with β-naphthol for a blue-black, or with resorcinol. The last mixture is the most common, as it yields a shade similar to that of Aniline Black. The bath is set with this by half filling the tank reserved for it with a solution of phenylenediamine (0·2 grm. per litre), resorcinol (0·2 grm.), and caustic soda (0·5 grm. at 40 B.). A solution of the same compounds, in the ratio of 0·15 part of the first and second to 0·5 of the third is placed in the feeding-tank, and suffices for the development of 100 parts of the dyed tissue.

After passing through the diazotising and developing baths, the tissues are allowed to lie for half an hour. They are then rinsed, and soaped if desired.—E. B.

*Lignorosin* [*"Calcium Lignosulphonate"*]: *A New Reducing Agent for Chromic Acid in Wool Mordanting.* A. Raaz. Färber-Zeit. 1898, **9**, [16], 245—246.

This compound, stated to be calcium lignosulphonate, is a by-product of one of the processes of manufacturing paper pulp from lignocellulose fibres. It appears in commerce in the form of a dark-brown syrup, with a caramel-like odour, and is found to serve in many cases as an efficient substitute for lactic acid in mordanting wool with chrome.

Dye trials were made with wool mordanted with 2·7 per cent. of lignorosin, 1·26 per cent. of potassium bichromate, and 0·66 per cent. of sulphuric acid.

Mordanting is complete after an hour's boiling, the whole of the chromium being absorbed, and the fibre is affected no more than it is with a mixture of potassium bichromate, lactic acid, and sulphuric acid. The quantity of chromium thus absorbed is the same as is absorbed from a mixture of 2·7 per cent. of lactic acid, 1·26 per cent. of potassium bichromate, and 0·62 per cent. of sulphuric acid; or from one of 3 per cent. of potassium bichromate and 2·5 per cent. of tartar.

The wool mordanted as described has a pale-brown colour, which renders it unsuitable for dyeing colours such as pale blues, greens, and pinks, but makes it more suitable than wool mordanted in other ways for dyeing medium and dark blues, browns, drabs, olives, and blacks, as less dyestuff is required. The saving, for instance, in the case of a dark brown from Diamond Brown R on worsted material, amounts to 3 per cent. of the commercial 20 per cent. paste, 12 per cent. of this giving the shade ordinarily produced with 15 per cent.

As the compound is much cheaper than lactic acid, it is considered likely to replace the latter to some extent in wool dyeing.

The author is indebted to a paper by H. Seidel, in the Mittheil. des k. k. Techn. Gewerbemuseums (this Journal, 1898, 844), for the information that colours dyed on wool mordanted by the aid of lignorosin are as fast as usual to water, soap, alkalis, and acids. Their fastness to light has not yet been determined, but no doubt is entertained that this will be found satisfactory too.—E. B.

*Indigo on Cloth, Estimation of.* A. Binz and F. Rung. Zeits. angew. Chem. 1898, [39], 904.

*See under* XXIII., *page* 957.

*Sumac, Recognition of Adulterations of.* M. Spica. Chem. Zeit. Rep. 1898, **22**, [69], 212.

*See under* XXIII., *page* 954.

*Tannic Acid, A Practical Method for the Analysis of.* P. Wilhelm. Rev. Gén. des Mat. Col. 1898, **2**, [20], 307.

*See under* XXIII., *page* 957.

## PATENTS.

*Bleaching Linen and other Objects, Manufacture of Conglomerates containing Alkaline Oxidising Bodies for.* L. H. Frentz, Brussels, Belgium. Eng. Pat. 27,049, Nov. 18, 1897.

A solution of sodium monosilicate of 1·65 density is prepared, in 53 kilos. of which, at a temperature of 50° C. 42 kilos. of dry sodium carbonate are incorporated in a jacketed mixing machine. When the mass becomes viscous, 5 kilos. of sodium peroxide are added by small portions, and the cooling mass is pressed into moulds. The blocks thus formed are cut into pieces, which are then dipped into a concentrated solution of an alkali silicate. On exposure to the air, carbonic acid is absorbed, and a siliceous coating is thereby formed on the surfaces, which protects the conglomerates from deliquescing. The protecting process may be hastened by exposing the dipped pieces to an atmosphere rich in carbonic acid, such as cold combustion gases. Another formula is given in which modified proportions of the constituents are used.—E. S.

*Dyeing Sliver for Textile Fabrics, Impts. in Apparatus for.* H. H. Lake, London. From D. Mattei, Genoa, Italy. Eng. Pat. 17,173, July 20, 1897.

The slivers are dyed in a continuous manner in a machine, the chief characteristics of which are:—(1) One or more injectors serving for the preliminary moistening of the slivers; (2) a number of consecutive steeping or dyeing vessels; (3) a device consisting of a guiding band conveying the sliver from one vessel and carrying it to a reel, which deposits it on a flexible wooden apron, and this carries it through the next dye vessel.—R. B. B.

*Washing and like Treatment of Printed Fabrics, Impts. relating to the, and to Apparatus therefor.* E. Remy, Mulhouse, Alsace. Eng. Pat. 22,351, Sept. 29, 1897.

In washing printed fabrics the chief difficulty is the removal of hardened thickening material. The improvement designed for this purpose comprises a drum or barrel mounted on a horizontal shaft. This drum is formed of two discs or end-plates connected by rollers, and paddles interposed between the rollers. The upper part of the drum is surrounded by a series of flexible rollers, each of which is enveloped in an india-rubber sheath; the lower part of the drum is immersed in the vessel containing the washing liquid. When the drum is revolved, the rollers of which it is composed strike against the flexible rollers, and the cloth passing between the two sets of rollers is violently struck. The thickening is thus disintegrated, and is then removed by the washing liquid.—R. B. B.

*Mordanting, Impts. in.* F. Haber, Carlsruhe, Germany. Eng. Pat. 9484, April 25, 1898.

Cotton is mordanted with chromium by first charging the fibre with the hydrate, carbonate, or tannate of an alkaline earth (*e.g.*, by steeping successively in tannin and lime water), and then boiling the cotton in a solution of chromium fluoride or other chromium salt.—R. B. B.

*Mordanting, Sizing, or Mercerising Hanks of Yarn, Impts. in Machines for.* W. P. Thompson, Liverpool, &c. From B. Cohnen, Grevenbroich, Germany. Eng. Pat. 12,379, June 2, 1898.

The machine consists of several pairs of yarn-carrying rollers arranged concentrically on a horizontal shaft, so that by rotation of the latter, the rollers change their position and can be in turn saturated with liquid, squeezed, washed, opened out or brushed, and removed. The stretching rollers are so adjusted by means of a cam groove that the tension of the yarn during the operations of sizing, mercerising, squeezing, &c., may be varied according to the stage of the process. This is stated to be particularly important when mercerising cotton yarn to obtain a silky lustre. Assistant stretching rollers are also provided for equalising the tension of the upper and under hanks of yarn, this being also effected by means of a cam groove.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Sodium Chloride Solutions, Specific Gravity of.* H. C. Hahn. J. Amer. Chem. Soc. **20**, [8], 621—630.

The author has applied a number of corrections to Gerlach's data for the specific gravity of solutions of sodium chloride, and has deduced a formula by which the percentage of sodium chloride ($p$) can, it is said, be calculated exactly to 0·01 per cent., if the specific gravity ($s$) at 15·08° C., air thermometer, be known to the fourth decimal—

$$p = 60{\cdot}209{,}585 - 626{\cdot}853{,}1\,s + 1067{\cdot}352{,}667\,s^2 - 633{\cdot}92\,s^3 + 133{,}333{,}333\,s^4.$$

A method of experimentally determining the corrections to be applied in order to reduce weighings in air to weighings *in vacuo*, is also described, and is stated to give more accurate results than those obtained by calculations based on observations of the pressure, temperature, and amounts of water and carbonic anhydride in the atmosphere at the time of weighing.—E. W. W.

*Alkali Chlorides, Electrolysis of.* H. Wohlwill. Zeits. f. Elektrochem. 1898, **5**, [5], 52—76.

Sodium chloride is selected as the type of the alkali chlorides, and its behaviour on electrolysis fully considered. Besides the ordinary formation of caustic alkali at the cathode and chlorine at the anode, certain other processes take place during the electrolysis of such a solution, which give rise to the salts of the oxy-acids of chlorine. The chief of these are hypochlorites and chlorates, and also, under certain conditions, perchlorates.

The question which is of both scientific and practical interest, is how a solution containing initially only the anions $\bar{\mathrm{Cl}}$, $\bar{\mathrm{OH}}$, and $\bar{\bar{\mathrm{O}}}$ becomes charged with the anions of the hypochlorites and chlorates, *viz.*, $\bar{\mathrm{ClO}}$ and $\bar{\mathrm{ClO_3}}$.

In order to solve the problem, the author has adopted the method used by Glaser (this Journal, 1898, 462—464) for the study of the electrolysis of aqueous solutions generally. The same problem has been attacked by Oettel (this Journal, 1895, 492; and Zeits. f. Elektrochem. **1**, 69), and also indirectly by Haber, Zeits. f. anorg. Chem. **16**, 198 and 329); but since the author's intention was to isolate the primary reactions, he took precautions which would enable him to observe the initial direction of the reactions and not the final product, as was the case with Oettel's and Haber's experiments. For the author's purpose, some of the objections which may be raised against the methods of other investigators are as follows:—The duration of the experiments is too long; the products of electrolysis are not separated from each other, and consequently undergo further action by the current; the composition and concentration of the solution is constantly changing, &c.

By avoiding these conditions, which give as a result only the sum of different reactions which cannot be distinguished from each other, the author has arrived at the following conclusions:—

(1.) With as low an anodic electromotive force as 1·2 volt (against a hydrogen electrode), formation of hypochlorite takes place. With an E.M.F. of about 1·2—1·3 volt the reaction is almost quantitative, apparently according to the equation—

$$\bar{\mathrm{Cl}} + \bar{\bar{\mathrm{O}}}(+\ +) = \bar{\mathrm{ClO}}.$$

(2.) A more extensive formation of hypochlorite only begins above 2·1 volt, and this depends on a secondary chemical action of the liberated chlorine on the alkali present.

(3.) Chlorate is formed:—

(*a.*) By an ionic reaction on supplying positive electricity, and not

(*b.*) By a direct union of $\bar{\mathrm{Cl}}$ and $\bar{\mathrm{OH}}$ ions (as maintained by Haber), but more probably from the $\bar{\mathrm{ClO}}$ ions already formed in the solution, according to the equation—

$$2\bar{\mathrm{ClO}} + \bar{\mathrm{OH}}(+\ +) = \mathrm{ClO_3H} + \bar{\mathrm{Cl}}.$$

—J. S.

*Alkali Chlorides, Diaphragms for the Electrolysis of.* H. Becker. Ind. Electrochim. 1898, **2**, 33—34; Science Abstracts, 1898, **1**, [9], 566.

Brief descriptions are given of a large number of materials which have been proposed and patented for use as diaphragms. Parchment paper has been proposed by Le Sueur, Kiliani, Rathenau, and Sutter; it is in actual use at Rumford Falls, U.S.A., and at Bitterfeld in Germany. At Leopoldshall, parchment protected by a coating of calcium or magnesium oxychloride is used in the electrolytic cells. Albuminised paper and albumin alone have been proposed by Rieckmann; paper or cardboard coated with collodion, by Hoepfner. Diaphragms of unglazed porcelain or baked clay have been patented by many, and are in actual use in three works. Asbestos as a material for diaphragms has been patented by Roberts and McGraw, Hargreaves and Bird, Riquelle, Wiernick, Waite, Hempel, Richardson, and Meister Lucius and Brüning; whilst porous partition walls of Portland cement have been claimed by Breuer, Hurter, and by Carmichael.—A. S.

*Nitrites, Electrolytic Production of.* A. Dubosc. Rev. Gén. des Mat. Col. **2**, [19], 268—270. (See this Journal, 1896, 652; 1897, 142.)

Bourgoin and Brester showed (1869) that, in the electrolysis of nitric acid of a certain strength in a vessel divided into two parts by a diaphragm, the liberated hydrogen in the negative cell attacked the acid, which rapidly became blue by solution of the reduction products. On continuing the current for some time, ammonium nitrate was finally formed.

The large expenditure of electrical energy, necessary to furnish hydrogen for the complete reduction of the nitric acid, renders the method too expensive for commercial purposes. With nitrates, since a much smaller quantity of hydrogen is needed for the reduction, less energy is required.

In the negative compartment of a divided cell a solution of sodium nitrate, and in the positive a solution of an oxidisable metallic sulphate or chloride of equal electrical conductivity, *e.g.*, ferrous chloride, is placed. With suitable difference of potential, which the author calculates to be 5—6 volts, sodium nitrite is produced in the cathode compartment. The electrical energy necessary for the production of 1 kilo. of sodium nitrite is estimated as 4,000 watts.

The author has elsewhere described an electrolytic method for the preparation of sodium thiosulphate in a similar apparatus, which, by slightly reducing the dimensions of the electrodes, can be used for the production of sodium nitrite.—H. I.

*Perchlorate in Chili Saltpetre, Estimation of.* C. Ahrens and P. Hett. Zeits. für öffentl. Chem. **4**, 115; Chem. Centr. 1898, **2**, [8], 558.

*See under* XXIII., *page* 950.

*Coke Ovens, and Ammonia as By-Product.* B. Terne. J. Franklin Inst. **146**, [2], 127.

*See under* III., *page* 912.

## PATENT.

*Sulphur Trioxide [Sulphuric Anhydride], Method for the Manufacture of.* O. Imray, London. From The Farbwerke vormals Meister, Lucius und Brüning, of Hoechst a/Main, Germany. Eng. Pat. 6057, March 11, 1898.

"In the manufacture of sulphur trioxide by the combination of sulphur dioxide and oxygen," the method is claimed, as described, "of raising these gases to the temperature required for the reaction, by transferring to them heat from the produced sulphur trioxide, which has a high temperature, resulting from the combining reaction" referred to. The method of heat transference is effected through the agency of a simple counter-current apparatus. It is said that by the means and method described, complicated machinery and much fuel are spared, and that large apparatus, once the process is in action, "work entirely without supply of heat from outside."—E. S.

# VIII.—GLASS, POTTERY, ENAMELS.

*Ceramic Ware, Manufacture of Glazed.* H. de Wit. Monit. Céram. **29**, [11], 123.

The biscuit prepared by the author is made from slag which may be of any kind, incorporated with plastic clay, so as to have the following average composition:—Silica, 50; calcium carbonate, 25; alumina, 20; other substances (iron, iron oxide, magnesia, manganese, mica, organic matter, &c.), 5 per cent. It may also contain a proportion of flux similar to that used for the glaze employed, in order to facilitate the adhesion of the glaze on firing.—C. S.

*Enamels of High Dilatation for Cast or Common Iron on the Basis of "Pandermite."* M. Saglio. Bull. de la Soc. d'Encouragement, 1898, **3**, [8], 1060—1080.

A series of experiments was carried out with the object of utilising "*pandermite*" as a substitute for borax in the preparation of enamels of high dilatation for coating metals. "*Pandermite*" is a natural calcium borate of the following composition:—Anhydrous boric acid, 55·73; lime, 37·07; magnesia, 0·45; sulphuric acid, 2·55; carbonic acid, traces; alumina and oxide of iron, 0·10; insoluble matter, 0·38; and water and organic matter, 3·63. Total, 99·91.

Mixtures of "*pandermite*" with felspar, in proportions varying from 30 to 70 per cent., all gave promising results, but especially so the mixture of 40 parts of "*pandermite*" to 60 parts of felspar. This mixture fuses below 800° C. without loss of boric acid, forming a homogeneous frit suitable for enamels and ceramic work. It is of average hardness and easily pulverisable.

Fluorspar and pandermite, mixed in proportions of 60 parts of the former to 40 of the latter, yielded a very fluid frit, fusing below 800° C. without loss of boric acid or fluorine, and capable of increasing the dilatation of enamels. 50 parts of this frit, 5 of rutile, 30 of sand, 8·6 of sodium carbonate, and 10 of cryolite yielded an enamel of high dilatation, fusing at 1,050° C. and only slightly attacked by acids. It would therefore appear to be suitable in all respects for application to iron. 90 parts of this mixture, used as a frit, with 10 parts of petalite, produced an enamel of high dilatation—1,079, measured by the Le Chatelier method—of sufficient fluidity, and fusing below 830° C. This, therefore, would also seem to be suitable for iron, and might possibly prove useful as a hygienic enamel for hardware.

Mixtures of cryolite with fluorspar and a little rutile, yield enamels of very high dilatation and sufficient fusibility, which may be recommended generally as suitable to metals of high dilatation.

Of simple mixtures, pandermite, sodium carbonate, and sand yield an enamel of which the dilatation is 894, and which might be found useful for cast and sheet iron.

An enamel composed of 80 parts of petalite to 20 of pandermite, of which the dilatation is 483, though somewhat expensive, owing to the large proportion of petalite used in it, is suitable for application to certain porcelains and stonewares of low dilatation.

Enamels of lower dilatation may be produced without having recourse to either cryolite or rutile by means of sand, sodium carbonate, pandermite, and other substances.

The experiments taken as a whole show that, by the use of boric acid, it is quite possible to produce hygienic calcareous enamels, free from lead and suitable for application to iron, as well as others of varied dilatation and fusibility, some of which are but very slightly attacked by acids. It would also appear that natural calcium borate provides an excellent economical substitute for sodium borate, at present almost exclusively used in the enamel and ceramic industries. The lime introduced is not at all prejudicial to the quality of the enamels.—H. H. B. S.

*Blue Glaze, Production of a, by reducing Tungstates in a Porcelain Furnace.* A. Granger. Comptes Rend. 1898, **127**, [2], 106—107.

The first attempts to make use of tungsten compounds in the ceramic industry were made at Sèvres by Lauth, but no practical results were arrived at. Taking into consideration the readiness with which tungstic anhydride is reduced, the author considered that it ought to be possible to produce the oxide $W_2O_5$ during the firing of the porcelain, and that there was room for further experiments in that direction. On adding tungstic anhydride, even in the smallest proportion, to a fusible silicate, such as porcelain glaze or glass, the substance becomes completely white and opaque. In this change the silica is set free, producing the opacity, and a silicotungstate is formed. In order to convert the whole of the liberated silica into silicotungstate, it would be necessary to add so much tungstic anhydride that the glaze would not melt, but would remain in a porous state on the porcelain. The author therefore tried experiments with a tungstate glazing, and chose for the purpose a sufficiently acid tungstate, such as $M_2O.4WO_3$. In order to have a sufficient degree of fusibility, a glaze was composed of a mixture of barium and sodium tungstates, the former by itself being too refractive and the latter too fusible. The proportion of the two tungstates was so calculated that the ratio of the sum of the bases to the anhydride was 1:4. On firing porcelain with this glaze at about 1,250°, blue colours were obtained, the shade varying from light blue to indigo according to the proportion of bases present and the quantity of glaze employed. Other modifications of the colour could be brought about by the introduction of borax or phosphates.—C. A. M.

### PATENTS.

*Kilns or Ovens for Firing Potters' Ware, Bricks, Tiles, and such like, and for Calcining various Substances; Impts. in.* M. G. Malpas, Tunstall, Stafford. Eng. Pat. 19,302, Aug. 20, 1897.

This is an arrangement of kilns on the continuous firing system. A block or series comprises 24 kilns which are arranged in two rows of 12, side by side. Each kiln is in communication with a common flue as well as with the kilns adjacent to it. A travelling fire underneath the kilns enables each to be fired in its turn. The products of combustion pass upwards into the kiln by way of the main firing flue, and, after playing upon the seggars, pass into ring flues at the top of the kiln, from whence, by way of vertical passages in the walls, they pass into the under-flue and so to the adjacent kiln. There are also supplementary ovens arranged on the tops of the kilns for use as hardening-on ovens. These are heated by the products of combustion as they pass from one kiln to the next. Each kiln has, of course, a suitable opening to admit of putting in and taking out the seggars.—H. H. B. S.

*Drying Apparatus for Earthenware Goods, Bricks, and the like; An Improved.* J. Rappold, Zürich. Eng. Pat. 30,333, Dec. 22, 1897.

The apparatus consists of a drying house, preferably of masonry, having a warm-air canal beneath it, heated by a suitable heating apparatus. The dry, hot air is set in motion by fans, the movement of the air being regulated by valves. When the air is fully saturated with water vapour, it enters a condensing tube in which water collectors are placed, from which the condensed water passes away through pipes. The cooled air is then again forced by the fan into the hot passages, where it is once more raised to a high temperature, rendering it capable of absorbing moisture. The drying process is thus based upon the capability of air of absorbing water at a high temperature and giving it up on cooling. The goods are conveyed into the drying house on waggons travelling upon rails.—H. H. B. S.

*The Gilding of Articles of China, Earthenware, Porcelain, and the like; Impts. relating to.* G. W. Tooker, New York, U.S.A. Eng. Pat. 10,128, May 3, 1898.

Equal parts of gold, one part prepared by precipitation from the chloride by mercurous nitrate, and the other part by potassium oxalate, are mixed, and fluxed with a stated proportion of bismuth oxide. The mixture is then mixed with oil of turpentine to a proper consistence for hand-painting. The articles to which this gold preparation have been applied, are fired at about 1,500° F., and, after burnishing, a "grounding oil" is brushed over the surfaces, upon which a mixture of "No. 8 flux" and of "special soft flux" is dusted, after which the ware is again fired at a lower temperature than before. When the decoration is to be applied to the ware by printing or transferring, printing oil is used instead of oil of turpentine for mixing with the gold. If "powdered gold" is to be applied to outlines formed in oil upon the ware, the "potash gold" is mixed with a stated proportion of calomel for the application.—E. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### PATENTS.

*Drying Apparatus for Earthenware Goods, Bricks, and the like; An Improved.* J. Rappold, Zürich. Eng. Pat. 30,333, Dec. 22, 1897.

*See under* VIII., *above.*

*Plastic Material for Use in Making Tiles and other Moulded Articles, and in Making Floors, Walls, and the like; Impts. in.* J. T. Szek, London. Eng. Pat. 6511, March 17, 1898.

Sawdust, wood-shavings, wood-wool, wood-pulp, or similar material, is first steeped in a solution of sulphate of magnesia, with or without the addition of borax. It is then dried and ground, and mixed with magnesia or burnt magnesite; or, if desired for the sake of cheapening the product, the drying and grinding may be omitted. The mixture so obtained may either be used as it is, or it may be further mixed with dilute hydrochloric acid before use. It may be coloured, either by painting the surface, or by incorporating suitable pigments with it.—H. H. B. S.

*Artificial Stone and Concrete, Impts. in the Method of Manufacturing.* L. P. Ford and T. W. Barber, both of London. Eng. Pat. 8226, April 6, 1898.

Improvements in the method of slaking lime and sand in the manufacture of artificial stone. The dry powder is rammed into moulds, which are then placed in a chamber connected with an air-pump and capable of being hermetically closed. The air is then exhausted, and hot water and steam are successively admitted, which has the effect of accelerating the formation of silicate, while maintaining a suitable temperature in the mass. The stone is dried by exhausting the chamber until the moulds are fit for removal. This latter operation may be carried out in a separate chamber if preferred, and the drying may also be accelerated by removing the stone from the moulds. The stone may be coloured by mixing dry powdered pigments with the other ingredients. To make a multi-coloured stone, blocks of stone are first made of the various colours required. These are then broken up, mixed with a fresh quantity of lime and sand, the whole rammed into moulds, and treated as before. In order to increase the hardness of the stone, a dry powdered alkaline silicate may be incorporated with the lime and sand.—H. H. B. S.

*Plaster for Walls, Ceilings, and the like; Impts. in the Manufacture of.* J. A. Jones, Middlesbrough-on-Tees. Eng. Pat. 21,141, Sept. 15, 1897.

Blast-furnace slag (by preference, grey annealed slag from Cleveland iron ores) is used as the basis of the plaster. This is ground with Portland cement, lime, and sand, in proportions depending upon the purpose for which the plaster is required.—H. H. B. S.

*Plaster or Cement for Covering the Roofs and (or) Walls of Buildings, An Improved Composition constituting a.* J. Goodwin, Glasgow. Eng. Pat. 12,902, June 9, 1898.

The composition consists of the following ingredients, mixed in the proportions stated:—

| | Parts. |
|---|---|
| Ground clay | 7 |
| China clay | 3 |
| Fossil-meal | 2 |
| Asbestos | 2 |
| Blue lias lime | 10 |
| Flax or other fibre | 2 |
| | 26 |

It is stated to be specially suitable for covering wood and iron buildings for tropical countries, being both heat-resisting and rainproof, as well as capable of great expansion and contraction without cracking.—H. H. B. S.

## X.—METALLURGY.

*Blast-Furnace Gas, "Simplex" Motor Using.* M. A. Witz. Iron and Steel Inst., Autumn Meeting, 1898.

The report of a 24-hours trial with a 200-h.p. engine at Seraing (this Journal, 1898, 765—766), using blast-furnace gas without any attention as to its condition, shows an effective horse-power per 3½ cb. m. of gas per hour, whilst consuming per hour and per horse-power about 100 litres of water and less than 18 grms. of oil and grease. The working is as regular as that of a steam engine, and the dust in the gas is in no way injurious to its continuous operation.—A. W.

*Iron and Steel, Solution Theory of.* H. Jüptner von Jonstorff. Iron and Steel Inst., Autumn Meeting, 1898.

Since his last paper on this subject (this Journal, 1898, 766—767) the author has seen the data in connection with the thermal relations of iron-carbon alloys, published in the fourth report of the Alloys and Research Committee, and has corrected some of his former figures and conclusions.

He modifies the molecular atomicity theory of the dissolved carbon, and now supposes that with the highest temperature (1,600° to 1,336° C.), and with low carbon percentage (up to about 2 per cent.), the carbon molecule dissolved in iron consists of two atoms, whilst with lower temperature and increasing carbon, more and more three-atom molecules are associated with these. The fluid eutectic alloy, which solidifies at 1,130°, contains these molecules in the proportion showing an average of 2·6 atoms per molecule.

From the fresh figures now obtained in connection with silicon and manganese, the following modified conclusions are arrived at:—(1) The carbon and silicon molecule dissolved in the molten iron, or molten iron and manganese, consists of an equal number of atoms; (2) The atomicity of this carbon and silicon molecule is independent of the quantity of manganese present; (3) It increases, however, with the total contents, expressed in carbon equivalents, of carbon and silicon; (4) In very dilute carbon, and probably also silicon solutions, these molecules consist of two atoms; (5) The mean atomicity of the molecule, increases for each 1 per cent. of carbon, or ⅔ per cent. of silicon, above a certain limit, about 0·25 atom, but with higher percentages of both elements together, about twice this; and (6) it reaches, in the case of a total of about 8 per cent. of carbon and silicon (in carbon equivalents), to 5 atoms.—A. W.

*Cast Iron, The Action of Metalloids on.* G. R. Johnson. Iron and Steel Inst., Autumn Meeting, 1898.

The effects of the various constituents of iron on its physical properties are described, and tables of analyses and physical tests of many Embreville irons are given, showing the effect of one element varying in quantity, whilst the others remain constant. The author sums up as follows:—

If ordinary soft grey castings are wanted, use graphitic iron, low in sulphur and manganese, but with from 0·75 to 1·0 per cent. of phosphorus; if a strong machinery casting be required, use an iron of close No. 3 to grey forge fracture, with silicon from 0·80 to 1·50 per cent., sulphur from 0·03 to 0·05 per cent., and phosphorus from 0·35 to 0·50 per cent. For hydraulic cylinders, &c., take the same composition, but with sulphur as high as 0·075 to 0·110 per cent. For chilled wheels the iron should contain silicon 0·5 to 0·8, sulphur 0·02 to 0·04, and manganese 1·0 per cent.

It should be remembered that while silicon is the governing element in chilled iron, phosphorus has its effect in changing graphitic into combined carbon. The low-phosphorus iron is best for making car wheels, chilled rolls, &c., since the chill fracture shows the white or chill part interlacing with or piercing the grey portion, whereas in the high-phosphorus iron, the line between the two parts is sharply defined, and the metal consequently weak.
—A. W.

*Cementation [Steel], Microchemistry of.* J. O. Arnold. Iron and Steel Inst., Autumn Meeting, 1898.

The main feature of this paper is a series of coloured micrographs of sections of cementation steels showing the different degrees of penetration of the cementation from the outside to the centre of the bars, and the constituent carbides of the same. The micrographs are both of the polished and etched surfaces, and show very clearly the structure of, and the carbide distribution in the metal. The process of cementation involves many problems, especially the form and manner in which the carbon diffuses from the charcoal into the hot iron, and in this question the author adopts the word "interpenetration" as being a suitable expression which does not prejudge the nature of solid diffusion. It would appear to be scarcely possible to introduce much over 2 per cent. of carbon into cemented iron, even after 10 conversions. The cementation up to the saturation point is very rapid, but afterwards the supersaturation proceeds at the rate of only about 0·05 per cent. of carbon per day.

From his researches on the diffusion of all elements through hot iron, the author makes the following provisional statements in respect to carbon:—(1) That the diffusion of carbon in the process of cementation presents two distinct varieties of interpenetration, (*a*) the interpenetration of the substance corresponding with the formula $Fe_{24}C$ and pure iron, and (*b*) the interpenetration of the normal carbide $Fe_3C$ and the sub-carbide $Fe_{24}C$; (2) That the interpenetration of $Fe_{24}C$ and iron is more rapid and takes place at a lower temperature than the interpenetration of $Fe_{24}C$ and $Fe_3C$; and (3) (*a*) That the interpenetration of $Fe_{24}C$ and iron begins at Ar 2 (about 759° C.) and is coincident with the evolution *in vacuô* of gases from the iron, and (*b*) that the interpenetration of $Fe_{24}C$ and $Fe_3C$ does not take place until the temperature of about 950° C. has been reached.

—A. W.

### *Steel, Diffusion of Sulphides through.* E. D. Campbell. Iron and Steel Inst., Autumn Meeting, 1898.

THE author, with the assistance of Mr. C. Sundström, has experimented further on the diffusion of sulphides through heated steel, this time using a mixture of 5 parts of an oxysulphide, containing a little manganese, and 3 parts of cuprous sulphide. On plugging this in a cavity in the steel and heating in an oxidising atmosphere for about two hours, after raising the temperature to 1,200° C., as before, it was found that practically the whole of the sulphide had disappeared. Estimations of the copper showed that 86·78 per cent. of it had diffused right through the steel into the asbestos mat on which it rested, 3·84 per cent. was in the scale formed on the outside of the bar, and 5·25 per cent. was in the bar itself, thus accounting for 95·87 per cent. of the total quantity taken. When the atmosphere around the bar was non-oxidising, the diffusion was not so rapid.

The condition of the surface of the cavity in which the sulphide is placed also affects the diffusion, inasmuch as when the surface is smooth it is only slight, whereas with the same surface roughened with hydrochloric acid, washed, and dried, the diffusion was rapid. The temperature is also important, since at 1,100° diffusion scarcely takes place, and only after 1,200° is reached is it complete.—A. W.

### *Soft Steel, Brittleness Produced in, by Annealing.* J. E. Stead. Iron and Steel Inst., Autumn Meeting, 1898.

THIS paper is written to show that a scientific investigation of the causes of brittleness is of great practical importance, and also to induce practical workers to pay some attention to a subject that might prove to be of great economic value. For the latter reason a terse review of the explanations of fractures according to crystalline structure is given. First, there is intergranular weakness, wherein the fracture follows the weakness between the grains of metal, of which there are two kinds, *viz.*, (*a*) where the grains join and touch one another, and (*b*) where the grains do not touch, but are encased in envelopes of substances which separate out on solidification of the metal. Second, there is the inter-crystalline weakness, which is represented by the true cleavage planes of the grains as a mass, and in which the fracture follows these planes and not the junction of the grains. In this connection there is strong evidence that pure iron consists of cubic crystals and not octahedra.

It follows that if the cleavage planes of the crystals or grains were in the same plane, the metal would be more liable to fracture from shock than if they were at different angles to one another. Hence a fine-grained metal in the former state might be much weaker than a coarse-grained and largely crystalline metal, in which the cleavage planes are arranged heterogeneously; although the finer the grain, other things being equal, the stronger should be the metal. This has a great bearing on the brittleness produced in iron by annealing, during which operation the metal becomes more granular.

Some bars of tin-plate steel were annealed at 700° C. for 48 hours, when the grain became larger, especially towards the outer edge, and the metal was easily fractured. On again heating to 900° and cooling naturally, the bars became exceedingly tough, and the external layer of coarse granular steel was transformed to a finer grain similar to the centre of the bar, leaving a coarse-grained portion between them. The strength of these bars lay in the fine-grain envelope, into which the brittle annealed material had been changed, which, however, now contained only traces of carbon in place of the 0·08 to 0·12 per cent. originally present. But it was found that the brittle portion was not always removed by re-annealing.

The examination of a large number of annealed sheets has revealed the facts that when their thickness is less than 18 B.W.G., brittleness is never developed, and that it is *occasionally* developed in sheets from 10 to 20 B.W.G. Further, that thicker sheets, which are not brittle on leaving the annealing pots, after heating to 400° or 500° and being subjected to shock, which may or may not be sufficient to give a permanent set, almost invariably show a development of brittleness. The character of the weakness is sometimes inter-granular, but more frequently inter-crystalline. In rolled sheets there is a fixed relation between the lines of fracture and the direction of rolling. Previous to annealing there is no tendency for these sheets to break up in rectangular directions, but rather to be fibrous and tough; whereas, after annealing, the structure is coarsely granular and shows a rectangular fracture. It would appear that rolling impresses a latent disposition in the steel to crystallise in certain fixed positions, and that the annealing afterwards develops it.

The cause of inter-granular weakness cannot yet be fully defined, but in two or three cases examined, the phosphorus was found to be excessive, and, as a consequence, it should not be allowed to exceed 0·08 per cent. In sheets where, in a small width of $\frac{1}{16}$ in., there exists both tough and brittle material, it is obvious that chemical composition does not play an important part, and it would seem that the absence of homogeneity is due to uneven heating in the annealing pots; so that the primary factor apparently is the temperature. Rolling a sheet in different directions, or at different angles from the straight, ought to reduce brittleness by altering the trend of the crystals; but, of course, it is impracticable, owing to the consequent waste of metal as scrap.—A. W.

### *Steel Guns, Action of the Projectile and of the Explosives on the Tubes of.* W. C. Roberts-Austen. Iron and Steel Inst., Autumn Meeting, 1898.

THE author has examined the eroded surface of the rifle-bore of a quick-firing gun, with a view to explain the change which has taken place, and its cause. It must be remembered that a high temperature is suddenly transmitted to the inner surface of the tube by the heat of explosion, and is rapidly abstracted by the mass of metal behind. Consequently, the change might be a complicated one, due to this thermal disturbance. In addition, there is the mechanical effect of the projectile passing along the tube. The metal was a gun steel containing 0·3 per cent. of carbon, 0·6 per cent. of manganese, less than 0·05 per cent. of sulphur and phosphorus, and not more than 0·15 per cent. of silicon. It had been oil-quenched at 800° C., and subsequently annealed at 500°. A micro-examination of such a steel should reveal more or less confused pearlite, produced by annealing, traversed by a network of ferrite. The transition forms, *Troostite* and *Sorbite*, might be present, but no *Martensite*. The micro-section of the gun showed the mass of metal to be in this condition, but at the edge of a "land" or the driving edge of a grove, the structure was different. The altered layer was only from 2/1000ths to 2/100ths of an inch deep, and the cross section of this was prepared by embedding the metal in steel and polishing the whole.

The examination proves that the action of the explosive is purely mechanical, so far as the mass of the tube is concerned, and there is no evidence of fusion nor of the formation of martensite; but in the altered layer, the ferrite bands have disappeared, and the carbides are present in a confused mass. If the steel had been superficially changed into ordinary hardened steel, the altered layer would consist of *Martensite*, but this could not be detected. Probably, the time of the operation was too short to permit the complete diffusion of the carbon in the layer. It is of course possible that the layer is a deposited one, but against this view there is the fact that the ferrite bands gradually fade away, in some cases, as the layer is approached, and in others, pass into it in fine lines.

In the case of an armour plate, which had been struck by a glancing shot, the micro-structure was not altered by the impact.—A. W.

*Gold Amalgamation, Note on the Influence of Temperature on.* F. F. Sharpless. Eng. and Mining J. 1898, **66**, [7], 183.

With regard to the known influence of temperature upon amalgamation, the author records his experience with a mill started in March of the present year. About the 1st of June it was noticed that the amalgam, instead of remaining in a plastic condition, formed a hard crystalline or granular alloy, that adhered with great persistence to the silvered apron plates. This could not be worked off with a stiff brush, and only very incompletely by prolonged rubbing with a rubber or cloth; for complete removal, a knife or other scraper was necessary. The inside plates, however, which were of plain copper, remained in good condition throughout the run. The apron plates continued to grow harder, and the tailings to contain more free gold, until, with the water at 83° F., it was found necessary to stop working. The author states that portions of the apron plates where all the silver had been worn off, remained throughout in good amalgamating condition, and he suggests that this points to the fact that raw copper plates would give better results than silvered ones when working with warm water.—A. S.

*Cyanide-Mill Solutions, Analyses of.* W. J. Sharwood. Eng. and Mining J. 1898, **66**, [8], 216.

The analyses are given of the solutions from four mills, all of the solutions being used in percolating vats except No. 9; lime was added in all cases before the cyanide treatment, and the samples were taken after precipitation of all but traces of the gold and silver by means of zinc.

The materials which had been treated by the different solutions were: (1) Carefully concentrated tailings almost entirely free from sulphides; (2) Tailings containing much oxide of iron but only traces of sulphur; (3)—(7) Tailings containing refractory sulpharsenides and sulphantimonides of silver and copper with which the gold was associated, and also carbonate of copper. In No. 3 an oxidising agent had been added to the cyanide; in Nos. 4 and 5 the "strong" and "weak" solutions were respectively used continuously in a short run, and in Nos. 6 and 7 in a longer run. The ore treated by No. 8 was fine-crushed, to favour battery amalgamation; the pulp was then separated by a point box and passed over concentrators. The finer slimes were run to waste; the coarser portion was allowed to settle, and dried before cyaniding, a considerable proportion of finely-divided sulpharets remaining. Owing to the fine crushing, it was found impracticable to leach without sizing and drying, and the solution had to be run to waste after using it three times, on account of the rapid accumulation of salts. No. 9 solution was used only once in an agitator on highly sulphuretted pulp from the same mine as No. 8.

The sulphurets in the ore treated by Nos. 8 and 9 consisted chiefly of magnetic pyrites, with a little copper pyrites, galena, and blende; the pyrites from this mine being particularly liable to oxidation.

Traces of magnesium were found in Nos. 2, 3, and 9; arsenic in Nos. 3 and 6; antimony in Nos. 1 and 2; and cobalt in No. 1. No carbonate was present except traces of $CaCO_3$. Sodium, potassium, and chlorides were not estimated. Traces of ammonia were present in all cases, and more complex organic bodies were found in Nos. 1, 2, 6, and 7, which had been used for the longest periods. Free cyanide was estimated by titrating with silver nitrate, with ferrocyanide as indicator, and total cyanogen by continuing the titration after addition of caustic soda and a little ammonia and potassium iodide; this latter, however, does not include the cyanogen present in double cyanides of copper, silver, gold, or mercury.

*Analyses of Cyanide-Mill Solutions. (The Amounts given are Grms. per 100 c.c.)*

| — | (1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) | (9) |
|---|---|---|---|---|---|---|---|---|---|
| Time used | 8 months | 6 months | 3 times | 12 days | 12 days | 4 months | 4 months | 3 times | Once |
| Tons of solution used | 150 | 200 | 10 | 500 | 400 | 500 | 400 | 30 | 5 |
| Tons of material treated | 16,000 | 17,000 | 30 | 5,000 | 5,000 | 45,000 | 45,000 | 100 | 6 |
| Mesh per linear inch | 30 | 30 | 30 | 30 | 30 | 30 | 20 | 60 | 60 |
| Nature of material treated | Clean tailings. | Tailings with much $Fe_2O_3$. | Tailings with some sulphides, &c. | Same as 3 | Same as 3 | Same as 3 | Same as 3 | Tailings with pyrites. | Pulp with much pyrites, &c. |
| Nominal working strength KCy | 0·25 | 0·20 | 0·30 | 0·25 | 0·08 | 0·20 | 0·08 | 0·5 | 1·0 |
| Nominal working strength =Cy | 0·10 | 0·08 | 0·12 | 0·10 | 0·032 | 0·08 | 0·032 | 0·2 | 0·4 |
| Cyanogen (in KCy and $K_2ZnCy_4$) | *0·037 | *0·045 | *0·074 | 0·13 | 0·05 | 0·164 | 0·055 | *0·095 | *0·05 |
| Zinc | 0·027 | 0·028 | 0·060 | 0·05 | 0·025 | 0·071 | 0·030 | 0·006 | 0·034 |
| Copper | 0·001 | 0·015 | 0·022 | 0·030 | 0·019 | 0·082 | 0·053 | 0·009 | 0·005 |
| Calcium | 0·015 | 0·034 | 0·060 | 0·066 | 0·0575 | 0·125 | 0·109 | 0·065 | *0·010 |
| Iron | 0·0005 | 0·0002 | 0·00035 | 0·004 | 0·00025 | 0·0006 | 0·0003 | 0·022 | 0·013 |
| Ferrocyanide | 0·0033 | 0·0013 | 0·0023 | 0·00264 | 0·00165 | 0·004 | 0·002 | 0·145 | 0·0855 |
| Thiocyanate | 0·0 | 0·0 | Traces | 0·0072 | 0·0036 | 0·035 | 0·025 | 0·065 | 0·025 |
| Sulphates (as $K_2SO_4$) | 0·0022 | 0·036 | 0·013 | 0·0135 | 0·020 | 0·0118 | 0·0365 | 0·250 | 0·0275 |
| Residue on evaporation | 0·139 | 0·310 | 0·478 | 0·581 | 0·377 | 1·045 | 0·624 | 1·020 | 0·408 |
| Residue after ignition | 0·119 | 0·280 | 0·412 | 0·570 | 0·367 | 0·961 | 0·580 | 0·907 | 0·321 |
| Alkalinity (c.c.'s of decinormal acid to neutralise 100 c.c.) a | 10 | Not observed | 30 | 24·5 | 18·5 | 84 | 56 | 32 | 22 |
| b | 16 | | 42 | 40 | 30 | 118 | 86 | 54 | 45 |

* These solutions had not been fortified since last used.

Calcium was determined by titrating the precipitated oxalate with permanganate, or by igniting it and weighing as oxide.

For the determination of iron, copper, and zinc, 100 c.c. of the solution were twice evaporated with nitric acid, redissolved in dilute sulphuric acid, the iron precipitated by ammonia, filtered off, redissolved in hydrochloric acid, and estimated colorimetrically as thiocyanate, or, if present in considerable quantity, by titration with permanganate, after first reducing with zinc. The copper was separated from the ammoniacal filtrate by acidifying with sulphuric acid and heating with a strip of aluminium. The deposited copper was washed, dissolved in nitric acid, and determined by the iodide-thiosulphate method. The filtrate from the copper was neutralised with sodium carbonate, acidified with a definite quantity of hydrochloric acid, diluted to 200 c.c., heated, and the zinc determined with ferrocyanide, with uranium indicator.

Thiocyanate was determined colorimetrically by means of ferric chloride, after first acidifying with hydrochloric acid; in some cases ferrocyanides were precipitated and had to be filtered off.

Sulphate was estimated by adding excess of hydrochloric acid, heating till all odour disappeared, filtering, and precipitating with barium chloride.

The solid residue was determined by evaporating 20—50 c.c. in a nickel or platinum dish; the former appears to be less attacked by cyanide solutions and fused residues. The alkalinity was determined with methyl orange as indicator—(*a*) by direct titration with decinormal acid, (*b*) by adding excess of acid, heating till all odour disappeared, and titrating back with standard alkali; the results were rendered somewhat uncertain by the precipitation of zinc compounds and ferrocyanides.—A. S.

*Gold in Nicaragua.* C. T. Mixer. Eng. and Mining J. 1898, **66**, [5], 125.

The author briefly describes a few of the most important properties:—

The Hidden Treasure, owned in Bluefields, has a 14 ft. vein, and is at present provided with a 3-stamp mill. Much ore from this property is said to have an assay value of 100 dols. to the ton.

The Siempre Viva, owned in Prinzapulka, has a 10-stamp mill, with water power in the wet season; vein 6—8 ft. wide; average value of milling ore, 20—30 dols. per ton. As much as 300 oz. of bullion a month has been produced.

The Concordia has 8 stamps, water power, three veins, 3 ft., 4 ft., and 12 ft. wide, and has ore similar to the Siempre Viva.

The Constancia, owned at Cape Gracias, has two 14-ft. arrastras, and is said to pay well.

The Atlas group has a 5-stamp mill, water power, an exceedingly wide ledge, and is at present experimenting with the cyanide process.

The Cape Gracias district has not been thoroughly explored, but good finds have been made there, placer nuggets being frequently shipped to Bluefields, some of which have weighed as much as 27 oz. The Jumbo Mine, owned at Bluefields, is situated about 38 miles from the coast, on the Wawa River. There are two ledges, 7 ft. and 11 ft. wide, and a 6-stamp mill.

The latest discoveries of gold have been on the Rama River, a tributary of the Escondido or Bluefields River, of which the most promising property is situated near the Mico River. The quartz ledge crops boldly across the country, and everywhere shows the presence of gold. It was cut by a tunnel 50 ft. below the apex, and was found to be several feet wide and to show up well in gold content. A lower tunnel is now being driven, and it is expected that the lead will slow up even better, and warrant experiments being made with a view of erecting a cyanide mill.

No duty is imposed on mining implements, and the recent export duty of 50 c. per oz. on gold, is expected to be shortly repealed.—A. S.

*Tin Slags, Extraction of Tin from, by Sulphuric Acid.* E. Bohne. Berg- u. Hüttenmänn. Zeit. 1898, **57**, 203.

In the ordinary furnace processes the amount of tin in tin-slags cannot be reduced below about 4 per cent. The author granulates these slags by pouring them from off the surface of the metal in the furnace into cold water. The finely granulated or powdered slag is treated in lead-lined wooden vessels with hot dilute sulphuric acid, which yields a partly gelatinous, partly granular, residue of silica, containing a little tin, and a solution of tin and iron. This solution, after separation from the residue, is electrolysed to separate the tin, and the mother liquors are then evaporated to furnish crystallised ferrous sulphate. The silicious residue is used up in making the bed of the furnaces.

—J. T. D.

*Copper, Estimation of Oxygen in.* M. Lucas. Bull. Soc. Chim. 1898, **19**, [16—17], 795.

*See under* XXIII., *page* 952.

*Telluride Ores [Gold], Assay of.* C. H. Fulton. School of Mines Quarterly, **19**, [4], 419.

*See under* XXIII., *page* 951.

*Enamels of High Dilatation for Cast or Common Iron on the Basis of "Pandermite."* M. Saglio. Bull. de la Soc. d'Encouragement, 1898, **3**, [8], 1060.

*See under* VIII., *page* 924.

*Platinum, Precipitation of, from its Solutions.* A. Atterberg. Chem. Zeit. 1898, **22**, 538; Analyst, 1898, **23**, [270], 247.

*See under* XXIII., *page* 951.

*Lead in Lead Ores, Determination of.* L. Schneider. Oesterr. Zeits. Berg-u. Hütt. **46**, 431; Chem. Centr. 1898, **2**, [8], 559.

*See under* XXIII., *page* 951.

*Iron and Manganese in Ores, Determination of.* P. Lehnkering. Zeits. für öffentl. Chem. **4**, 959; Chem. Centr. 1898, **2**, [7], 507.

*See under* XXIII., *page* 951.

## PATENTS.

*Ingots, Impts. in the Method of Casting, and in the Moulds and Appliances connected therewith.* J. O. Arnold, Sheffield. Eng. Pat. 23,668, Oct. 14, 1897.

A vertical runner-box, preferably rectangular in section, is mounted on a suitable bed-plate; one of the sides is removable, being held in position by clamps; a series of pairs of recessed openings are provided opposite one another on opposite sides of the runner-box. Into each of these openings is fitted the open end of a slightly tapered, close-ended, horizontal ingot mould, the surfaces of mould and box being made true on the plane of contact. These moulds are clamped to the box at the one end, and are supported at the other end on trestles fitted with movable shelves. If preferred, the runner-box may have lugs instead of recesses for carrying the moulds. The moulds have narrow vents either at their extreme outside ends or on the upper sides near the runner. When the runner-box has been rammed, channels are cut, either horizontal or inclined slightly upwards, to connect the runner with the moulds. There is thus a double series of superposed horizontal ingot-moulds connected to one vertical runner. In pouring, each pair of moulds acts as a head to the pair below, the topmost pair depending for a head upon a prolongation of the runner-box. The object of the invention is to prevent the formation of blowholes, to prevent liquation, to avoid piping, and to produce ingots with a less proportion of head and runners than is usually necessary.

—W. G. M.

*Iron or Steel Plates or Sheets, to be afterwards Coated with Tin or Terne Metal, or other Coating Metal or Alloy, or to be otherwise Coated, or used Uncoated; Impts. in the Treatment of, and in Apparatus employed in the said Treatment.* R. B. Thomas, Chepstow, and H. S. Thomas, Lydbrook. Eng. Pat. 23,883, Oct. 16, 1897.

This invention is intended to facilitate the "black pickling" of plates or sheets. The plates are passed singly through the acid bath, being carried forward automatically by studs on the periphery of rotating wheels, or by fingers attached to a travelling endless band, or by a series of rollers, after which they are conducted one by one, through guides to a pair of rollers, which feed them into a swilling bosh, where they are subjected to the action of jets of water. If preferred the plates may be dropped singly into a rising and falling trough, which lowers them into the acid bath, and when at the lowest position, allows them to fall over against guides, so that when it rises again they are fed into rollers, which lift them out of the vat. The vats and swilling tanks are warmed externally by grates, or internally by hot water or steam pipes. If preferred, either pickling or washing may be conducted in bulk, the process being applied only to one part of the treatment.—W. G. M.

*Steel, An Improved Process for Hardening.* W. P. Thompson, Liverpool. From G. Behr, Cologne, and T. Wallfisch, Magdeburg. Eng. Pat. 11,992, May 26, 1898.

The article to be hardened is coated, before being heated, with a substance composed of waste lime (or ground clay, white lead, zinc white, or the like) and a suitable binding material, such as varnish, with the object of forming a protective casing for the article. Lead shavings are mixed with the substance, or the casing is sprinkled either with potassium cyanide before heating, or with cyanide and coarse salt during heating. The effect of the casing is to render the action of the cyanide uniform, whilst the surface of the article itself does not warp or crack. The crust cracks off spontaneously on cooling.—W. G. M.

# XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## (*A.*)—ELECTRO-CHEMISTRY.

*Electrolysis, Secondary.* E. Andréoli. Ind. Electrochim. 1898, **2**, 36—38; Science Abstracts, 1898, **1**, [9]. 565

The author observed that if an electrolytic cell be divided into three compartments by means of porous partition walls, and be filled with the same electrolyte, a current passed through by means of electrodes in the two outer compartments, does not produce any change in the central compartment of the cell. If, however, one or more insulated metal plates be immersed in the liquid in the latter, electrolytic decomposition occurs, and to this is given the name of "secondary electrolysis." The following examples are given:—

1. Deposition of gold from a gold-cyanide solution placed in the central compartment, lead plates being used to receive it:—In the outer compartments, solution of sodium chloride with gas-carbon anode and iron cathode.

2. Deposition of gold from gold-cyanide solution in the central compartment, the outer compartments being also filled with the same solution:—No alteration in the gold-content of the solution in the outer compartments could be detected, even after several days.

3. Conversion of sodium bisulphite into hydrosulphite:—The side compartments may contain any electrolyte; the central compartment is fed with a solution of the bisulphite, and only two metal plates are immersed in it. Bleaching operations may be effected by means of the liberated hydrosulphite.—A. S.

*Phenyl-β-hydroxylamine, Electrolytic Preparation of.* F. Haber. Zeits. f. Elektrochem. 1898, **5**, [6], 77—78.

The author prepared phenyl-β-hydroxylamine in the following manner:—A porous pot, containing dilute sulphuric acid and a platinum anode, was placed inside a wider cylindrical vessel containing the solution to be electrolysed, and a cathode, which consisted of a thin platinum tube, wound spirally round the porous pot, and having an area of 50 sq. cm. exposed to the electrolyte. The whole was kept cool in a mixture of ice and salt, whilst at the same time a current of ice-cold water was sent through the platinum spiral, and thence through a U-tube placed in the anode compartment. The electrolyte in the cathode compartment was prepared by dissolving 20 grms. of nitrobenzene in 130 c.c. of glacial acetic acid, and diluting to 850 c.c. with water. In each experiment 300 c.c. of this electrolyte were used. If the dilute sulphuric acid be placed in the porous pot some time before it is required, so that the walls of the pot become saturated with the acid, and not *vice versâ*, the electromotive force of the ordinary domestic supply will be sufficient for the electrolysis. The current strength used by the author was four ampères, and a current of hydrogen was blown through the cathode compartment to ensure a uniform low temperature.

The yellow liquid was then extracted with ether and water, and the ethereal solution washed with water, then agitated with ice-cold water containing suspended magnesia, to remove free acid, dried with calcium chloride, and evaporated. The oil so obtained was treated with light petroleum spirit to remove nitrobenzene and azo oxybenzene, when the phenyl-β-hydroxylamine immediately crystallised out in fine needles. It may be further purified by dissolving in benzene and reprecipitating with petroleum spirit. The yield from each experiment varied from 0·5 grm. to 0·9 grm. with a consumption of about 10 ampère hours.

*o*-Tolylhydroxylamine has been prepared from o-nitrotoluene in a similar manner.—J. S.

*Silicide of Tungsten.* E. Vigouroux. Comptes Rend. **127**, 393.

*See under* XXIV., *page* 961.

*Alkali Chlorides, Electrolysis of.* H. Wohlwill. Zeits. f. Elektrochem. 1898, **5**, [5], 52.

*See under* VII., *page* 923.

## PATENTS.

*Ozone, Generating of* [*Electrically*], *and Apparatus therefor.* H. H. Leigh, London. From H. Abraham and L. Marmier, Paris. Eng. Pat. 20,572, Sept. 7, 1897.

"An ozone generator" is claimed, "all the electrodes of which are circular or rectangular, and give off electric effluviæ from their two surfaces, and the centres of which are provided with an opening for the sucking up or delivery of the gases submitted to the effluviæ substantially as described and illustrated." Six claims relate to specific combinations of parts of the apparatus, methods of cooling by water or some other fluid, and a seventh to "The combination of the ozone generator with an auxiliary generator of low power, actuated by the current discharged by the main ozone generator by means of a small auxiliary transformer . . ."—J. C. R.

*Insulating Material* ["*Viscose*" *and Bituminous Matter*] *for Electrical Purposes, Manufacture of.* C. Beadle, Erith, Kent. Eng. Pat. 22,022, Sept. 25, 1897.

"Viscose," on evaporation, becomes a solid known as "viscoid," which can be employed as an insulating material for electrical purposes. With the object of obtaining a better and cheaper solid than "viscoid," it is proposed to intimately mix with "viscose," bituminous matter such as tar, pitch dissolved in coal tar, or pitch itself, or other bituminous substances melted and mixed with heated "viscose." Bituminous coal, finely pulverised, and mixed with the "viscose" by active agitation, may also be employed. A material suitable for certain purposes can be made with as little as 5 to 7 per cent. of cellulose or as much as 95 per cent. of cellulose. Claim: "An insulating material consisting of 'viscose' having bituminous matter compounded with it substantially as described."—J. C. R.

*Compound or Material applicable for Electrical or Heat Non-conducting or Insulating Purposes, A New or Improved.* H. J. Brown, South Darenth, Kent; O. R. H. Schmidt, Forest Hill, Kent; and A. Mackenzie, Juniper Green, Midlothian. Eng. Pat. 22,610, Oct. 2, 1897.

The compound consists of waste mica and silk or other suitable fibrous material reduced to a fine state of division and mixed with resin, pitch, gum, or similar cementing or binding material.—H. H. B. S.

*Electrolytic Processes, Electrodes* [*Platinum, &c.*] *for; Impts. in the Manufacture of.* A. Zimmerman, London. From The Chemische Fabrik auf Actien vormals E. Schering, Berlin. Eng. Pat. 23,352, Oct. 11, 1897.

The claims are:—

I. The manufacture of electrodes by coating glass, porcelain, mica, enamel, stoneware, or other similar material, with an alloy of the noble metals, platinum, gold, &c., "with bismuth and other metals," &c.

II. "Strengthening the metallic coating of the electrodes forming the subject of the first claim by electrolytic or galvanic deposits of the noble metals."

Examples: (1) "95 parts of fine platinum powder and 5 parts of basic bismuth nitrate are rubbed with thick oil (resinous turpentine oil) to a uniformly thin pulp, which is placed by means of a brush upon a backing such as porcelain and the like, and burnt in, in a muffle. (2) 94 parts of fine platinum powder, 1 part of basic bismuth nitrate, and 5 parts of boric acid or 5 parts of anhydrous phosphoric acid, are rubbed and burned as in example 1. (3) 95 parts of platinum dissolved in sulphur balsam, and 5 parts of bismuth dissolved as an organic salt in oil, are mixed together, the solution being laid on by means of a brush, and, after drying, burnt in, in a muffle."—J. C. R.

### (*B.*)—ELECTRO-METALLURGY.

*Metals [Copper], Electro-deposition of.* J. C. Graham. Elect. Rev. 1898, **42**, 278—279, 319—320, 357—358, and 458—460; Science Abstracts 1898, **1**, [9], 564.

The results are given of experiments carried out chiefly with regard to the electro-deposition of copper. It is stated that in consequence of the small compensation by molecular motion for the dilution caused by the deposition at the cathode, copper cannot, under ordinary circumstances, be satisfactorily deposited if the current-density at the cathode much exceed 300 ampères per square meter. The current-density, however, may reach 10 times this value if the electrolyte be given a very rapid circulation, and the cathode be shielded about the edges to prevent the formation of "trees." In the case of copper anodes, the current-density is found to fall very rapidly from its initial value (when high), but this does not occur with lead anodes. If there be a rapid circulation, this drop does not take place. The pressure between the electrodes also varies abnormally unless the electrolyte is in circulation. Examples are given of the deposition of copper from impure solutions. The colour of the deposit is brown if the solution contain much chloride.—A. S.

## XII.—FATS, OILS, AND SOAP.

*Oils, Lubricating, Lubricating Power of.* E. Weiss. Dingler's Polyt. J. 1898, **309**, [4], 76—80.

The judgment as to the suitability of an oil for lubricating purposes is largely based on the results furnished by the viscosimeter, but, in the author's opinion, the viscosity by itself should not be taken as the sole criterion, for, under such circumstances, one would have to assign to paraffin oil a far higher place as a lubricator than to rape oil, which is obviously contrary to the truth.

By means of an apparatus, devised by the author, it is possible to determine the consistency of an oil, and from the result to calculate its "mobility," ("Beweglichkeit") which furnishes a means of judging which of two oils, with the same viscosity, is the more suitable for lubricating purposes. The consistency measurer consists of a small metal disc, about 10 cm. in diameter, which is made to revolve within the oil, by means of clockwork actuated by a weight, the number of revolutions in a given time being dependent on the consistency of the oil. The weight is so chosen that the disc makes 150 revolutions in water at 15° C. in 30 seconds. A weight of about 1,147 grms. is usually required.

On charging the vessel with a thick liquid, and varying the driving weight, the resistance of the liquid is proportional to the rapidity with which the disc revolves, or $P = m\,u$ where P represents the driving force, $u$ the number of revolutions in 30 seconds, and $m$ a constant of the oil under examination. In the case of rape oil this constant was found to be 64·27. For more mobile liquids, it was determined, experimentally, that up to a weight of 2,500 grms., the following formula was applicable: $P = a + b\ u\ 3/2$ in which P and $u$ have the same significance as in the preceding equation, whilst $a = 43{\cdot}5$ and $b = 0{\cdot}60853$.

For liquids the consistency of which lies between that of water and rape oil, the relation between the driving weight and rapidity of revolution is more complex; but here, too, the author shows that there is a constant relation, and arrives at the formula—$P = A\,\mu\,v_\circ\,(1 - \alpha)$ in which A is a constant depending on the dimensions of the consistency apparatus, $v_\circ$ indicates the rapidity of the revolution of the oil in the vicinity of the metal disc, $\mu$ is the coefficient of the friction of the liquid, and $\alpha$ denotes what fraction the rapidity of revolution near the surface of the liquid is of the rapidity of revolution near the disc.

For Engler's viscosimeter the law of Poiseuille holds good. $T = B\,\mu$ in which T = the time taken by the liquid in running out, and B is a constant of the particular apparatus employed.

Incorporating this formula into his previous equation, the author arrives at the formula—$T\,v_\circ = \frac{P\ B}{A} \cdot \frac{1}{1-\alpha}$. If, then, in the examination of a number of oils, the same viscosimeter and consistency apparatus are always employed, and the same weight invariably used, $\frac{P\ B}{A}$ has always the same value, and the product of the number of revolutions and of the time taken in running from the viscosimeter must be proportional to a constant $\frac{1}{1-\alpha}$, which varies with the value of $\alpha$. Thus, $\alpha$ and $Tv_\circ$ furnish a measure of the mobility of a liquid, since the more mobile a liquid, the less difference will there be in the rapidity of its revolution, in different parts of the vessel, Hence the product, $T\,v_\circ$, is termed by the author the "mobility" ("Beweglichkeit").

If the mobility were entirely dependent on the coefficient of internal friction, all liquids with the same viscosity would have the same "mobility," which is not the case. The "mobility" steadily decreases as the temperature rises, and when the consistency exceeds 30 revolutions, fatty oils lose their oily character. The curves representing the changes in the viscosity and consistency at different temperatures do not run parallel, and are of different grades in different liquids. In rape oil, for instance, the viscosity curve appears to be a hyperbola, whilst the consistency curve has an S-formed shape.

In the case of rape oil, the relation between the consistency values U and the viscosity values V can be expressed with approximate accuracy by the formula—

$$(V + 1{\cdot}92)(U - 0{\cdot}08) = 387{\cdot}54$$

and this formula was used to calculate the numbers in the fourth row in the subjoined table, which give the comparative results obtained with that oil at varying temperatures.

| No. of revolutions in consistency apparatus | 17 | 20 | 24 | 28 |
|---|---|---|---|---|
| Minutes (viscosimeter) | 21·0 | 17·3 | 14·2 | 12·0 |
| $Tv_\circ$ .................... | 357·0 | 346·0 | 340·8 | 336·0 |
| Minutes, calculated .... | 20·98 | 17·53 | 14·28 | 11·96 |

| No. of revolutions in consistency apparatus | 30 | 36 | 40 | 50 | 56 |
|---|---|---|---|---|---|
| Minutes (viscosimeter) | 11·15 | 9·0 | 7·9 | 5·8 | 4·5 |
| $Tv_\circ$ .................... | 334·5 | 324·0 | 316·0 | 290·0 | 252·0 |
| Minutes, calculated.... | 11·03 | 8·87 | 7·78 | 5·84 | 5·01 |

The curves of the changes in the viscosity and the consistency caused by dilution are parallel to the curves of those brought about by heat.

A comparison of results given in the original article in tabulated form, shows that, as regards mobility, the substances referred to stand in the following ascending order: soluble glass, glycerin, soap solutions, gum solutions, oils. The oils, compared with one another, have the following classification: rape oil, rape oil containing colophony, melted lard, mineral oil II., liquid paraffin, castor oil, liver oil, mineral oil I. Castor oil is characterised by its "mobility" increasing as it becomes more liquid.

—C. A. M.

*Candle-Nut Oil.* G. De Negri. Oesterr. Chemiker-Zeit. **1**, 202—203; Chem. Centr. 1898, **2**, [7], 493.

The oil extracted from the candle-nut (yield, 62·25 per cent.) by petroleum ether or ether is light yellow, easily becomes rancid, and possesses marked drying properties. It is soluble in ether, petroleum ether, and chloroform, slightly soluble in alcohol, insoluble in acetic acid in the cold, soluble in an equal volume at boiling heat. It is saponifiable by alcoholic potash to a soap completely soluble in water. Specific gravity at 15° C., 0·920—0·926. It is still fluid at 18° C. M. pt. of fatty acids, 20°—21° C.; Sol. pt. of same, 13° C.; saponification number, 184—187·36; iodine number, 136·29—139·34; iodine number of fatty acids, 142·71 — 144·13; refractometer number at 15° C. (Zeiss, Wollny), 76—75·5. Brullé's reaction: no coloration in the cold, but distinct red colour on warming. Baudoin's reaction: no coloration. Becchi's reaction: the oil extracted with petroleum spirit gives an intense brown coloration; that extracted with ether, a very faint coloration.—A. S.

*Edible Oils, Adulteration of: Examination for Cotton-seed and Sesamé Oils.* F. Jean. Ann. Chim. Anal. Appl. **3**, 217; Chem. Centr. 1898, **2**, [9], 584.

*See under* XXIII., *page* 955.

*Fats, Determination of the Ash of.* A. Delecœuillerie. Rev. Pharm. de Flandres, **5**, 65; Pharm. J. 1898, **61**, [1469], 217.

*See under* XXIII., *page* 958.

*Lard, Detection of Cotton-seed Oil in, by Means of the Phytosterol Test.* A. Bömer. Zeits. f. Untersuch. d. Nahr. und Genussmittel, 1898, [8], 544.

*See under* XXIII., *page* 954.

*Fatty Acids, Liquid, The Application of Benzene in the Determination of the Iodine Value of.* K. Farnsteiner. Zeits. f. Untersuch. d. Nahr. und Genussmittel, 1898, [8], 529.

*See under* XXIII., *page* 958.

*Cotton-seed Oil, Oxidised, Notes on; with a Method for the Analysis of Oxidised Oils in General.* W. Fabrion. Zeits. angew. Chem. 1898, [34], 781.

*See under* XXIII., *page* 958.

*Fatty Acids, Liquid, Separation of, in the form of Zinc Salts.* A. Bomer. Zeits. f. Untersuch. d. Nahr. und Genussmittel, 1898, [8], 541.

*See under* XXIII., *page* 958.

### PATENTS.

*Soap, Improved; The Manufacture of an.* E. Wilczynski, Berlin. Eng. Pat. 24,773, Oct. 26, 1897.

Fat is heated with milk of lime until completely converted into the calcium soap. A solution of sodium fluoride is poured over this and the mass heated till the required condition is attained. Suitable proportions of lime and sodium fluoride mentioned, are 4 parts by weight of the former to 6 of the latter. It is stated that the soap thus prepared is readily soluble in water, and has great cleansing and disinfecting properties.—C. A. M.

*Wax-like Substance, by the Action of Oxidising Agents upon a Mixture of Resin and Paraffin; Manufacture of a.* E. Schliemann, Hamburg, Germany. Eng. Pat. 16,472, July 28, 1898.

A resin, preferably American or French rosin, is melted with a suitable proportion of paraffin wax or similar hydrocarbons, and a blast of air or oxygen blown through the mixture. Excessive oxidation is prevented by the presence of the paraffin wax, and the resulting product is a homogeneous unalterable elastic mass, closely resembling wax. The oxidation can also be effected by means of other oxidising agents, such as chromic acid, &c., for the use of which claim is also made.—C. A. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

*Linseed Oil, Some Phenomena of the Drying of, and the Influence of Pigments thereon.* C. Boettinger. Chem. Zeit. 1898, **22**, 558.

When a piece of linen is soaked in linseed oil and hung up in the air for 10 days, the oil dries to a soft but not sticky coating, and its colour is white. If the cloth be first wetted with weak milk of lime, wrung out, and then treated as before, the oil becomes bright yellow, and dries to a hard but still sticky layer. On extracting the cloth with ether, oil is removed, but some alteration-products are left undissolved; for if the colourless, unlimed cloth, after extraction, is heated to 100° C., it becomes bright yellow. This change of colour is very characteristic; it also occurs with the colourless ethereal extract of the plain cloth, with the faintly yellow solution from the limed cloth, and even when the linseed oil has been previously mixed with white lead or zinc white, as described below. The yellow substance, moreover, is no longer soluble in ether, and but slightly so in chloroform: it dissolves in hot linseed oil, yielding solutions resembling those containing resins. The ether removes from the coated linen certain matter volatile at 80° or 90°, while, after evaporation, the residue is a soft yellow solid, like that obtained in linoleum manufacture, having an iodine number of 78·9 when the cloth has been limed, and 58·1 when unlimed. If the cloth, exposed in the above manner, is wetted again with linseed oil, this begins to act as a solvent on the partially dried film; and only when the initial exposure has been much more prolonged, is the coating comparatively insoluble in ether or oil. After a second exposure of a month's duration, far less matter is found soluble in ether, and the dried extracts have an iodine number of about 48.

The zinc white and white lead employed by the author were of several different makes, the true specific gravity of the former ranging between 5·076 and 5·733; of the latter, between 6·574 and 6·876. The apparent density of the white lead was 1·377 to 1·534 grms. per c.c., while that of the zinc oxide was only 0·421 to 0·495; so that, as each particle of the pigment is normally covered with a film of air, much more oxygen must be added to linseed oil when zinc white is ground into it than when the same weight of white lead is used. Yet this does not appear to affect the drying. 10 grms. of oil were mixed with 5 grms. of each colour, the paint was stirred up twice a day for 4 weeks, and the gain in weight noted at intervals. The mean of the best results was as follows:—

| Days. | Zinc White. | White Lead. |
|---|---|---|
| | Per Cent. | Per Cent. |
| 14 | 1·3 | 2·9 |
| 21 | 2·4 | 4·0 |
| 28 | 3·3 | 5·0 |

After the 28th day, the samples were extracted with ether, which left a variable amount of insoluble organic matter that could not readily be estimated. The solutions were evaporated and dried at 100° C., when they turned yellow, as before. The average specific gravity of the oil recovered from the zinc paint was 0·967, and its iodine number 119·1; from the lead paint, 0·9838 and 109·1 respectively. But whereas these two constants in each case varied inversely one with another, there seemed no connection between the increase in gravity of the oil and the gain in weight of the paint. The ash in one specimen of zinc oil was 0·46 per cent., in a lead oil 0·16 per cent.; the acid and saponification numbers of another zinc oil were 28·2 and 209·2; of a lead oil, 15·4 and 198·1.

10 grms. of castor oil (sp. gr. 0·9671, iodine number 88·3) mixed with 10 grms. of a steel-gray "Panzerschuppenfarbe" formed a paint which changed very little on exposure; after three months the ethereal extract still had an iodine number of 73·7. Two samples of blown castor oil (sp. grs. 0·9813 and 0·9911, iodine numbers 79·7 and 74·4), mixed with half their weight of the same pigment, gave extracts having the sp. grs. 0·9877 and 0·9993, and the iodine numbers 73·6 and 72·1 after $3\frac{1}{2}$ months; whereas raw linseed oil under like conditions altered very considerably, the iodine number of the extract falling to 103·5 in $2\frac{1}{2}$ months, and to 60·8 in $3\frac{1}{2}$. Similarly treated, oxidised linseed oil gained in density from 0·9623 to 0·9667, and from 0·9598 to 0·9626; the iodine number falling from 135·2 to 130·7 and from 141·6 to 138·9. Silver bronze and umber also act energetically upon raw linseed oil.—F. H. L.

*Red Lead, Assay of.* Baucher. Petit. Monit. de la Pharm. **47**, 2651; Pharm. J. 1898, **61**, [1469], 238.

*See under* XXIII., *page* 953.

*Chrome Yellow, Commercial; Analysis of.* Willenz. Bull. Assoc. Belge des Chim. **12**, [4], 163.

*See under* XXIII., *page* 953.

### PATENT.

*Paint, An Anti-corrosive Damp-resisting.* T. Turnbull, Wormit, co. Fife, N.B. Eng. Pat. 10,263, May 5, 1898.

Virgin pitch, 200 lb.; crude naphtha, 27 galls.; dissolved para rubber, 18 lb.; sifted whiting, 10 lb.; resin, 10 lb. Mixed together and used as an ordinary quick-drying waterproof paint.—F. H. L.

## (*B.*)—RESINS, VARNISHES.

### PATENT.

*The Production of Chemical Compounds from a certain* [*Tung*] *Oil or Oils, or the Fatty Acids of same; Impts. in, and the Employment of such Compounds in the Manufacture of Drying Oils, Varnishes, Enamels, Lacquers, Anti-fouling Compositions, and the like.* S. G. Rosenblum and "The Commercial Ozone Syndicate, Ltd.," London. Eng. Pat. 12,508, June 3, 1898.

This is an amplification of Eng. Pat. 16,147, 1897 (this Journal, 1898, 775); the process for manufacturing the various substances being as previously described. The present claims are for the preparation of metallic compounds of tung oil; for the use of the lead or manganese salt as a drier and a coloured or plain varnish or lacquer; for the aluminium salt as a waterproofing material; and for the addition of the mercury or other poisonous salt to anti-fouling compositions.—F. H. L.

## (*C.*)—INDIA-RUBBER, &c.

### PATENTS.

*India-rubber, Gutta-percha, and other Natural Elastic Substances; Impts. in the Treatment of.* C. Repin, Paris. Eng. Pat. 12,327, June 1, 1898.

Tung oil, with or without the addition of linseed or other oil, is gradually heated to 250° C., when it is converted into a solid body. The residue is moistened with "petroleum" to render it more friable, and, after the hydrocarbon has evaporated, the dried oil is mixed with india-rubber, &c., and the solidification completed by heat or some compound of sulphur.—F. H. L.

[*Waterproof*] *Composition, An Improved, and Process for its Manufacture.* J. Macneil, Glasgow. Eng. Pat. 15,115, July 9, 1898.

This material is specially adapted for the manufacture of articles which are usually made of rubber, &c.; it can be moulded while plastic or rolled out into sheets. Gelatinous animal tissue (skins, intestines, sinews, &c.) is gradually dried at a temperature not exceeding 65° C., then shredded or powdered, and mixed with 25 or 35 per cent. of glue, and 1 or 2 per cent. of potassium bichromate, with or without an antiseptic.—F. H. L.

# XIV.—TANNING, LEATHER, GLUE, SIZE.

*Bates and New Bateing Materials.* Der Gerber, 24, 573—576.

*The Bran Drench.*—The usual method in practice for drenching skins is to make up a fresh solution for each pack, the skins are kept in until the solution is in violent fermentation. Better results are obtained if the skins, previous to drenching, are first scudded, then put into tepid water for a short time, and afterwards drenched. The activity and effect of the drench depends on the temperature, the time in which the skins are left in, the activity of the fermentation, and the strength. Experience has shown that a temperature of 30° C. is probably the best. The time is dependent upon the purpose to which the skins are to be put; generally speaking, they may be left in until the appearance of small blisters. If, however, the action is allowed to go as far as this, it requires very careful watching. A suitable strength is 1 part of bran to 50 parts of water. If much stronger is used, there is danger of the skins going into holes, or the subsequent production of hollow and spongy leather. In some places the bran bate is liable to go wrong, in practice called "turning." If the bate turns, usually a good deal of acid is produced, which causes the leather to swell up, small quantities of lactic acid are formed, a large quantity of acetic, and sometimes butyric. The presence of even small quantities of the latter is very detrimental. The acetic acid is formed by the oxidation of the alcohol.

A peculiar defect which is sometimes produced on the pelt by the turning of the bate is that the skins become covered all over, sometimes only in parts, with a dull-grey slimy matter, which, when scudded off, leaves the grain of the pelt dull underneath. Investigation of this dull, slimy matter showed it to contain large quantities of "bacillus megaterium." These bacilli secrete a fluid which attacks and bites into the smooth surface of the leather, giving rise to the dull appearance both on the pelt and on the finished leather.

Another very important matter in drenching is the purity of the water used; it should be free from bacteria or other organic matter.—J. G. P.

*Sicilian Sumac and its Adulterants.* F. Andreasch. Der Gerber, 24, 573—576.

The method described in this Journal, 1898, 775, is that which is used all over the island of Sicily for the preparation of pure sumac, but, unfortunately, it is not always exported by the manufacturers in a pure state. It has gradually become the custom, in order to meet the low prices offered by the merchants, to adulterate the sumac, either by mixing with it sumac of poorer quality or by grinding up the stems and leaf-veins with the pure leaf, and even in some cases, where the sumac leaves the island in a pure state, it is still more frequently adulterated by the merchants. The mode of adulteration may be divided under two classes:—(1) Adulteration by mixing the stems with the leaf; sumac which has been spoilt in the harvesting, by rain or otherwise; also the admixture of other tanning materials in ground form. (2) The adulteration of sumac with foreign materials; large quantities of sand of a special yellow colour. This is largely done by the merchants. Sumac which has already been used in the tanyards, and from which the strength has been removed, is re-bought, dried, and ground in with the good material. But the more common adulterants are the ground leaf of Carobbe (Cistus salvifolius), fig leaves (Ficus carica), vine leaves, Ailanthus glandulosa, Pistacia lentiscus, and Tamarix africana. The last two are used perhaps more than any other. The Pistacia plant grows all over Sicily, and is even imported from Africa in large quantities specially for the purpose of adulteration. The Tamarix plant also grows in most parts of the island. The adulteration with Pistacia has of recent years greatly increased, as it is impossible to detect its presence in the ground sumac without the aid of a microscope, and the growers in Sicily blame the merchants for causing this state of affairs by continually demanding a low-priced sumac. The leaves of *Rhus coriaria* (sumac leaf) are characterised by being from 10 to 15 cm. long and egg-shaped; also by being thickly covered with minute hairs, as are also the stems. The stems and leaf stalks, when dry, are moreover of a red colour.

*Pistacia lentiscus.*—The leaves are of a dark green colour with a leathery feel; the leaf-veins protrude from the leaf; when dry are of a duller-red colour than the real sumac; the chief difference, however, being that the leaves are perfectly free from any hair growth. The powdered material is light green or grey-green in colour, with a sharp, penetrating smell.

*Tamarix africana.*—The leaves of this plant are small, like fish scales, and when powdered, produce a light green or yellow-coloured powder.

The leaves of the *Ailanthus glandulosa* (American sumac) are two or three times as large as the leaves of the *Rhus coriaria*, and when powdered are of a brown-grey colour.

Sumac, when not pure, is usually adulterated with from 25 to 50 per cent. of any of the above-named materials, Pistacia being, perhaps, the most frequently used.

To give an idea of the strength which good sumac should show on analysis, the following table is instructive. Table I. gives the result of 10 samples of pure sumac taken from different parts of the island. The lowest contained 21·2 per cent. of tannin substance and 18·13 per cent. of non-tannin matters; the highest gave 28·61 per cent. of tannin, and 16·27 per cent. of non-tannins. Table II. shows the average percentage of tannin in the sumac in the different stages of manufacture.

| — | Tannin. | Non-tannins. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Dry raw material | 20·70 | 19·14 |
| | 19·00 | 16·70 |
| After winnowing | 24·91 | 15·75 |
| | 24·28 | 16·67 |
| After ventilation | 25·82 | 16·48 |
| After the first grinding | 27·28 | 16·11 |
| After ventilation and second grinding | 28·98 | 16·44 |
| Stems and leaf-veins only | 14·70 | 11·70 |
| | 8·30 | 14·43 |
| | 11·53 | 17·77 |
| Sumac leaf, flesh only | 23·11 | 17·17 |
| | 29·45 | 15·89 |

The percentage of stems and leaf-veins which remain in the sumac after the winnowing depends, of course, upon the care with which the process is carried out. In samples examined, it showed from 7 per cent. to as high as 22·8 per cent.

In general, Sicilian sumac should never contain less than 22 per cent. of tannin; the average, from a large number of samples analysed, came out between 23 and 24 per cent.; and in all cases where the tannin percentage is below 22 per cent., it may be taken as an indication that the sumac is either adulterated with foreign materials, such as Pistacia, &c., or with a large percentage of stems. If sumac contain a large percentage of the stems, a few drops of acetic acid applied to a solution of the same gives a distinct red colour, which increases in density with the proportion of stems present in the powder. The percentage of tannin in Pistacia ranges from 13 to 17 per cent., and in Tamarix from 8 to 10 per cent.; the non-tannin matters in Pistacia, from 20 to 22 per cent., and in Tamarix, from 23 to 26 per cent. A sample of commercial sumac containing more than 20 per cent. of non-tannins may therefore be indicative of the presence of either of these materials.

The following qualitative chemical tests were found to give excellent results with solutions made from sumac containing adulterants from 8 to 60 per cent. About 20 grms. of the material to be investigated was treated with a litre of water at 60° C. and filtered. If Pistacia be present, the addition of two or three drops of a 40-per-cent. solution of formaldehyde gives, in a neutral solution, a light yellow flocculent precipitate, insoluble in cold water. A solution of arsenious acid, when warmed with a solution of Pistacia, gives a white powdery precipitate. Mercurous nitrate produces an egg-yellow precipitate, which turns to olive green, and finally dark green. The speed of these changes depends on the quantity of Pistacia present. The best reagent, however, is formaldehyde, which, with a pure sumac solution, gives no precipitate. With Tamarix, sulphurous acid produces a cloudiness, finally a white precipitate. Potassium cyanide gives a dirty-yellow flocculent precipitate. With sumac, neither of these reagents react. In order, therefore, chemically to decide whether a sumac is pure or adulterated with either of the above-mentioned materials, the following tests are sufficient:—I. Analysis of the percentage of tanning matters and non-tanning matters. II. Test for Pistacia, with formaldehyde. III. Test for Tamarix, with potassium cyanide and sulphurous acid.

These materials may also be easily detected by careful microscopic analysis. Sumac under the microscope is easily detected by (*a*) the number of single and double hairs upon the surface; (*b*) the striped cuticle; (*c*) the appearance of the hair sacs; (*d*) the appearance of the mesophyll; (*e*) the presence of crystals; (*f*) the appearance of the pores.

With Pistacia, the characteristic appearance:—(1) the double row of cells; (2) the position of the pores; (3) the appearance of the leaf nerve. With Tamarix, the characteristic appearance:—(1) the polygonal cells; (2) no trace of hair, but the whole of the surface of Tamarix is covered with minute papillæ. The papillæ appear to lie on top of each cell, are colourless, and not easily seen.—J. G. P.

*Glue, The Viscosity of.* W. Melfert. Chem. Zeit. **22**, 506.

The author tested the accuracy of the statement made by Fels (Chem. Zeit. **22**, 376), that it is possible to calculate the tenacity of glue from its viscosity. The conclusion he arrives at is, that this cannot be done with sufficient accuracy; nevertheless, the viscosity of a glue is a criterion as to whether it be of good, medium, or bad quality.
—W. P. S.

*Tannin, Estimation of.* L. Vignon. Comptes Rend. **127**, [8], 369.

*See under* XXIII., *page* 957.

### PATENT.

*Artificial Leather, Impts. in the Manufacture of.* A. E. E. Cantley and E. Springborn, Leyton, Essex. Eng. Pat. 19,356, August 21, 1897.

Three parts of asphaltum, 6 to 8 of Stockholm tar, 16 of "resin," 4 of paraffin, and 12 to 18 of "benzoline" are boiled together, and a suitable fabric, such as canvas, is drawn through the solution, passed between rollers, dried, and finished by brushing with wax or spirit varnish in any desired colour; the whole operation being conducted at a temperature of 25° to 40° C. When treating a material having a nap or pile on its surface, like flannelette, 1 to 9 parts of sulphur and about ½ part of camphor are added to the bath.—F. H. L.

## XV.—MANURES, Etc.

*Soil Inoculation for the Growth of Leguminous Plants.* F. T. Shutt. Annual Report of Exper. Farm, Ottawa, 1897, 141—146.

Experiments, conducted in pots, were made with horse beans and red clover, with and without nitragin, which was applied to the seeds, and directly to the soil, respectively.

In the case of horse beans, the greatest amount of produce (chiefly due to increase in root production) was obtained under the influence of soil-inoculation, and the produce contained, generally, considerably more organic matter, nitrogen and ash (actual, not per cent.), than was the case with the other pots. "Seed-inoculation" gave results intermediate between the "soil-inoculated" pots and those which were not inoculated.

Inoculation also considerably increased the yield of red clover. In this case "seed-inoculation" gave the best results The inoculated plants contained slightly higher percentages of nitrogen (especially in the roots) than those which were not inoculated.—N. H. J. M.

*Unexhausted Manures, Valuation of; The Royal Commission on Agricultural Depression and the.* J. B. Lawes and J. H. Gilbert. Journ. Roy. Agric. Soc. Engl. 1897, [3], **8**, 674—711.

When organic manures, such as farmyard manure and rape cake, have been applied to the land and grown a crop, a residue remains which becomes available to future crops only gradually, though more rapidly on light than on heavy soils. The value of manure produced by the consumption of different cattle foods varies very greatly (see tables in original paper).

Nitrate of soda is not fixed in any way by the soil, and, unless taken up by vegetation, is liable to be lost in drainage, or to pass into the subsoil beyond the reach of the roots. When ammonium salts are used, a part of the ammonia is temporarily fixed by the soil; but it is more or less rapidly nitrified, and is then liable to loss by drainage—most when the land is occupied by grain crops, least with root crops or permanent grass.

When these artificial nitrogenous manures are used for grain crops, a more or less nitrogenous residue (stubble and roots), according to the amount of manure applied, remains. With root crops much more of the nitrogen will be recovered than in the total produce of a grain crop. In the case of permanent grass, more of the supplied nitrogen will be recovered, both in the increase of crop and in the crop residue, than with grain crops.

When phosphatic manures and potassium salts are applied as manure, a large quantity soon becomes fixed in the soil, especially in heavy soils. In this condition the manure becomes available very slowly, and the compensation value of the unexhausted residue is very small.

It must always be a difficult matter to distinguish between the landowners' and the tenants' fertility, and to assign to the latter a commercial value, especially as some soils contain vast stores of natural fertility, others very little.

—N. H. J. M.

*Oil-Cakes, Manurial Value of.* L. Malpeaux. Ann. Agron. 1897, **23**, 28.

Whilst oil-cake is chiefly utilised for cattle-feeding, a considerable amount, for various reasons unsuitable for this purpose, is sold as manure. In the north of France as much as 500—1,000 kilos. is applied per hectare for various crops (especially tobacco), and in the south it is becoming more and more employed for grain crops and vines, &c. The value of oil-cake depends mainly on the large amount of nitrogenous matter it contains, its percentage of nitrogen being greater than that of farmyard manure. Oil cakes contain comparatively little phosphoric acid (0·82 to 1·9 per cent. was found in the various samples examined), and still less potash. The manurial value of different cakes was found to vary considerably, and decreased in the following order:— Sesamé cake ($N = 7$); camelina ($N = 5{\cdot}5$); earth-nut ($N = 6{\cdot}5$); cotton-seed ($N = 4{\cdot}5$); and castor-oil cake ($N = 4$ per cent.). Of course, in practice, the relative cost of the various cakes must also be taken into account.

The imports and exports of oil-cake from France amounted in 1877 to 11·3 and 67·7 million kilos., and in 1897 to 47·0 and 75·5 million kilos., respectively.—N. H. J. M.

*Blood from the Abattoirs of Small Towns, Utilising, as Manure.* A. School. Bull. Assoc. Belge des Chim. **12**, [4], 151—158.

The method adopted by the author at Gembloux consists in storing the blood in cemented cisterns, wherein it is mixed, in alternate layers, with peat and superphoshate in the (approximate) proportion of 1 part of peat, 3—4 of blood, and 2 of superphosphate. The functions of the latter ingredient are to assist the peat in facilitating deodorisation, and to improve the physical condition and manurial value of the product.

The first batch of manure made in this manner is not yet ripe for analysis, but from the results obtained in the preliminary tests, made on a manufacturing scale, it is believed that nearly the whole of the nitrogen in the blood (averaging about 2·4 per cent.) is retained.—C. S.

*Phosphoric Acid in Phosphates, A New Solvent for.* W. Hoffmeister. Landw. Versuchsstat. 1898, **50**, 363.

*See under* XXIII., *page* 951.

*Sugar-Cane, Experiments on Manuring.* E. Silz. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [12], 1227.

*See under* XVI., *adjoining column.*

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar-Cane, Chemical Physiology of the.* P. A. F. C. Went. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898. **15**, [12], 1217—1226.

From a micro-chemical examination of the constituents of the sugar-cane, it appears that glucose (invert sugar) and saccharose, starch, and tannin are found in the parenchymatous cells, and not in the vascular bundles, while the contrary is the case with the albuminoids. The absence of sugar in the vascular bundles confirms the results obtained by Winter and Beeson, that those parts of the cane which contain much vascular tissue and little parenchyma, that is, the nodes and the periphery of the internodes, are very poor in sugar; and the observation of Winter, that the juice which runs, at the mill, from the extremity of vascular bundles contains no sugar.

Diagrams are given of longitudinal sections of the top of the cane, buds, and root, in which the distribution of glucose, starch, tannin, and protein is indicated. The growing point contains no glucose, which only begins to appear at the fourth or fifth internode (about 2 cm. from the top), and increases rapidly in each succeeding internode until a maximum is reached, whence it again diminishes until there is practically none at the lower part of the stalk.

Saccharose seems to appear lower down than glucose, but it is only the oldest part of the stalk which contains a minimum.

Starch is found in great quantity at the growing point, but diminishes lower down; at first it is distributed uniformly, but where glucose begins to appear it is found only in the internodes, and as a sheath round the vascular bundles. Less and less starch is found in the older internodes, and it disappears completely in the internodes which have reached full growth.

Tannin is found in great quantity at the top of the cane; lower down it is found only in the nodes, and ceases in the organs that are fully developed. The epidermic cells with coloured contents always contain tannin. The tannin of the stalk gives a green precipitate with iron salts; that of the epidermic cells, a dark blue precipitate.

Albuminoids are found exclusively at the growing point.

The developing bud draws its nutriment from the cutting. It appears to exert an influence on the saccharose, chiefly in the neighbourhood of the vascular bundles, causing inversion. The glucose is carried towards the shoot and deposited partially around the vascular bundles as starch, to be transported later.

Where cellular division is taking place, albuminoids are found and much starch, but no glucose; where there is lengthening of the cells, and therefore growth, no albuminoids are found, little starch, and much glucose.

The carbohydrates necessary for the growth of the roots are drawn from the internode to which the roots are fixed, in the form of glucose from the inversion of saccharose. The glucose is accumulated in that part of the root where there is lengthening of cells, and therefore growth. In the zone of growth the glucose disappears, since it combines there with nitrogenous organic matters to form albuminoids. Finally, a part of the glucose is deposited as starch in the root cap as a reserve.

—L. J. de W.

*Sugar-Cane, Experiments on Manuring.* E. Silz. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [12], 1227—1232.

The authorities of the Botanical Garden of British Guiana have made important experiments on manuring sugar-cane during 1893, 1894, and 1895, and the results are collected in an interesting report, of which the following are the conclusions:—

Phosphatic manures give considerable and very remunerative increase when used along with manures containing nitrogen and potash. Phosphate slag seems to be an excellent source of phosphorus for cane plantations, and ought to replace superphosphate. Nitrogen in the form of sulphate of ammonia, nitrate of soda, and dried blood, shows a favourable influence on the yield. The best results were obtained with a mixture of $\frac{1}{3}$ nitrate of soda and $\frac{2}{3}$ sulphate of ammonia, although, for large quantities, sulphate of ammonia gives a better pecuniary result. Dried blood was notably inferior. Potash seems to be of only small efficacy. The employment of sulphate of potash on land not limed gave a slight benefit, but a loss on limed soils. The use of nitrate of potash instead of nitrate of soda did not furnish satisfactory results.

The employment of lime gave great increase of yield, the effect on the first crop being sufficient to pay the cost of its application, while the increase in the shoots constituted a net gain.—L. J. de W.

*Beetroot Juice [Sugar], Peptones in.* A. Rumpler. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **15**, [12], 1276—1277. (From Deutsche Zuckerind. 1898, 22.)

The author acidulated 50 c.c. of first carbonatation juice with acetic acid, and then added 700 c.c. of 95 per cent. alcohol and allowed the mixture to stand for eight days, mixing occasionally. The brownish precipitate formed was collected on a filter, air-dried, and washed with very little distilled water. The peptones are found in the washings, while the undecomposed albuminoids rendered insoluble by the alcohol remain on the filter. The brownish solution was acidified with 10 per cent. sulphuric acid, and, on the addition of phosphotungstic acid, became very cloudy, and during the night gave a flocculent precipitate.

To show the presence of peptones, this precipitate was treated with baryta, a little water added, and a small quantity of a very dilute solution of sulphate of copper (two drops of a 3 per cent. solution to 10 c.c.). The dirty-blue coloration indicates the presence of peptones.

Although very diffusible, neither peptones nor their salts are crystallisable, and therefore, in all probability, exercise a melassigenic action when present in low products. Their presence in saturation juice shows how slowly they are decomposed, and this decomposition becomes still slower as the alkalinity of the juice diminishes. The presence of peptones in the juice may explain the action of animal charcoal, which absorbs them completely. Stammer pointed out long ago that, by filtration over char, the purity of syrups was increased by 5 per cent. This is explained by the strong levo-rotation of peptones ($a_D$ = 70° to 80°). The peptones of the juice, being probably combined with lime, the latter is likewise absorbed by the char.

Although filtration by char is now abandoned in sugar works, the theoretical interest of the matter still remains. —L. J. de W.

*Syrups [Sugar] Filtration of.* K. Andrlik. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1828, **15**, [12], 1258—1259. (From Zeits. Zuckerind. in Böhmen, 1897, 212.)

An analysis of the deposit from the syrup filter of the third cylinder of a multiple effect gave the following result:—Insoluble in water, 13·43 per cent.; sugar (by polarisation), 43·41; soluble non-sugar, 22·70; water, 20·43 per cent. The portion soluble in water had a quotient of purity of 65·6. The portion insoluble in water consisted principally of silica, alumina, and oxide of iron, lime, with oxalic acid and fat, as well as insoluble caramels. The deposit was gelatinous, and resembled molasses. With water it gave a turbid liquid of light yellowish-brown colour, and the gelatinous character disappeared.

Analyses of these deposits on the parchment paper during the osmosis of unfiltered syrup and molasses gave, on the dry substance:—

| —— | A. | B. | C. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Ash | 53·08 | 32·17 | 60·98 |
| Fatty matters | 12·93 | 25·86 | 10·03 |
| Oxalic acid | 10·66 | 10·20 | 7·62 |
| The ash contained— | | | |
| Silica | 18·92 | 33·64 | 19·38 |
| Iron and alumina | 9·83 | 13·06 | 26·52 |
| Oxide of copper | 2·49 | 3·02 | .. |
| Lime | 30·21 | 27·07 | 16·67 |
| Magnesia | 2·71 | 2·79 | 1·08 |
| Sulphuric acid | 8·31 | 6·01 | Traces |
| Phosphoric acid | 2·44 | 2·78 | Traces |
| Carbonic acid | 19·28 | 11·40 | .. |

These results show that the filtration of syrups is an indispensable operation, for the precipitated non-sugars re-dissolve during evaporation and boiling, and hinder crystallisation.—L. J. de W.

*Milk Sugar, Examination of.* C. Beuttner. Schweiz. Woch. f. Chem. Pharm. 1897, 589; Pharm. J. 1898, **61**, [1469], 238.

*See under* XXIII., *page* 959.

*Dextrin containing Starch, Analysis of Commercial.* R. Hefelmann and W. Schmitz-Dumont. Zeits. für öffentl. Chem. **4**, 448; Chem. Centr. 1898, **2**, [8], 561.

*See under* XXIII., *page* 960.

*Honey, Optical Determination of, by the Polarimeter.* R. Frühling. Chem. Centr. 1898, **2**, [4], 305.

*See under* XXIII., *page* 959.

*Pentoses, Revised Method of Determination.* A. Grégoire and E. Carpiaux. Bull. Assoc. Belge des Chim. **12**, [4], 143.

*See under* XXIII., *page* 959.

## PATENTS.

*Lactose, or Sugar of Milk, Impts. in or relating to the Manufacture of.* H. Higgins, Cambridge. Eng. Pat. 23,875, Oct. 16, 1897.

Separated milk is treated with hydrochloric acid to precipitate the casein, which is filtered off. The patentee claims the process of neutralising the excess of acid in the filtrate with calcium carbonate, and then preparing milk sugar from it by any suitable method.—W. P. S.

*Purification and Decolorisation of Liquids and the like, Impts. relating to the, and to Apparatus therefor.* P. Pesse, Marcq en Baroeul, Nord, France. Eng. Pat. 8017, April 4, 1898.

Syrups from sugar works, and similar products, are mixed with animal charcoal in a state of an impalpable powder, and the whole, after more or less prolonged agitation, filtered in such a manner that the whole of the charcoal may be recovered. The charcoal may be completely or partially washed, or may be used as it is for a similar, but still more impure, liquid. An automatic mixing apparatus is described, which is characterised by an automatically regulated inlet valve; the automatic supply of the purifying material in the required amount; and a series of baffle plates by means of which the liquid and the purifying materials are kept in contact in successive portions, no portion being able to leave the apparatus before the preceding portion has left it.—N. H. J. M.

# XVII.—BREWING, WINES, SPIRITS, Etc.

*Sorbose, Bacteria of, Action of, on Xylose.* G. Bertrand. Comptes Rend. 1898, **127**, [2], 124—127.

After finding that the bacteria of sorbose oxidised certain of the higher alcohols, such as sorbite, mannite, and glycerin, converting them into ketonic sugars (Comptes Rend. **126**, 762, 842, and 984; this Journal, 1898, 592), it occurred to the author to determine their behaviour towards certain other substances, and he has therefore made the first experiments with xylose. On inoculating a culture medium containing xylose as its base with the bacteria, the small colonies which form unite finally into a single membrane, but do not develop so rapidly as in sorbite or glycerin solutions. In consequence, the zooglœa has a different appearance, and, instead of being homogeneous, opaque, white, and resistant throughout, has only these characteristics in certain parts, each of which corresponds to one of the primitive colonies. The remainder of the membrane is transparent and without consistence.

In the experiments, a broth consisting of 1 litre of 0·5 per cent. yeast-water containing 20 grms. of crystallised xylose was used as the culture medium, and the cultivations were incubated at 29° to 30° C. After a fortnight and even a month, the liquids still reduced Fehling's solution strongly, and in one experiment nearly half the original sugar was found after the latter period. At the same time the solution acquired a characteristic property. After neutralising the free acid with standard alkali, the liquid became acid again in a few seconds, and this process of neutralising successively appearing portions of acid was continued until eventually complete neutrality was reached. Like other acids of its series, xylonic acid readily undergoes partial dehydration with the formation of a lactone, the reaction of which is neutral. This transformation is only arrested when there is a certain ratio (which varies with the conditions of temperature, concentration, &c.) between the amount of lactone and of acid. If the latter be neutralised, the equilibrium is destroyed and a part of the lactone is reconverted into acid. Thus, it is only by successive titrations that the whole of the xylonic acid in a free state and as lactone can be determined. The amounts of acid and of sugar found in cultivations containing originally 5 grms. of xylose were :—

| — | Acidity as Xylonic Acid. | Xylose corresponding to Xylonic Acid. | Residual Sugar. | Total. |
|---|---|---|---|---|
| | Grms. | Grms. | Grms. | Grms. |
| After 2 weeks ..... | 1·46 | 1·32 | 3·52 | 4·91 |
| „ 4 „ ..... | 2·27 | 2·04 | 2·70 | 4·74 |

Taking into account the absence of volatile acids and the behaviour of the liquid on titration with alkali, the author considered it extremely probable that the acid formed was xylonic acid, and he proved the truth of the hypothesis by isolating it as xylonobromide of cadmium—($C_5H_9O_6.CdBr + H_2O$) (Bull. Soc. Chim. **15**, 592).

The sorbose bacteria, therefore, oxidises xylose by transforming it into the corresponding monobasic acid—xylonic acid. Whether they behave in the same way in the case of other reducing sugars is a point which the author is now investigating.—C. A. M.

### *Raffinose, The Fermentability of, and Analytical Value of.* A. Bau. Zeits. f. Spiritusind. 1898, **21**, 241; and Wochenschr. f. Brauerei, 1898, **15**, 397—399.

It has been pointed out by Berthelot, Loiseau, Scheibler, and later by the author (this Journal, 1895, 289), that low-fermentation yeasts ferment raffinose completely, while high-fermentation yeasts, after splitting this sugar into melibiose and levulose ferment only the latter, thus leaving the former in solution. This behaviour has resulted in raffinose being used as a means for identifying the yeasts of these types and of mixtures of them. The author now finds that low-fermentation yeasts of the Frohberg and Saatz types, as well as *Saccharomyces Pasteurianus III.*, can completely ferment raffinose and melibiose.

For the detection of low- in high-fermentation yeast, Herzfeld mixes 1 grm. of the yeast with 10 c.c. of a 1 per cent. solution of raffinose, and fills up an Einhorn's fermentation saccharometer with the mixture, avoiding the formation of bubbles. The side tube is trapped with a few drops of mercury, and the whole apparatus allowed to remain for 24 hours at 30° C. The volume of carbon dioxide is then read off, and from it is subtracted the volume of the same gas developed in a control experiment, using sterilised water and a similar quantity of yeast. Einhorn's apparatus allows a volume of 5 c.c. to be read ; if 2 or at the most $2\frac{1}{2}$ c.c. of carbon dioxide be formed, the yeast is considered pure, but if this volume be exceeded, the high-fermentation yeast is adulterated with low-fermentation yeast.

According to the author's method, in three test tubes are placed 10 c.c. of a 1 per cent. solution of raffinose, and each tube plugged with cotton wool. After the tubes and their contents have been sterilised, to each is added 0·4 grm. of the yeast, and the tubes kept at 25° to 30° C. At the end of the first, second, and third day, the contents of a tube are filtered, and 3 c.c. of the filtrate heated in a boiling water-bath for five minutes with 1 c.c. of Fehling's solution. If the solution, after settling, remain blue in the first tube, there is over 10 per cent. of low-fermentation yeast present ; if in the second, 5 per cent.; if in the third, 1 per cent. Should, however, the solution be reduced or over-reduced, the yeast under examination is technically pure high-fermentation yeast. This method is said to give good results with high-fermentation pressed yeasts prepared either in the old way, or according to the new aëration process (Lufthefe).

To determine whether Buchner's preserved yeast [Dauerhefe] (this Journal, 1898, 594) is prepared from a high- or low-fermentation variety, or a mixture of the two, the author adds 2 per cent. or more of the material to be tested, containing 1 per cent. of toluene to a 1 per cent. solution of pure melibiose, and keeps the mixture for one to three days at 25° C. The solution is filtered and treated with phenylhydrazine acetate. Should the resulting osazone dissolve in hot water, top-fermentation yeast has been employed, but if an insoluble osazone is present, low-fermentation yeast has been used. The author describes a series of determinations on these lines, showing that an addition of 10 per cent. of low-fermentation yeast can be detected in the preserved yeast. To obtain pure melibiosazone, the fermented solution is precipitated with lead acetate, and the excess of lead removed from the filtrate by sulphuretted hydrogen. The osazone is then prepared, and, after several crystallisations, it melts at 177° to 178° C.

For the preparation of melibiose solutions there are two methods. According to Scheibler, raffinose is partially inverted with sulphuric acid, and when by polarimetric examination, the hydrolysis to *d*-fructose and melibiose is found to be complete, the acid is neutralised and the syrup obtained by evaporation extracted with absolute alcohol until all the levulose is removed, as shown by the osazone test. The second method is to infect a 1·737 per cent. solution of raffinose in sterilised yeast decoction with a pure cultivation of high-fermentation yeast. On complete fermentation, a solution containing approximately 1 per cent. of melibiose results. Before employing this or Scheibler's preparation, it must be submitted to the osazone test to ensure the absence of hexoses. The latter method is more convenient than Scheibler's, but it takes longer, as fermentation must proceed to the end.

Raffinose may be estimated in all fermentable sugar mixtures by sowing a solution of the sugars in a nutrient medium with pure cultures of high-fermentation Frohberg yeast, and a second with a low-fermentation Frohberg yeast, and allowing the two to ferment completely. The difference in solid matter, in the polarisation and reducing power of the two fermented solutions, gives the measure of the melibiose formed, and if this be multiplied by 1·737, the amount of crystallised raffinose in the mixture is obtained.

Hitherto there has been no other method for estimating raffinose in molasses than the ordinary acid inversion. (Compare Ling and Baker, this Journal, Discussion, 1898, 113.)

The author removes the volatile acids from molasses by adding citric acid and distilling in a current of steam. Some yeast decoction is then added and the molasses divided into two solutions of equal volume; each is sterilised and sown with pure cultures of high- and low-fermentation yeast respectively. When the fermentation is over, the difference in the extracts is due to melibiose, and this can be calculated in terms of raffinose. Since the fermentation takes 16 days, the method is too lengthy for ordinary purposes ; but, in the author's opinion, it is well suited for a control over the ordinary acid inversion process, since the numbers obtained by the respective methods agree closely.—J. L. B.

### *Yeast, The Activity of Differently Nourished. Diastase as a Yeast Food.* G. Heinzelmann. Zeits. für Spiritusind. 1897, 296 and 311—312.

A wort was prepared by mashing 100 grms. of starch and 25 grms. of malt at a low temperature, and the volume of the wort made up to 500 c.c. Experiments were made to determine the rapidity of fermentation of this wort with and without the addition of a mineral nutriment consisting of potassium phosphate and magnesium sulphate, and a nitrogenous nutriment of asparagine.

The addition of the mineral nutriment did not increase the rapidity of the fermentation, but that of the asparagine did.

The yeasts obtained in these fermentations were then sown in a wort, and the rapidity of the fermentation determined. Practically no difference was found.

Experiments were then made to determine the effect of the addition of diastase or peptone compared with that of asparagine on the rapidity of fermentation. Both the former increased the rapidity of fermentation, but neither to the same extent as asparagine, peptone having more effect than diastase.

Further determinations of the rapidity of fermentation of a cane-sugar solution were made in which the only nitrogenous nutriments were respectively asparagine, peptone, diastase, diastase which had been rendered inactive by heating, and one with no nitrogenous nutriment. The most rapid fermentation was that in which asparagine was used, and the next most rapid that with peptone, the next that with active diastase, the next that with inactive diastase, and the slowest that in which no nitrogenous nutriment was added.

No diastase remained active in the fermented solutions, and this the author ascribes to the destructive action of the acids produced. Yeast nourished on diastase was found to be no more able to ferment dextrin than a yeast nourished on asparagine.—A. L. S.

### *Yeast, Proteohydrolysis.* H. Will. Zeits. für d. ges. Brauwesen, **21**, [12]: [13], 153—155; [14], 167—169; [15], 181—183.

From the experiments made by the author with 27 yeasts and a mycoderma, he concludes that proteohydrolysis is a normal function of the vital activity of these organisms. It is due on the one hand to a lack of soluble nutrient material (nitrogen in particular), *i.e.* to a state of famine, and on the other to the necessity for oxygen experienced by the cells embedded in the solid medium.

The liquefactive energy exerted by the cells depends on the species of the yeast, the method of preparing the cultures, and the temperature. For instance, in puncture cultures, liquefaction ensues at a later date than when the yeast is uniformly distributed through the gelatin. In the former cultures the greatest liquefactive energy is displayed by the yeasts having the greatest affinity for oxygen, *e.g. S. anomalus, mycoderma*, and the top-fermentation beer yeasts. As a rule, liquefaction commences in the channel of the puncture; in individual cases, *e.g. S. anomalus*, it also begins on the under side of the superficial covering. Generally speaking, liquefaction is retarded by reductions of temperature, though the converse is observed in some instances.

The maximum liquefactive activity is exhibited when the yeast is distributed throughout the gelatin, it being feasible to effect the almost total liquefaction of 10 c.c. of gelatin within 48 hours. The action begins underneath a zone of maximum activity of growth, the depth of which varies with the yeast, whereas within that zone itself, the rate of liquefaction is slow, and probably due to a proteohydrolytic enzyme in the liquefied gelatin, not to the yeast embedded therein.

Apparently, when the gelatin is much fissured as a result of fermentation phenomena, liquefaction may be entirely absent. A copious development of yeast cells occurs on the surface of the fissures, but where the latter are of moderate extent, liquefaction occurs underneath the zone of such secondary growth, these phenomena indicating that oxygen exerts a direct or indirect influence on protehydrolysis.

Where the yeast is distributed through the gelatin, the difference in liquefactive energy between top- and bottom-fermentation yeast is comparatively small, though in individual cases the former again proved the more active of the two.—C. S.

### *Brandy, The Quality of.* B. Jürgens. Farmaz. J. 1898, **20**, 294.

Russian cognac has properties very similar to those of French cognac, though the prices do not correspond at all with the analytical figures. The following comparison is given by the author between French brandies costing 3 to 6 roubles per litre and Russian brandies costing 0·7 to 1·6 roubles. The upper figures refer to the French product.

| Alcohol. | Rose's Number. | | |
|---|---|---|---|
| | First Distillation | Second Distillation. | Difference. |
| ° Tralles. | C.c. | C.c. | C.c. |
| 47·7—50·0 | 0·10—0·25 | 0·00—0·18 | 0·01—0·07 |
| 43·1—48·8 | 0·06—0·33 | 0·05—0·26 | 0·01—0·07 |

| Acid per 100 C.c. calculated as Acetic Acid. | Sugar in 100° C.c. | | Extract other than Sugar in 100 C.c. |
|---|---|---|---|
| | Before Inversion. | After Inversion. | |
| Grm. | Grm. | Grm. | Grm. |
| 0·048—0·07 | 0·45—0·56 | 0·83—1·64 | 0·22—0·48 |
| 0·012—0·08 | 0·25—0·92 | 0·62—1·35 | 0·04—0·13 |

—J. T. D.

### *Beer, The Production of a Brilliant.* W. Duncan. J. Fed. Inst. Brewing, 1898, **4**, 477—505.

The three essentials which a wort should possess, so that a beer of rapid clarification may be produced, are (1) a suitable proportion of fermentable sugars; (2) a smaller proportion of less fermentable carbohydrates, facilitating early cask condition; (3) a sufficiency of nitrogenous and phosphatic extract to sustain the growth of the yeast, and at the same time, no excess of these substances, which are frequently a cause of tubidity and unsatisfactory fining.

Since the "Free Mash Tun" Act of 1880, differences have arisen in brewing processes, the chief points being an alteration in striking heat, and the large proportion of substitution materials used in the production of modern pale ale. When under-cured malts are used, they produce, on account of their high diastatic capacity, a small quantity of amyloins of a fermentable type, so that the finished beer exhibits a great tendency to develop a violent but transitory cask fermentation, probably an actual "sick fret." Any attempt to restrict the action of diastase by high mashing temperatures, although bringing about an increase in the proportion of amyloïns, also yields carbohydrate constituents incapable of producing a sufficiently early cask fermentation. When such grains have to be used, the author employs a modified decoction system of mashing, which is stated to give worts of good composition. Spontaneous clarification of beer does not take place with the requisite speed for modern conditions of trading, so that artificial fining is almost universally adopted. The fining capacity of a beer is determined by the following influences:—(1.) The matter in suspension must be readily separable. (2.) The residual carbohydrate extract must be of a type favourable to steady fermentation. (3.) The new yeast must not be produced in any excessive quantity.

The view that light foreign grain tends to yield less soluble nitrogenous extract than English malts is erroneous. Notwithstanding this fact, their presence is conducive to the production of brilliant beer; thus, foreign grain, in having a larger proportion of nitrogenous extract and a smaller proportion of substances liable to cause "haze," is a not unimportant factor in deciding early brilliancy in English beers. The author considers that boiling worts under pressure favours the transformation of peptones and other forms of nitrogenous extract into amides under the influence of malt and hop acids, and consequently leads to early brilliancy. The hot aeration of worts brought about by exposure on the cooler at temperatures ranging from 195°—140° F. also exerts a considerable influence on the brilliancy of the finished beer. It is said that a large proportion of nitrogenous and resinous matters are removed from the wort by this treatment, and modifications in the degree of cooler aeration have frequently solved difficulties in connection with "resin haze."—J. L. B.

### *Cask Plant.* L. Briant. J. Fed. Inst. of Brewing, 1898, **4**, 509—518.

Two beer casks of Memel oak were examined, one comparatively new, the other six or seven years old, to ascertain to what extent organisms had penetrated into the wood.

Staves were taken from each cask, and thin longitudinal sections cut, which were at once placed in sterile wort and incubated at 90° F. The results obtained are given on next page.

The casks were unsteamed, and were only washed with water after being returned from the consumer to the brewery.

*New Cask.*

Inside of Stave.

| | In. |
|---|---|
| Many organisms, both yeast and bacteria... | ⅛ |
| One or two bacteria ........................ | ⅛ |
| No growth ........................ | ¼ |
| No growth ........................ | ¼ |

Outside of Stave.

*Old Cask.*

Inside of Stave.

| | In. |
|---|---|
| Many organisms, both yeast and bacteria ... | ⅛ |
| Yeasts (a few) and bacteria ................ | ⅛ |
| One or two bacteria ........................ | ¼ |
| No growth ........................ | ¼ |

Outside of Stave.

To ascertain how far casks are rendered sterile by ordinary treatment, sections were taken first after steaming, and secondly, after they had been filled with boiling water, left over night, and emptied and steamed the following morning. It was found that whilst organisms were destroyed on the inner surface, those which had penetrated into the wood were not with any certainty killed. The results were irregular, and the success of the steaming operation varied with the character of the cask. In all cases, prior filling with boiling water much assisted in the sterilisation, and there can be doubt as to the value of this practice, for, in addition to the more complete sterilisation, acids and objectionable flavours are soaked out. In view of the failure of the ordinary steaming operation to secure perfect sterilisation, experiments were made to ascertain the temperature to which the wood was raised. A tabulated list of temperatures of the air inside the cask, of the wood ⅛ in. from inner surface, of the middle of the wood, and of the wood ⅛ in. from outer surface for periods varying from 5 to 60 minutes is given. The ordinary time for steaming a cask may be taken as 10 minutes, and it was found that whilst the inside had reached a temperature of 209° F., the heat at a distance of ⅛ in. from the inner surface had only risen to 130° F., whilst the middle and outside of the stave were much cooler. It is obvious that 10 minutes steaming in insufficient, for if any organisms had penetrated beyond as ⅛ of an inch they would not be destroyed. Some experiments are described in which it was found that casks with the bung and tap holes open could be rendered sterile by placing them in a chamber through which steam was blown. The non-volatile acids can then be removed by soaking in water.—J. L. B.

*Dextrinous Mashes, Process for the Fermentation of, by Means of an Acclimatised Yeast.* J. Effront. Wochenschr. f. Brauerei, 1898, **15**, 435. (Belgian Pat. 134,692.)

This process depends upon the property which certain races of beer yeast possess of fermenting dextrin, and this function can be very considerably enhanced by growing beer yeast in suitable nutritive media. For this purpose yeast is grown in a 15 per cent. solution of cane sugar or glucose containing yeast ash, potassium nitrate, and aldehyde, and the operation repeated 40 to 50 times, the quantities of potassium nitrate and aldehyde being increased at each fermentation. Following this treatment, the yeast is grown in mashes of a gradually increasing content of dextrin containing cane sugar, yeast ash, potassium nitrate, and aldehyde in the same proportion as at the end of the first operation. At the end of successive fermentations, the quantity of cane sugar is diminished until finally none is left, and the yeast ferments the dextrinous mash alone. The yeast at this stage may be filter-pressed, or dried *n vacuo* at 30° to 40°, and used for fermenting distillery worts.—J. L. B.

*Furfural in Malt, Wort, and Beer; Occurrence of.* J. Brand. Zeits. für d. ges. Brauwesen, **21**, [21], 255—258.

The beers examined at the Munich Scientific Station for Brewing are always tested for furfural — colour reaction with aniline and glacial acetic acid—but only traces are ever found, and that rarely, though the substance is usually present in wort. Moreover, tests made with the object of ascertaining whether the bread-like flavour of pasteurised beer is due to this aldehyde have constantly yielded negative results.

From his experience in caramel malts and beer worts the author concludes that furfural is a normal constituent of the aromatic roasted malts of the kind used for Bavarian beer, and also of the worts extracted therefrom, but that it is rapidly eliminated when the worts are boiled. He also finds the aldehyde a regular constituent of beer colorants, and considers its presence may be utilised as an indication of the addition of such substances to the finished beer.

Owing to its volatility, furfural cannot be regarded as likely to prove injurious to beer, the quantity present in wort being too small to have any ill effect.—C. S.

*Malt, Influence of Kilning and Storage on Yield of Extract and Maltose as well as on Diastase, and Albuminoid-, Peptone-, and Amide Nitrogen.* A. Schulte im Hofe. Zeits. für d. ges. Brauwesen, **21**, [19], 231—234; [20], 246—248.

*Diastatic power* is apparently increased during withering, owing to the germination of the malt not suffering any sudden interruption during that process. The reverse occurs, however, on the malt kiln, the main factors influencing the change being the length of exposure and the degree of heat. On the other hand, storage of the malt results in a re-increase.

The yield of extract is greatest in green malt and that kilned at low temperatures, an initial yield of 81·45 per cent. recedingto 79·49 per cent. after 33 hours' kilning at a final temperature of 52·5° C., followed by 26 hours, at a final temperature of 80° C.; the continuance of the operation for a further 37 hours (f. t. 131·2° C.) reducing the yield to 37·97 per cent. In this respect storage results in a loss when a low kilning temperature is in question, but in a gain under converse conditions.

Kilning greatly reduces the percentage of maltose, though only slowly at first, the diminution being from 60·43 per cent. to 50·50 per cent. during 49 hours, with a final temperature of 80° C., whilst after a further 13 hours (f. t. 100° C.) the percentage was 41·92 per cent. The effect of storage is parallel to that observed in respect of total extract.

The proportion of soluble nitrogen is greatest in green malt, but the effect of kilning varies with the nature of the nitrogenous matter. For instance, in the case of a green malt containing 0·869 per cent. of soluble nitrogen, *viz.*, albuminoid nitrogen, 0·089; peptone nitrogen, 0·170 per cent.; amide nitrogen, 0·610—the first-named after attaining its maximum at the end of nine hours' withering, rapidly decreased, becoming only 0·048 per cent. after kilning, whether at 80° or 100° C. Storage further reduced the content of this albuminoid nitrogen, the effect being greatest in malt kilned at low temperatures.

On the other hand the peptone nitrogen was increased (by kilning) to a maximum of 0·194 per cent. with 80° C. as the final temperature, but receded again, as the heat increased. Storage brought about an increase in malt kilned at 53·75° C., but the converse when the temperatures exceeded or fell below this figure.

Amide nitrogen diminishes steadily during kilning—in the present case, to 0·261 per cent. (f. t. 100° C.). Storage affects a further reduction when low-temperature kilning is practised, but the converse result when higher temperatures are in question. The main factors influencing all these alterations in the nitrogen-content appear to be temperature and water-content rather than the duration of exposure on the kiln.—C. S.

*Acetifying Bacteria, Investigations on.* A. Mayer. Zeits. f. Spiritusind. **21**, [38], 334.

The author has studied the action of certain of the acetic acid-producing bacteria on various alcohols, sugars, and fatty acids. In most cases a decoction of yeast was used as the nutrient medium. The following conclusions are drawn as a summary of the work:—

Ethyl, propyl, butyl, and isobutyl alcohols are oxidised by *B. Pasteurianum* and *B. Kützingianum* to the corresponding acids, but the action, in the case of isobutyl alcohol, is only slight, unless specially favourable nutrient conditions are provided. Methyl and isopropyl alcohols, on the other hand, are unaltered by these organisms, and *B. Pasteurianum* has no effect on amyl alcohol, the action of *B. Kutzingianum* being as yet undetermined.

Ethylene glycol is oxidised to glycollic acid, and the product of the reaction exerts a retardation on the further growth of the bacteria. Both the above-mentioned organisms have only a slight effect on glycerin.

Mannite is not oxidised by *B. Pasteurianum*, whilst *B. Kutzingianum*, unlike *B. aceti Hansen* and *B. aceti Brown*, only converts it into levulose with difficulty.

Only *B. xylinum* oxidises sorbite with the formation of sorbose, and none of the bacteria in question have any action upon dulcite.

Glucose is converted into gluconic acid. *B. Kutzingianum* is more energetic in this respect than *B. Pasteurianum*. The gluconic acid produced tends to retard the development of the organism in both cases.

Neither *B. Pasteurianum* nor *B. Kützingianum* are able to ferment levulose or maltose.

Acetic acid is readily oxidised to carbon dioxide and water by both bacteria, whilst propionic and butyric acids appear to be unaffected.

On the whole, the oxidising power of the acetifying bacteria on the monohydric, primary alcohols decreases with increase in the number of carbon atoms. Of the bacteria studied, *B. Pasteurianum* has the weakest action on the polyhydric alcohols and glucose.

The author finally points out that it is quite probable that some of the organisms which have little or no oxidising effect with yeast-water nutrition, may be found to give totally different results with some other nutrient which stimulates their physiological activity, as, for instance, in the case of the increased action of the bacteria on isobutyl alcohol in the presence of malt wort.—J. F. B.

*Vinegar Bacteria, Beijerinck's Work on the Species of.* F. Rothenbach. Wochenschr. für Brau. **15**, [35], 445—447.

Beijerinck (Centralbl. für Bacteriol. 1898, [6]) deprecates the tendency to regard the numerous vinegar organisms as distinct species when they differ only in some slight, hereditary characteristic. He contends that only a few well-defined types should be recognised as species, and the large number of vinegar bacteria should be classified as varieties of these.

Beijerinck proposes the following four groups as species:—

(1) The bacteria of the "quick vinegar process," represented by *B. aceti Pasteur.*

(2) The beer-vinegar bacteria, represented by *B. rancens.*

(3) Those which give the blue cellulose coloration with iodine and hydriodic acid, such as *B. Pasteurianum.*

(4) Those bacteria which, like *B. xylinum*, bring about the consumption of the acetic acid in the vinegar.

In the present paper the author, after quoting Beijerinck's work at considerable length, points out that *B. aceti Pasteur* does not convert alcohol of such high strength as is worked in the "quick vinegar process," nor with the same rapidity, and cannot rightly be included in the first group.

The author remarks that, in the present state of our knowledge, the only convenient method of classification is from a purely technical standpoint, and proposes the following grouping:—

(1) The true quick-vinegar bacteria of Schützenbach's process.

(2) Those bacteria, intermediate between group 1 and group 3, which are capable of deriving their nitrogen from ammonia and form slight pellicles; *B. aceti Pasteur* belongs to this group.

(3) The true industrial wine, malt, beer, and fruit vinegar bacteria, which form a decided pellicle.

(4) Non-industrial fruit vinegar bacteria.

(5) Non-industrial wort and mash bacteria which oxidise sugar directly and those whose infection causes acidity in brewery and distillery worts.

(6) Pathological bacteria, which cause turbidity or form a leather-like pellicle, as *B. xylinum.*

An alternative classification, according to the pellicle formed is also given:—

A. Those which form no pellicle: the true quick-vinegar bacteria.

B. Those which form a pellicle on the surface of the liquid.

(1) The pellicle is thin but comparatively tough, as with *B. acetigenum.*

(2) The pellicle is thin and easily broken, as with *B. oxydans.*

(3) The pellicle is thicker but easily broken.

  (*a*) Dry pellicle, as with *B. Pasteurianum* and *B. acetosum.*

  (*b*) Slimy pellicle, as with *B. industrium.*

(4) The pellicle is thick and tough, as with *B. xylinum.*
—J. F. B.

*Furfural in Beer, Occurrence and Detection of.* C. Heim. Zeits. für d. ges. Brauwesen, **21**, [21], 258.

*See under* XXIII., *page* 955.

*Wines, Sweet; Determination of Phosphoric Acid in.* F. Glaser. Zeits. f. Untersuch. d. Nahr.- und Genussmittel, 1898, [8], 553.

*See under* XXIII., *page* 960.

## PATENTS.

*Weak Alcoholic Liquids, Impts. in the Utilisation of.* E. J. Johnson, Stratford, Essex. Eng. Pat. 20,955, Sept. 11, 1897.

An ordinary distillery wash instead of being subjected to a process of distillation, is oxidised by any of the well-known methods. The solution of acetic acid thus formed is neutralised with an alkali or alkaline earth, the resulting acetate decomposed with sulphuric or other acid, and the acetic acid distilled off. Claims are made for the above described treatment and utilisation of weak alcoholic liquids and the production of acetic acid from them.—J. L. B.

*Filtering Apparatus, [Wines, Spirits, &c.], Impts. in.* T. F. Seitz, Kreusnach, Germany. Eng. Pat. 5221, March 3, 1898.

The inventor claims:—"A filtering apparatus for wines, spirits, and the like, into which the liquids requiring to be filtered are introduced with the admixture of fibrous filtering material, such as asbestos and the like, characterised by a cylindrical casing of cast metal, provided at the bottom with a flat or grooved distributing cover or plate, wherein the cylinder is provided below with a cylindrical or conical extension, having walls perforated after the manner of a sieve, or made of fine wire gauze or fabric, and constituting the filtering-vessel proper, in which, when it assumes a cylindrical form, can be introduced or can be firmly fixed, a filtering vessel," which is "a second perforated body or appendage of suitable form for increasing the filtering surface, or an impermeable hollow body for the purpose of reducing the said filtering surface, while in both cases reducing the space for the liquid requiring to be filtered." When the filter has a conical filtering vessel, there is "an outer funnel-shaped collecting receptacle with a lower discharge tap and an upper overflow pipe for the purpose of distributing the fibrous material over the perforated walls." In another arrangement, in which the filtering vessel is cylindrical, it has also an "inner conical sieve-like body or appendage, which can be used alone in an inverted position after having been screwed on to a tube provided with a funnel for filtering small quantities of liquids." When an inner conical sieve-like body is used "firmly connected with the outer perforated filtering vessel," there is provided "an upper pipe, for the escape of air, extending from the inner body to the walls of the cast casing or cylinder, as well as a tap operated at the top by a rod with a plug opening upwardly and arranged in the connecting bottom ring below between the inner and outer casings for the purpose of allowing the unfiltered liquid to run off sharply."—R. S.

*Brewing, Impts. in or relating to, and Apparatus therefor.* G. Kreutzer and J. Schmitz, Cologne, Germany. Eng. Pat. 12,660, June 6, 1898.

A wort is run off from the taps at a temperature of 80° C., and passes through a special cooler, which lowers the temperature from 65° to 70° C., before entering the boiling pan. The starch in the wort is then completely converted by the introduction of a "cold deposit," consisting of a diastase solution, either drawn off previously, or prepared separately by a small mash tun of particular construction. The wort is subsequently boiled with hops in the usual manner.—J. L. B.

*Filters for Malt Beverages, Impts. in.* W. H. Wheatley, London. From The Plattsburgh Filter and Process Co., New York, U.S.A. Eng. Pat. 16,304, July 26, 1898.

The claim is for "a filter comprising top and bottom sections, and a series of intermediate sections, having communicating inlet and outlet passages, a foraminous diaphragm interposed between the said sections, and filtering media supported by the said diaphragms, and consisting of sections of filtering material of different degrees of porosity." —J. L. B.

[*Yeast*] *Material suitable for Use as Food, and in the Preparation of Foods of various Kinds; Impts. in the Manufacture or Production of.* J. E. J. Johnson, London. Eng. Pat. 29,183, Dec. 9, 1897.

*See under* XVIII. A., *page* 942.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A*.)—FOODS.

*Proteids of the Pea, Lentil, Horse Bean, and Vetch.* T. B. Osborne and G. F. Campbell. J. Amer. Chem. Soc. 1898, **20**, [7], 410—419.

Legumin is a globulin which forms the principal proteid of the vetch, pea, lentil, and horse bean. By diluting concentrated solutions the legumin separates in a viscid, translucent condition, and this when treated with water becomes opaque and solid. Conglutin from lupin seeds and amandin from almonds behave similarly, as do gliadin, of wheat and rye, and hordein of barley. Legumin is insoluble in pure water, but if the solution from which it is precipitated contain acid, this may combine with the legumin, and the resulting substance, like other acid globulins, will then dissolve in water.

Legumin may be extracted by water from seeds containing this proteid, and it can be precipitated from the aqueous extract by dialysis in water, by the addition of acids, and by calcium salts, and very slightly by free dilution with water. These aqueous extracts react acid with litmus and alkaline with lacmoid. The authors believe that the extraction of legumin by water from seeds is due to the presence of acid potassium salts of phosphoric and organic acids, and that in consequence of the varying proportions of these in the different seeds, amounts of legumin *pro rata* are extracted from them.

Legumin extracted without neutralising the natural acid of the seed and precipitated by dialysis, either directly or after precipitation with ammonium sulphate, is usually converted, to a large extent, into insoluble "albuminate." This "albuminate" differs from the similar insoluble products obtained from most other globulins in that it appears to become hydrated by salt solution and dehydrated by pure water. If, however, the acid of the seed be previously neutralised, the globulin extracted by salt solution yields hardly any "albuminate," which indicates that the latter is a product of the action of the acid of the seed. With nitric acid, Millon's and Adamkiewicz's tests, proteid reactions are obtained. With the biuret test, a violet colour is given, which, on standing, becomes a deep rose-red, similar to that given by peptones. Legumin has been supposed to contain phosphorus and is therefore one of the nucleo-proteids; the authors cannot however, confirm this, many of their preparations being free and others only containing a trace of phosphorus.

Vicilin is a globulin associated with legumin in the pea, lentil, and horse bean. The most remarkable characteristic of vicilin is its content of sulphur, less than that of any other known proteid, and this content diminishes with repeated precipitations. In salt solution, vicilin is far more soluble than legumin, and this circumstance can be made use of for separating the two globulins. 10 per cent. sodium chloride solutions of vicilin become turbid at 90° C., and completely coagulated at 100° C. Vicilin is insoluble in water, but soluble in sodium chloride.

Legumelin is found in most leguminous seeds. Like other proteids, notably edestin, it undergoes a change in solubility, becoming gradually more and more difficult to dissolve.

In their work on the cow pea (this Journal, 1897, 820) legumelin was regarded as a globulin. They now, however, consider this substance to belong to the albumins. The coagulated product is soluble in very dilute alkalis, but insoluble in hydrochloric or acetic acid.

Solutions of the proteose of the pea and horse bean are not precipitated by saturation with sodium chloride, but by subsequently adding acetic acid the proteose is thrown down. Nitric acid in the aqueous solutions of the pea proteose gives no precipitate unless the solution is previously saturated with salt, when a precipitate, soluble on warming and reappearing on cooling, is given by that portion of the proteose precipitable by acetic acid from a salt-saturated solution, while the part not thus precipitable gives only a turbidity. Both these portions of the pea proteose are precipitated by copper sulphate, and give a rose-red biuret reaction.—J. L. B.

*Gluten Preparations free from Starch.* A. J Cowoley. Pharm. J. 1898, **61**, [1473], 327.

The results are given of the determination of starch in a large number of the so-called "Starch-free" commercial glutens. The materials examined contained the following percentages of starch:—Gluten flour, 63·5, 63·4, 61·2, 5·4, and 2·8; bran biscuits, 19·8, 14·4, 14·3, and 6·7; gluten bread, 63·0 and 37·4; gluten macaroni, 65·2; gluten vermicelli, 62·1; and "gluten," 62·1 per cent. It is thus obvious that there is much room for improvement in the preparation of diabetic gluten food, since the majority of the samples examined show the presence of an inordinate amount of amylaceous material.—A. S.

*Butter, Rancidity of, and Detection of Formaldehyde in.* J. Mayrhofer. Zeits. f. Untersuchung der Nahr- u. Genussmittel, 1898, [8], 552—553.

In a German imperial edict on the chemical examination of fats and cheese, there is a test for formaldehyde, which consists in passing a current of steam through the melted fat, and adding an ammoniacal solution of silver nitrate to the distillate. The author states that in his examination on the rancidity of fats, he has arrived at the same conclusions as Schmid (this Journal 1898, 610), finding that rancid butter contains bodies of an aldehydic or ketonic nature, which are volatile with steam, in addition to compounds of an acid character. He has made experiments to determine the quantity of these volatile constituents, by oxidising the distillate with permanganate in alkaline solution, by attempting to estimate the amount of silver, which separates from an ammoniacal solution of silver nitrate, and by determining the degree of coloration obtained with Schiff's reagent, and has obtained promising results by the first method. Farnsteiner (Forschungs Berichte, 1896, **3**, 363 and 369) pointed out that when milk becomes sour, a volatile substance is formed, which reduces ammoniacal silver nitrate, and prevents the detection of formaldehyde by that reagent, and the author considers that in the light of the experiments mentioned above, the test is equally unreliable in the case of butter.—C. A. M.

### PATENTS.

*Proteid Breads, Biscuits, and Cakes, Impts. in.* H. Higgins, Cambridge. Eng. Pat. 19,357, Aug. 21, 1897.

To prevent milk-casein from "binding" when used in making bread, &c., the patentee adds a quantity of gluten which has been heated and so caused to lose its "binding" properties. When casein is used with starchy materials, the "non-binding" material is unnecessary.—W. P. S.

*Tea, Elimination of Tannin from.* A. W. Rothwell, Forest Hill, Kent. Eng. Pat. 20,527, Sept. 7, 1897.

A current of oxygen (more or less pure) is passed into the infusion, and the resulting "tannate which floats in the liquid in the form of minute particles of semi-solid matter" is removed by filtration.—F. H. L.

*Acidity of Milk and other Liquids, Impts. in Apparatus for Testing the.* A. W. Stokes, London, and R. A. Lister and Co., Dursley, Gloucester. Eng. Pat. 22,027, Sept. 25, 1897.

*See under* XXIII., *page* 919.

*Organic Substances, Process of Preserving.* H. A. Lacroix and A. C. de Caudemberg, Paris. Eng. Pat. 22,112, Sept. 27, 1897.

The substances to be preserved (such as meat, game, fish, &c.) are soaked in a bath formed by mashing hay or poplar leaves in cold water, with the addition of 1 to 5 per cent. of nitre. After steeping, the meats are air- or sun-dried.—W. P. S.

*Foodstuff, suitable also for Mixing with Flour and other Foodstuffs, an Improved.* E. B. Watson, London. Eng. Pat. 27,554, Nov. 23, 1897.

The substance claimed is a specially prepared pea-nut meal. The nuts are decorticated and sifted, to remove the skins and germs, and then subjected to a moist heat of 212° F. The oil is now removed by pressing, and the cakes are ground up in the usual way. A small quantity of hypophosphite of lime or soda may be added, and the flour may be heated to drive off moisture in order to improve its flavour or to modify its colour.—W. P. S.

[*Yeast*] *Material Suitable for Use as Food, and in the Preparation of Foods of Various Kinds, Impts. in the Manufacture or Production of.* J. E. J. Johnson, London. Eng. Pat. 29,183, Dec. 9, 1897.

The material is prepared by heating yeast with a half per cent. solution of hydrochloric acid in a closed vessel for 1 to 2 hours at a pressure of 2½ atmospheres. The contents are then filtered, the filtrate is neutralised, and is evaporated to dryness or only to a semi-liquid state.—W. P. S.

*Albumin, An Improved Process for the Production of Milk-like Solutions of.* J. Welcker, Cologne. Eng. Pat. 11,599, May 23, 1898.

The claims are for the production of artificial milk, and more especially of artificial human milk, by building it up from its various constituents. Casein obtained from cow's milk and albumin are mixed with butter fat, and made into an emulsion in a slightly alkaline liquid. The correct quantity of mineral matter and milk sugar dissolved in the proper percentage of water still required, are then added. All the ingredients are sterilised, preferably before mixing. The final product is then stored in sterilised bottles.

Medicinal milk preparations may be prepared as above, therapeutic substances being added during the manufacture. —W. P. S.

*Food for Horses, Cattle, Sheep, Swine, and the like; An Improved.* G. Perron, Frankenthal, and J. Mayer, Neustadt, Germany. Eng. Pat. 12,630, June 6, 1898.

The food consists of equal quantities of dried brewers grains and molasses-turfmeal. The latter is obtained by mixing 80 parts of molasses with 20 parts of turf or peat-moss meal, drying the mixture and powdering it.—W. P. S.

*Casein and its Derivatives, Process for the Production of Water-soluble Compounds of.* M. Riegel and J. A. Rose, Cologne, Germany. Eng. Pat. 2287, Jan. 28, 1898.

The patentees claim the processes for producing compounds of casein and its derivatives soluble in water, which consist in treating these substances or mixtures of them and calcium chloride with alkali alcoholates prepared from anhydrous alcohol.—A. C. W.

*Artificial Butter and the like, An Improved Process for the Production of.* J. Wohlgemuth, Berlin. Eng. Pat. 15,535, July 15, 1898.

The claims are for a process for the production of artificial butter by adding to margarine a quantity of the glycerides of the volatile fatty acids (butyric, caproic, caprylic acids, &c.).—W. P. S.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Water Supplies of Yorkshire.* T. Fairley. Jour. Fed. Inst. Brewing, 1898, **4**, [5], 393—405.

The waters found in Yorkshire differ greatly in composition according to the place at which they are found, the variations being due to the wide range in the character of the rocks and strata. The softest waters, containing up to 7 grains per gallon of solids, are found in the north, north-west, and west, where the rocks are principally igneous or sandstone. Waters of moderate softness, with 7 to 15 grains of solid matter per gallon, come from the carboniferous limestone in the north-west of the county. Wells of a moderate depth also yield waters of this class. Hard waters, containing from 15 to 30 grains of solid per gallon, are obtained from wells near the limestone, from the coal measures of the south-west, the triassic deposits in the vale of York, and also from the oolitic and lias formations in the north-east. Deep-well waters, with 30 to 60 grains per gallon, are to be obtained from the coal measures and triassic deposits in the centre of the county, and from the magnesian limestone. Saline waters, containing 45 to 60 grains of solids, are also found at greater depths in the same strata.

Waters, rising from the red sandstone, contain a considerable quantity of calcium sulphate, and dissolved solids from 50 to 150 grains per gallon.

Alkaline waters containing sodium carbonate and sulphate are found in the coal measures and in other parts. Free sulphuric acid is sometimes found in colliery waters, due to the oxidation of the sulphur in the coal.

Mineral waters are found in various parts of the county.

In the original paper the author has tabulated the different waters so as to illustrate their general composition. —W. P. S.

*Sewage Analysis and Standards of Purity for Effluents.* C. G. Moor. Analyst, 1898, **23**, [269], 198—209.

In this paper the author discusses the variations in quality which characterise sewage coming from different sources, and suggests a scheme for observations which would be applicable in the majority of cases where the efficiency of the purifying process is to be judged. This scheme includes the noting down of local causes of variation in the sewage, such as source, rainfall, volume, &c., and also the physical and chemical examination of the sewage and effluent. Particular value is attached to the determination of the "loss on ignition." As regards the bacteriological investigation, the writer concludes that the same "would be no light matter; and in the present state of our knowledge it could not yield results of anything like the practical value that we can derive from a chemical examination."

The author further suggests a maximum limit of impurity which should in any circumstances be allowed.—W. P. S.

### PATENTS.

*Sewage and other Foul Waters, An Improved Method of Purifying, by Means of a Secondary Process following other Primary Processes.* J. B. Petrie, Milnrow, near Rochdale, Lancashire. Eng. Pat. 17,261, July 22, 1897.

The effluent from settling tanks is brought into contact with ferric salts in order to oxidise the dissolved putrescible matter, and thus do away with the necessity for large area filtration.

Instead of the direct use of ferric salts, a solution of ferrous salts, through which a current of electricity is passed, may be employed; or an electric current may be passed through the settled effluent without previous addition of ferrous salts.—N. H. J. M.

*Sewage and other Impure Water, Impts. in the Method of, and Apparatus for Purifying and Softening, or Similarly Treating.* W. Watson, Bradford, Yorkshire. Eng. Pat. 21,982, Sept. 25, 1897.

The water, contained in a tank provided with a sump, is forced by means of compressed air, steam, or a mixture of air and steam, into a cylindrical vessel. The liquid enters this vessel through a delivery tube extending a considerable distance from the top and opening horizontally; so that the contents of the cylinder acquire a gyratory motion. An acid, or other suitable liquid is fed by a pipe opening immediately adjacent to the orifice of the delivery pipe. For removing scum, skimmers are provided which may be adjusted at any required depth. The liquid overflows, passing through a baffle chamber into a cylindrical neutralising vessel, with a conical bottom, provided with a pipe supplying the neutralising agent. The precipitate may be discharged by means of an internal chamber near the bottom. When neutralised, the liquid passes through a baffle box to the softening chamber which can be heated by live or exhaust steam.—N. H. J. M.

*Sewage and other Foul Liquids, Impts. in and connected with the Filtration of.* H. F. Hibbert, Chorley, Lancashire. Eng. Pat. 22,500, Oct. 1, 1897.

In order to avoid disturbing the surface of a filter of sand by the flow of liquid, it is proposed to discharge the liquid into channels (constructed of wickerwork, for instance) arranged on the surface of the filter. These channels consist of two parallel walls and a bottom, with or without foot-plates at each side (of the same perforated material) to prevent disturbance from the liquid flowing through the walls.—N. H. J. M.

*Treating, Softening, and Purifying Water, Sewage, Slimes, or other Liquids or Matters held in Solution or Suspension; Impts. in or relating to Machinery or Apparatus for.* W. H. L. Galloway, Johannesburg, South African Republic; and Inverurie, N.B. Eng. Pat. 20,428, Oct. 12, 1897.

*See under* I., *page* 905.

*Sewage Water or other Liquids, Impts. in connection with Filter Beds for Filtering.* R. O. Wynne-Roberts, Oswestry, Shropshire. Eng. Pat. 24,276, Oct. 20, 1897.

An automatic arrangement for effecting, by means of intermittent tilting, the discharge of filtered water from each of several filters in succession.—N. H. J. M.

*Burning Refuse Matter or other Material, Impts. in Apparatus or Furnaces for.* J. E. Slack, Old Trafford, Manchester. Eng. Pat. 25,118, Oct. 29, 1897.

The furnace consists of two adjacent cells with an intervening bridge, over which the gases,—given off by the refuse, &c., in that cell which is freshly charged,—pass into the other cell (which is at its hottest) on their way to the common combustion chamber, the direction of the draught being changed by dampers.—C. S.

*Sewage and other Liquids, Impts. in Apparatus for the Treatment of.* D. Cameron, F. J. Commin, and A. J. Martin, Exeter. Eng. Pat. 27,349, Nov. 22, 1897.

*See under* I., *page* 905.

*Effluent Water of Factories and the like, also River Water and Sewage, Improved Means for Purifying the.* W. Greig and W. E. Greig, Dartford, Kent. Eng. Pat. 29,070, Dec. 8, 1897.

The effluent water of chemical works, &c. is passed through a series of closed settling chambers, connected by means of upturned pipes, to prevent washing out of the sediment, and a filtering tank. The latter may be of any required extent, and is provided with any required number of compartments each having a filtering receptacle filled with ashes and covered with fine-meshed wire gauze. Each settling chamber has an inclined bottom with an outlet, and is also provided with a manhole to facilitate access to its interior.
—N. H. J. M.

### (C.)—DISINFECTANTS.

*Disinfection, A new Method of.* R. Walther and A. Schlossmann. J. Prakt. Chem. 1898, **57**, [12], 512—534.

Further details are given with regard to the method of disinfection briefly described in this Journal, 1898, 484. It is essential that the air of the room and all permeable substances in it should be thoroughly damp before application of the germicide. This may be accomplished by a preliminary spraying with water, 10 lb. per 1,000 cubic feet being the quantity recommended. The same result may be attained by the admixture of a hygroscopic substance with the active agent. A solution containing 30 per cent. of formaldehyde and 10 per cent. of glycerin is the most suitable. The spray is so finely divided that the quantity of glycerin collected on clothing, even after 10 exposures to the process, is insufficient to cause permanent dampness. The apparatus consists of a ring-shaped copper kettle in which water can be boiled by a large spirit flame; 1·5 litre of water is brought to the boil in six minutes. The steam enters the reservoir (which is surrounded by the kettle) containing the formaldehyde solution, forcing the solution out through small orifices. One apparatus is required for about each 2,500 cubic feet, which space is entirely permeated within 10 minutes after the apparatus commences to work. Disinfection is complete after three hours.
—A. C. W.

## XIX.—PAPER, PASTEBOARD, Etc.

*Rosin Size, The Production of, under Pressure.* Papier Zeit. 1898, **23**, [66], 2455. From a Lecture by Mr. Fr. Arledter at Strassburg, June 7, 1898.

According to the theory that it is the free rosin which acts as the principal sizing agent in rosin soap, it is necessary to make a rosin soap containing a high percentage of free rosin, and in which the rosin particles are as small as possible. Neutral rosin soap may be regarded as a solvent for free rosin, in the same way as alcohol or ether.

An excellent rosin size, fulfilling the above conditions can be obtained in a simple manner by boiling rosin with alkali and water, with continual stirring and under pressure. As there is no fear of the froth overrunning in a closed boiler, the rosin can be treated at an even temperature and pressure from start to finish, and in consequence of the pressure being even, the process of boiling is everywhere uniform. In this way the highest absorbing capacity of the neutral liquid for free rosin is reached. By the microscope, it is seen that the particles of free rosin are only about one-tenth of the size of the particles in the soap made by the old system.

Soon after starting the boiling in open vessels, layers are formed, due to the varying specific gravities of the different resins contained in ordinary commercial rosin. The heavy bottom layers are first saponified, whilst the lighter layers on the top are not sufficiently boiled, and the easily volatile resins and ethereal oils are volatilised and escape. This is all prevented by using closed boilers.

By Dr. Boek's treatment, carbonic acid is passed into the solution to improve the size. If common sodium carbonate be used in closed boilers, the carbonic acid can be collected and passed into the boiled size.

Rosin soap, when saturated with free rosin, has still the capacity for taking up starch. The latter can be boiled at the same time, the cells very soon bursting on boiling under pressure. This mixture of rosin and starch is, however, only required in a few cases.

Very little attention is required when using closed boilers, for once the boiler is filled and revolving, it is only necessary to watch the pressure gauge. When the boiling is finished, the steam is shut off, and the rosin soap is driven through pipes into the storage tanks by the pressure remaining in the boiler. A filter is connected with the piping, consisting of cylindrical tubes filled with coke, asbestos, charcoal, &c., so that well filtered rosin soap is delivered from the pipes. These filters are cleaned with steam, and changed about once every fortnight. If the rosin soap be filtered through charcoal under pressure, the colouring matter is partially retained. The rosin may also be discoloured by melting it in the boiler with 3 or 4 per cent. of alkali, the colouring matter being extracted. The milk made from such discoloured rosin is blue-white.

The rosin is ground in ball mills, consisting of a perforated revolving cylinder with a wire jacket. The ground rosin falls into a chest, and is then in a condition suitable for boiling.

The rosin soap is obtained in solution by treating with cold water in agitators.

By the use of closed boilers about 30 per cent. of fuel can be saved, and the time required for boiling is reduced to about one-half. Double cylindrical or spherical boilers are the most suitable, and old boilers can be reconstructed cheaply for the purpose.—S. P. E.

*Lignorosin* [*"Calcium Lignosulphite"*], *A New Reducing Agent for Chromic Acid in Wool Mordanting.* A. Raaz. Färber-Zeit. 1898, **9**, [16], 245.

*See under* VI., *page* 923.

## PATENTS.

*Peat, Impts. in the Treatment of, to obtain therefrom Material for the Manufacture of Paper and for other Purposes.* Adolf Feszty, Esterhaz, Austria Hungary. Eng. Pat. 15,601, June 30, 1897.

The peat is washed and boiled in dilute acid, or solutions of acid sulphate salts, the time of boiling depending on the pressure, which may vary from two to seven atmospheres. After boiling, the liquid is drawn off, and the peat thoroughly washed with clean water. According to the kind of fibre required, the peat material is boiled with a caustic alkaline solution or an alkaline carbonate solution, with continual stirring and under pressure. The increase of pressure allows a reduction of alkali, and under 10 to 15 atmospheres, no alkali need be used.—S. P. E.

*Pulp and Sawdust, Non-inflammable; Impts. relating to.* E. M. Fox, London. Eng. Pat. 20,664, Sept. 8, 1897.

Pulp made from wood or other material is digested in a solution of 1 lb. of ammonium phosphate and 2 lb. of ammonium sulphate in three gallons of water, at a temperature of about 120° F. for about 5 hours. The pulp thus treated is pressed and dried for manufacture into paper, &c. Sawdust may in like manner be rendered non-inflammable, and be then applied to the jacketing of boilers and other purposes. Reference is made to Eng. Pat. 7,032, 1896. (This journal, 1896, 596).—E. S.

*Paper, Impts. in the Manufacture of.* Sidney Jaques, Hylton, near Sunderland. Eng. Pat. 30,579, Dec. 28, 1897.

The pulp or half stuff, as it passes the filtering or straining machine is conveyed by a travelling belt to pressing rolls, the water containing a certain percentage of fibres being returned to the filters.

After bleaching the pulp, the excess of spent liquor is removed in the same manner. After passing these squeezing rolls, the pulp is passed into an apparatus consisting of a cylindrical vessel having a number of perforated transverse partitions with intervening blades or arms, the latter being mounted on a shaft, driven in any suitable manner. In this apparatus the pulp is subjected to a separating or beating action. An air relief pipe is provided, through which the air, water, and pulp are conveyed back to the box from which the beating apparatus or refiner is fed.
—S. P. E.

*Cellulose, Impts. in the Preparation of, for Use in the Manufacture of Paper and for other Purposes.* Friedrich Bühler, Cassel, Germany. Eng. Pat. 6,651, March 18, 1898.

In place of the alkaline or acid lyes at present in use for obtaining cellulose from wood, the inventor proposes using tar oil, phenol, or phenol esters. This liquid is a product of the dry distillation of wood or coal. The tar thus obtained is distilled, and the benzol, naphtha, and water are first driven off, which takes place at 160°—180° C. The so-called tar oils, which distill off from 180° C. to 320° C., form the liquid required, and this consists mainly of phenols and allied substances having the general formulæ $C_6H_6O$ and the derivatives $C_6H_5(OH)$, $C_6H_4(OH)_2$, $C_6H_3(OH)_3$, and so forth. These are mainly "phenol, tricresyl, guaiacol, cresol, and like phenols."

The boiling with this liquid takes from 3 to 12 hours, and may be carried on in an open vessel, as no deleterious gases are given off. The boiled material is pressed and treated with a solvent to remove the tar oil, and is then ready for treatment into paper or like product.—S. P. E.

*Cellulose or Paper Pulp and the like, Impts. in or relating to Apparatus for separating Impurities from.* Albert Aberg Gorz, Austria. Eng. Pat. 12,096, May 27, 1898.

The apparatus consists of a vessel tapering downwards, into which the cellulose pulp is introduced and caused to rotate. Laths or ribs are fastened on to the interior of the vessel at certain distances apart, against which the movement of the mass produces eddies or dead corners in which the specifically heavier portions settle down.—S. P. E.

# XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Monazite Sand, Yttrian Earths of.* G. Urbain. Comptes Rend. 1898, **127**, [2], 107–108.

By fractional precipitation of the ethyl sulphates of the earths of the yttrium series, a preliminary separation is effected into groups which can subsequently be more readily subdivided. The difficulty of completely separating yttrium from small quantities of the terbian earths ("terbines") is increased by the fact that the furthest limit of fractionation is reached with a mean atomic weight of 97. The terbian earths are for the most part precipitated by potassium sulphate, so that on attempting to free the yttria from the last traces of didymia by means of that salt, the yttria remains contaminated with a small quantity of these earths, and the mixture with an atomic weight of about 97, shows a remarkable stability. Schutzenberger and Boudouard were the first to note this phenomenon, but did not explain its nature. The author, however, isolated from this mixture yttrium with an atomic weight of 89, and earths of the terbian series with an atomic weight of 151·4, a number which he regarded as probably below the truth. In the last fractions of the ethyl sulphate precipitations, which contained only yttrium, true erbium, and ytterbium, he was able to easily separate the yttrium by precipitation with ammonia. Attributing this to the absence of terbian earths, he made experiments to determine whether these could not be concentrated in the first fractions of a repeated crystallisation of the ethyl sulphate precipitates (initial atomic weight, 97), just as the didymia was accumulated in the preceding fractions. This was found to be the case, and after a sufficient number of crystallisations, the different fractions were converted into oxides, the colour of which varied from orange (first fractions) to pale rose (last fractions). The latter only contained a trace of erbium, which could be removed by crystallising, the oxalates from nitric acid. The last mother liquids contained the yttrium (atomic weight = 89). The coloured oxides of the terbian earths were characterised by yielding formates which were but slightly soluble. Examined spectroscopically only two bands could be observed in the visible part of the spectrum.

| Microm. | λ | —— | |
|---|---|---|---|
| 70 | 0·0000 | Hardly perceptible...... | Holmium. |
| 122—130 | 0·0000 | Intense.................. | Dysprasium. |

The author concluded that these characteristics showed the complexity of this substance; but that his experiments proved that the yttria of monazite sand is composed entirely of yttrium, with an atomic weight of 89, and that there is no element present with an atomic weight of about 100 or 97.—C. A. M.

### *Colloidal Silver.* A. Lottermoser and E. von Meyer. J. Prakt. Chem. 1898, **57**, [12], 540–543.

In a former communication (this Journal, 1897, 1,035) the amounts of different acids required to convert colloidal into ordinary silver were found by a titration method. The authors have now determined the time required for this conversion under the influence of different quantities of acid on solutions of varying strength. The acid is added all at once, and the end point taken when a drop of the mixture on a glass plate over white paper clearly shows the separation of silver particles. With equal concentration, the longer time is required the less the quantity of acid, and the quantities of acid which cause the change in solutions of different concentration in the same time, are inversely proportional to the concentration. Reducible metallic chlorides cause the formation of silver chloride and insoluble silver. But if the solutions be very dilute, the silver chloride and the insoluble lower chloride often do not separate, but remain in colloidal solution. This is the case with mercuric chloride. Colloidal silver iodide would appear to be adapted to therapeutic but not photographic uses.

—A. C. W.

### *Strychnine.* J. Tafel. Annalen, 1898, **301**, [2 and 3], 285—348.

On reduction by hydriodic acid and phosphorus, strychnine forms de-oxystrychnine, $C_{21}H_{26}N_2O$ (Annalen, **268**, 235). The raw products of this reaction by treatment with sodium in boiling amyl alcohol solution yield strychnoline, $C_{21}H_{26}N_2$. By electrolytic reduction of strychnine and de-oxystrychnine the author has now obtained three further products. The essentials of the process are as follows:—A solution of 30 grms. of strychnine in 180 grms. of sulphuric acid and 120 grms. of water, was placed in a porous cell surrounded by 20 per cent. sulphuric acid, containing, as anode, a hollow lead cylinder. The cathode consisted of a lead plate, and also served as stirrer. Two cells were used in series, with a current of 5 ampères, and a tension of rather over 3·5 volts for each cell. After 10 hours the colourless liquid was diluted, nearly neutralized with barium carbonate, and filtered. The boiling filtrate was made alkaline with baryta water, and any excess of baryta was precipitated by carbonic acid. On filtering, the residue contains mainly strychnidine, $C_{21}H_{24}N_2O$, whilst tetrahydrostrychnine, $C_{21}H_{26}N_2O_2$, is found in the filtrate.

*Strychnidine* is obtained from the residue by the action of excess of sulphuric acid, and precipitation by caustic soda. Strychnidine is a crystalline substance melting at 250°·5 C., and very slightly soluble in water, the solution being alkaline to litmus, and fairly soluble in alcohol and benzene.

Strychnidine forms, with one equivalent of acid, stable salts of neutral reaction to litmus, and with two equivalents of acid, salts with an acid reaction towards litmus, and which readily lose acid. It forms an addition compound, $C_{21}H_{24}NO.CH_3I.2H_2O$, with methyl iodide.

*Tetrahydrostrychnine* is obtained as sulphate on the evaporation *in vacuo* of the acidified solution of the base, resulting from the reduction of strychnine. The base is obtained by the action of caustic soda and may be recrystallised from benzene, and finally from alcohol. It crystallises in colourless prisms with 1 mol. of alcohol, which is lost at 100° C., but not over sulphuric acid *in vacuo*. The base melts at 202°, and distils at a higher temperature with little decomposition.

*Strychnoline*, $C_{21}H_{26}N_2$, is prepared by precipitating the hydriodic acid solution of de-oxystrychnine with alkali, dissolving in amyl alcohol, and boiling the solution with sodium. After addition of water and expulsion of amyl alcohol, alkalis produce a gelatinous precipitate, which, by treatment with ether, becomes crystalline. Strychnoline melts at 175°—178° C. and is almost insoluble in water,

*Dihydrostrychnoline*, $C_{21}H_{28}N_2$, is obtained by the electrolytic reduction of de-oxystrychnine. It melts at 129° C., and distils undecomposed at 267°—270° under 16 mm. pressure. It is very soluble in alcohol, ether, acetone, chloroform, acetic ether, and benzene; it is slightly soluble in water, and the solution has an alkaline reaction.

*Colour Reactions.*—Strychnidine and tetrahydrostrychnine give no coloration on the addition of oxidising agents to a solution in concentrated sulphuric acid. The solution of strychnoline in sulphuric acid is deep red to violet, the solution of dihydrostrychnoline becomes red on strongly heating, and then brown. The solutions of strychnidine, tetrahydrostrychnine, strychnoline and dihydrostrychnoline, in dilute acids give intense colorations with oxidising agents, of which ferric chloride gives the most delicate reaction. A solution of 1 grm.-mol. of strychnidine in 500 litres of $\frac{1}{100}$N-hydrochloric acid becomes rose-red on the addition of dilute ferric chloride, on standing or warming, the coloration becomes deep blood-red; the reaction becomes uncertain on dilution to 1 in 500,000. Methyltetrahydroquinoline (Kairoline) gives a reaction of equal delicacy, as do also tetrahydrostrychnine and dihydrostrychnoline, but the coloration due to the latter is fugitive and soon becomes yellow; strychnoline produces an unstable magenta. A test for strychnine is founded on the colorations produced by its reduction products:—A solution of the alkaloid in warm dilute hydrochloric acid after reduction with a small piece of zinc or sodium amalgam gives a yellowish red colour, not destroyed on boiling, on the addition of ferric chloride to the still slightly acid solution. The solution of strychnidine of the strength mentioned gives, on the gradual addition of dilute bromine water, a rose-red colour turning to the tint of potassium permanganate solution.

*Physiological Action.*—Strychnoline and dihydrostrychnoline, though poisonous, do not produce the peculiar convulsive effects of strychnine. Strychnidine has not so powerful an action as strychnine, and de-oxystrychnine is less powerful still. The violent action of strychnine is to be attributed to the presence of two groups in the molecule, each of a similarly active character.

*Action of Nitric Acid on Strychnine.*—Boiling 5 per cent. nitric acid converts strychnine into the nitrate of dinitrostrychnine hydrate, a golden yellow crystalline powder soluble with difficulty in hot water. This substance, $C_{21}H_{23}N_5O_{10}$, appears to be identical with the kakostrychnine of Claus and Glassner (Ber. **14**, 774).—A. C. W.

### *Atroscine of Hesse and i-Scopolamine of Schmidt, Identity of.* J. Gadamer. Chem. Zeit. 1898, **22**, 227. From Arch. Pharm. 1898, **236**, 382.

The atroscine of Hesse and the *i*-scopolamine of Schmidt are hydrates of the same alkaloid; the former containing two, the latter one molecule of water. Hesse's atroscine is the labile form of *i*-scopolamine, and is only obtained under certain definite conditions. Scopolamine is converted into the inactive modification by an alcoholic solution of caustic soda. The author proposes that the name "atroscine" should be abandoned.—T. E.

### *Dioscorine.* Plugge and Schütte. Pharm. J. 1898, **61**, [1469], 217. From Arch. Intern. de Pharm.

Dioscorine, $C_{13}H_{19}NO_2$, was first isolated in a crystalline form by Boorsma, from *Dioscorea hirsuta*, one of the most poisonous of the Yam tribe, growing in Java. The pure alkaloid melts at 43°·5 C., is monobasic, and belongs pharmacologically to the picrotoxin group of poisons.—A. S.

### *Aloes, Chemistry of.* A. R. L. Dohme. Amer. J. Pharm. **70**, 398; Pharm. J., 1898, **61**, [1473], 325.

The following are the chief results of investigations carried out by the author, and also by Tschirch and G. Pedersen:—

Socotrine, Curaçoa, and Cape aloes gave respectively 7·5, 18·5, and 4·5 per cent. of aloin. The Curaçoa variety is as efficient as that from Socotrine, and is cheaper, and moreover it forms the greater portion of the so-called Socotrine aloes of commerce. The resin of aloes is an ester and varies according to the kind of aloes. The varying constituent appears to be the acid, the alcoholic part—aloresinotannol—being the same in the two varieties so far examined, *viz.*, Barbadoes and Cape aloes. The laxative property of aloin is probably due to the emodin contained in it, many laxative drugs — senna, cascara sagrada, rhubarb, buckthorn—owing this property to the emodin or other similar substance derived from anthraquinone and isomeric with it.—A. S.

*Theobromine, Homologues of.* H. Brunner, Schweiz. Wochenschr. Pharm. **36**, 303—331; Chem. Centr. 1898, **2**, [7], 474.

The author discusses the derivatives of theobromine, prepared by him in conjunction with Leins (this Journal, 1898, 78). Experiments carried out by the author and Reiss account for the difference between their views and those of Van der Sloten (Apoth. Zeit. **12**, 5), in that two kinds of homologues of theobromine exist, of which the one variety, the real homologues, crystallise with difficulty and melt above 270 C., whilst the other kind, that may be described as homologues of caffeine, embrace derivatives which crystallise easily in long needles and have a lower melting point. Theobromine silver may be used for the preparation of both series of compounds. With methyl iodide, only caffeine was obtained, but two isomers were produced with ethyl iodide, the one a crystalline powder with melting point above 270°, and the other, which crystallises out from the mother liquor, with a melting point of about 165 C. The latter compound is identical with that obtained by Van der Sloten, whilst the first-named corresponds with the one described by Philips (Ber. **9**, 1308). Propyl iodide appears to give only the micro crystalline modification with the high melting point.—A. S.

*Menthone of Bourbon Geranium Oil.* J. Flatau and H. Labbé. Bull. Soc. Chem. 1898, **19**, [16—17], 788—790.

The essential oil was saponified and the alcohols converted into phthalic esters, the sodium salts were well washed with ether. The residue on evaporation of the ethereal solution formed about 15 per cent. of the original oil, and by fractionation it yielded about 40 per cent. of menthone. By a comparison of the melting points of the semi-carbazones of natural and *l*-menthone, the menthone extracted from the geranium oil was identified as the natural variety. Bourbon geranium oil is of remarkably constant composition, it contains, in addition to 8 per cent. of menthone, 10 per cent. of geraniol and 70 per cent. of citronellol.—A. C. W.

*Fruit Juices, a Constituent of, which reduces Fehling's Solution.* Aderhold and Heintze. Chem. Zeit. 1898, **22**, [63], 632.

Finding discrepancies between the sugar-content of expressed melon-juice and that of the water-extract of dried cucumbers, the authors endeavoured to remove the pectins from the fresh juice by alcohol. Addition of a comparatively small amount of alcohol precipitated the pectin; but when to the filtered liquid a larger quantity of alcohol was added, a yellowish-brown resinoid substance was deposited, which acted as a powerful reducer of Fehling's solution. The authors have found the same body in other fruit juices (gooseberries, cherries, apples), more abundantly in unripe than in ripe fruits. The substance is readily soluble in water, and when the solution is evaporated and the residue dried on the water-bath for some hours, the reducing power disappears, though the dried residue is still soluble in water. This behaviour probably accounts for the authors' original observation. Further researches are in progress.—J. T. D.

*Sassafras, Oil of.* H. C. La Wall and R. C. Purcell. Amer. Jour. Pharm. 1898, **70**, 310—312.

This oil is frequently adulterated but admixture is not easily detected. Safrol, which constitutes nine-tenths of the pure oil, was formerly used as an adulterant, but it is now dearer than the oil. The chief constants for judging the purity of the oil are its specific gravity, general appearance, and odour. The specific gravity of safrol is 1·108, whilst that of sassafras oil varies from 1·07 to 1·09. A lower gravity than 1·07 points to adulteration with fractionated camphor oil. As crystals of safrol separate out in cold weather, care must be taken to obtain a homogeneous mixture before determining the specific gravity. During the last three years the authors have examined a considerable number of samples, with the following results:—

| — | Maximum. | Minimum. | Mean. |
|---|---|---|---|
| 1896 | 1·0840 | 1·0500 | 1·0654 |
| 1897 | 1·0850 | 1·0610 | 1·0734 |
| 1898 | 1·0830 | 1·0450 | 1·0713 |

The pure oil varies in colour from deep yellow to colourless.—W. P. S.

*Ammonium Citrate Solution, Method of Preparing a Neutral.* A. D. Cook. J. Amer. Chem. Soc. **20**, [8]. 585—586.

To prepare a neutral solution of ammonium citrate the author dissolves 740 grms. of commercial citric acid in 1,900 c.c. of a 10 per cent. solution of ammonia by vigorously stirring the mixture, whereby sufficient heat is developed to expel the excess of ammonia. The volume is then made up to 4,000 c.c. and the liquid again well stirred. After remaining overnight in a large evaporating basin, the solution contains large crystals and is neutral; it is finally brought to 20° C. and diluted until of sp. gr. = 1·09.

—E. W. W.

*Quinine, Detection of.* E. Pollacci. Gaz. chim. ital. **28**, [1], 891; Chem. Centr. 1898, **2**, [5], 386.

*See under* XXIII., *page* 955.

*Boric Acid, Quantitative Determination of.* C. Montemartini. Gaz. chim. ital. **28**, [1], 344; Chem. Centr. 1898, **2**, [4], 314.

*See under* XXIII., *page* 951.

*Boric Acid, Estimation of as Potassium Borofluoride.* Thaddeeff. Rev. intern. falsific. **11**, 98; Chem. Centr. 1898, **2**, [4], 314.

*See under* XXIII., *page* 953.

*Formic Acid, Determination of, in presence of Acetic Acid, and of Easily Oxidisable Organic Substances.* A. Leys. Monit. Scient. 1898, **12**, Sept. 1898, 632.

*See under* XXIII., *page* 957.

*Iodoform, Estimation of.* G. Meillere. Ann. Chim. Analyt. **3**, [5], 153; Analyst, 1898, **23**, [270], 233.

*See under* XXIII., *page* 960.

*Orange Oil and Bergamot Oil, Adulteration of with Oil of Turpentine; New Method for the Detection of.* G. Mancuso-Lima. Staz. sperm. agric. ital. **31**, 244; Chem. Centr. 1898, **2**, [9], 585.

*See under* XXIII., *page* 955.

*Caffeine and Theobromine, Separation and Quantitative Determination of.* H. Brunner and H. Leins. Schweiz. Wochenschr. Pharm. **36**, 301; Chem. Centr. 1898, **2**, [7], 512.

*See under* XXIII., *page* 961.

### PATENTS.

*Adenine, Impts. in the Manufacture or Preparation of.* J. Y. Johnson, London. From C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 23,004, Oct. 7, 1897.

The claims are for the production of adenine (6-aminopurine) or its alkyl derivatives, by the action of reducing agents (hydriodic acid and phosphonium iodide) on 6-amino-2.8-dichloropurine or its alkyl derivatives.—A. C. W.

*Heteroxanthine, Paraxanthine, and Methylated Hypoxanthines; Impts. in the Manufacture or Preparation of.* J. Y. Johnson, London. From C. F. Boehringer and Soehne, Waldhof, Mannheim, Germany. Eng. Pat. 23,005, Oct. 7, 1897.

Heteroxanthine is obtained by treating the 7-methyl-2.6-dichloropurine obtained by the action of phosphorus oxychloride on theobromine, with a mineral acid. Paraxanthine is prepared by converting the same methyldichloropurine into methyloxychloropurine by the action of dilute alkali, methylating the latter compound, and heating the product with mineral acid. The 7-methyl-6-oxy-2-chloropurine obtained in the last process yields on reduction 7-methylhypoxanthine, from which 1.7-dimethylhypoxanthine may be prepared. The processes described are claimed.—A. C. W.

*Phenolic Carbonates, Impts. in the Production of.* C. J. Mewburn, London. From Chemiscke Fabrik von Heyden, Radebeul, Germany. Eng. Pat. 24,793. Oct. 26, 1897.

The claims are for the method of preparing phenolic carbonates, which consists in heating the phenol, or one of its salts, with a carbonic ester or chlorocarbonic ester. To prepare guaiacol carbonate, diphenyl carbonate, or any aromatic carbonate with a lower boiling point than guaiacol carbonate may be employed. The transformation with alcoholic carbonates is more difficult. If the alkyl chlorocarbonic esters are employed, the alkyl carbonate of guaiacol is formed as an intermediate product, which is decomposed on heating, either alone or in presence of excess of guaiacol—

$$2CO(OC_2H_5)OC_6H_4.OCH_3 = CO(OC_2H_5)_2 + CO(OC_6H_4.OCH_3)_2$$

$$CO(OC_2H_5)OC_6H_4.OCH_3 + C_6H_4(OH)OCH_3 = CO(OC_6H_4.OCH_3)_2 + C_2H_5.OH.$$

—A. C. W.

*Tonic and Medicinal Preparation or Composition [Hop Extract], An Improved, applicable also for the Manufacture of Yeasts, Bitters, Beer, and the like.* F. C. Doolittle, Waterville, United States. Eng. Pat. 6238, March 14, 1898.

The patentee mixes powdered hop lupulin with semi-fluid hop extract in stated proportions, and prepares pills of the desired shape and size, which are subsequently coated in the usual manner.—J. L. B.

*Sandal-wood Oil, Impts. in or relating to the Treatment of, for the Production of Santalol.* C. A. Steche, H. Steche, and T. Habenicht, Leipzig, Germany. Eng. Pat. 13,965, June 23, 1898.

The process for the production of pure santalol is claimed, which consists in the saponification of sandal-wood oil by alcoholic alkali, and fractionation of the product by superheated steam or *in vacuo*.—A. C. W.

*New Perfumes [Violet], the Manufacture of.* J. C. Mewburn, London. From L. Durand, Huguenin and Co., Basel, Switzerland, and P. Barbier, Lyons, France. Eng. Pat. 15,210, July 11, 1898.

The aldehydes of the formula, $C_{10}H_{16}O$, such as lippial from the essential oil of *lippia citriodora* and citral, condense with unsaturated ketones and in particular with mesityl oxide, to produce an aliphatic ketone, $C_{16}H_{24}O$. This boils at 185° C. under 10 mm. pressure, and has no distinctive odour. The process claimed is for the production of this ketone by the condensing action of alkalis, and its transformation into an isomeric tetrahydro-benzene derivative boiling at 162° C. under 10 mm. pressure, by the action of "acid condensing agents," such as sulphuric acid of 60—70 per cent. strength. This cyclic ketone is a golden yellow oil with a delicate and penetrating odour of the iris and violet. The two ketones are claimed as new products.

—A. C. W.

## XXI.—PHOTOGRAPHY.

*Sodium Thiosulphate [Photographic Fixing] Solutions, Decomposition of, on Exposure to air.* P. Michaelis, Phot. Rundsch, 1898, **12**, 221; through Chem. Zeit. Rep. 1898, 192.

The ordinary photographic fixing-bath decomposes somewhat on keeping, free sulphur being deposited and sodium sulphate formed. After making proper allowance for the water evaporated, and titrating the liquid with iodine, a 10 per cent. thiosulphate solution exposed in a basin in a cool room was found to lose during 1, 2, 3, 4, 5, 9, 13, and 21 days 0·74, 1·98, 3·72, 4·46, 5·2, 6·2, 7·44, and 9·92 per cent. respectively of its active ingredient; and although this deterioration takes place rather more rapidly at the outset, seeing that in practice some of the water is lost also, the diminution in strength during reasonable periods of exposure is of very little importance in actual photography.

—F. H. L.

*Alkyl Radicles, Substitution of, in Developers.* Lumière and Seyewetz. Bull. Soc. franc. de phot. 1898, **8**, 158.

At least two free hydroxyl-groups in the ortho- or para-position must remain in a developer if its developing power is not to be destroyed. Any number of alkyl groups may be introduced into the diamine-group which determines the development, without affecting the developing power. The loss of developing power in amidophenolene always occurs when substitution takes place in the pseudo-grouping, provided that there is no other hydroxyl group remaining in the molecule in the ortho- or para-position relatively to that which generates the amido-group.—J. T. D.

### PATENT.

*Lithographic Stone or Metal, Process for producing by either a Gelatin, Intaglio, or Surface Printing, Mezzo-tints, or Chromo-Mezzo Tints on, by Means of Transfer.* R. J. Sturgess, London. Eng. Pat., 23,034 Oct. 7, 1897.

I. To produce a plate for transfers, a sensitised gelatin supported by either glass or metal, is exposed under a negative, and treated as collotype plates to produce a printing surface. It is then treated with an aqueous solution of sodium thiosulphate and sodium chloride, and is washed till grain appears, when, after dipping in alcohol, it is dried and etched with equal parts of water and glycerin, to which calcium chloride may be added.

II. To produce a plate "from which transfers can be obtained by surface printing, giving a grained positive from which negatives can be taken," a plate is prepared as for collotype by a prescribed formula, and after drying, is exposed under a negative until the shadows are solid. After prolonged washing and drying, it is etched with water and glycerin, dried by a damp cloth, and after heating to 68° is dusted with the finest graphite.

III. To produce "an intaglio in either grain or line by means of photography from any photograph, print, or drawing for transferring to stone or metal for surface printing," a copper plate is dusted with bitumen, and the grain thus obtained is melted on to the plate, on which a carbon transparency is then squeezed, and developed in hot water. When dry, it is etched with a strong solution of ferric chloride, until all except the highest lights are touched, and the plate is then finished for pulling transfers.

—E. S.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Glycerin, Nitration of.* R. Auzenat. Monit. Scient. **12**, Sept. 1898, 635.

From experiments made with nitroglycerins prepared with nitric acid containing various amounts of $N_2O_4$, the author finds that although the use of an acid containing more than 2 per cent. is prohibited, it is possible to obtain a nitroglycerin with an acid containing 3 per cent. of $N_2O_4$, which will satisfy the English legal "heat" test in not coloring the sensitive paper in less than 15 minutes, (this Journal, 1897, 283). When acids which contain more than 2 per cent. are employed, however, decomposition occurs frequently during nitration. Washing with sodium carbonate solution at high temperatures serves only to increase the stability of the product. The "heat" test does not indicate the presence of free sulphuric acid but is very sensitive towards nitric acid, a content of over 0·003 per cent. being sufficient to condemn the sample. The presence of acidity due to sulphuric acid cannot be necessarily ascribed to defective washing of the nitro-glycerin since a mixture of sulphuric and nitric acids is employed in the manufacture, but when an impure absorbent has been used, a free non-volatile acid may be introduced. Hence dynamite should be first tested with litmus and if neutral or alkaline, then the presence of unstable nitric esters is alone to be feared, and in this case the test is sufficiently sensitive.—E. W. W.

*Oxymercarbides.* K. A. Hofmann. Ber. **31**, 1904–1909.

Ten grms. of sodium are dissolved in 150 grms. of alcohol, and 40 grms. of precipitated mercuric oxide boiled with the solution for 16 hours. The spirit is then evaporated, and the residue extracted with water, when a greyish yellow powder of "Oxyhexamercarbide" remains. This substance forms a dense citron-yellow powder, sensitive to light, having the formula $C_2Hg_6O_4H_2$. It is a basic substance, readily forming with nitric or sulphuric acid the salt, $C_2Hg_6O_4H_4(NO_3)_2$ or $C_2Hg_6O_4H_4(SO_4H)_2$, and converted by hydrochloric acid into $C_2Hg_6Cl_6$. From any of these substances the base is regenerated by treatment with sodium hydroxide solution. At a temperature of 200 C. the base darkens in colour, and at about 230° C. explodes violently. The salts are not at all explosive, the base itself is not affected by shock or blow, and as on heating, water seems to be given off before explosion, the explosive substance appears to be $C_2Hg_6O_3$.—J. T. D.

*Cordite, Manufacture of.* E. W. Anderson. Proc. Inst. Civil Eng. 1898, **132**, Part ii., 1—65.

The constituents of cordite are nitroglycerin, 58 parts; guncotton, 37 parts; mineral jelly, 5 parts.

Guncotton in the form of finely divided pulp, is mixed by hand with the requisite quantity of nitroglycerin, the result being a dry-looking mass termed "paste" which can be safely transported from place to place.

The next process is to place the "paste" in a Pfleiderer mixing or kneading machine, and after the proper amount of acetone has been poured in, the cover is placed on to prevent evaporation of the acetone as much as possible, and the machine is started. The mixers now used generally hold 150 lb. of explosive, and 20·83 parts of acetone are required for every hundred parts of cordite.

After the incorporation has gone on for 3½ hours, the mineral jelly is added and the kneading continued for 3½ hours more. The material has now a consistency about equal to that of stiff dough, and is ready for conversion into the form of cord, and this is done by forcing it through a die of the proper size and shape.

A cylinder of suitable size fitted with a die at one end, and a close-fitting plunger, capable of being forced into it, is filled with the dough. On forcing in the plunger, the explosive emerges from the die in the form of a cord.

A strainer is inserted in the cylinder over the die so as to prevent any foreign matter which had accidentally got into the dough entering the die and obstructing it.

Details and drawings are given of the mechanism employed in operating the press.

As the cordite issues from the press, it is wound on to reels in the case of the small diameters; when of larger size, it is received on to a travelling band provided with transverse knives at suitable intervals, and on passing beneath a roller, the cordite is pressed down on to the knives and is thus cut into the required lengths.

The cordite now, either on reels or in lengths which are placed on trays, has to be dried, *i.e.*, the remainder of the acetone has to be driven off, and for this purpose is placed in the drying-house, where a temperature of 100° F. is maintained, for 3½ to 15 days depending on the size of the cord.

The cordite manufacture is now complete, but in order to secure the greatest uniformity in the result, the important process of blending has to be carried out in which a systematic mixing of different batches is made, so that an average quality may be produced. In the case of the cordite of large diameter the sticks from the different batches are selected by hand, but for rifle cordite the threads from 10 reels are formed into an untwisted rope and wound on to another reel, and then the threads from six such reels are united, and wound on to another so-called 60-strand transport reel. This 60-strand rope is then passed into a cartridge-filling machine, which feeds a certain length of the rope into an empty cartridge case and then cuts it off.
—W. M.

*French Match Works.* Report to the Home Office by T. Oliver. Eyre and Spottiswoode, 1898.

Dr. Oliver visited the works at Aubervilliers, Pantin, and Prado, Marseilles. At the first, which is the oldest, 105 women and 95 men are employed in manufacturing the ordinary matches containing white phosphorus. The paste is mixed in covered iron vessels, fitted with exhaust pipes, which prevent the escape of all fume. The matches are tipped by means of machine rollers fed mechanically with the paste, and provided with aspirating hoods, and are dried in thoroughly ventilated air-tight chambers. The boxes are filled by women in a separate room, and here the odour of phosphorus was strong, even at the other end where the labelling and packing were being carried on. The separation of these departments into different rooms is recommended by the author. At Pantin, the manufacture of (1) ordinary matches, (2) of tison, a kind of safety vesuvian which only strikes upon paper prepared with red phosphorus, and (3) of safety matches, finds employment for 400 women and 200 men, and though the methods are practically the same as those at Aubervilliers, the works are more modern and better kept. In the factory, Prado, Marseilles, 460 women and 80 men are employed, and about five millions of wax vestas and 20 millions of ordinary matches are made daily. No safety matches are made. The processes of mixing, tipping, and drying are carried on in a building detached from the rest, and by men only. "The tipping of the vestas is done by one man on a marble slab," provided at the front and one side with aspirating arrangements, which, however, were not in operation at the time of the visit, and consequently the odour of phosphorus was strong. The operator had worked 36 years at the process, and only been once away ill from some minor dental affection. Although the paste contains red oxide of lead, no symptoms of lead poisoning are said to have occurred in the workpeople. The boxing is done by women in well-ventilated rooms, on benches provided with aspirating gratings. In the boxing department for ordinary matches, where the arrangements are similar, there was considerable fume, due to occasional conflagrations. The dining-room is a separate block, with an outside covered passage, in which are hung on the wall large bottles filled with turpentine gargle. A well-appointed wardrobe for the women is provided, and each worker is supplied with a tin mug for drinking and gargling purposes. About 22—23 workpeople are away through illness on the average each month. According to M. Desbrosses, women are much more easily fatigued and more susceptible to poisonous fumes than men. Three cases of necrosis were examined; all had lost teeth and a portion of the jaw-bone, but two were allowed to work in the splint department. The filling of the boxes when done by hand is more deleterious to health than any of the other processes, and employs more hands, who are mostly women. The following are a few of the instructions which are printed and hung about the factories. Before taking food, which is only allowed in the dining-room, the working clothes must be left in the cloak-room, the hands washed with soft soap and water, and the mouth gargled with the gargle provided; the same precautions are to be observed on leaving work. The teeth are examined on prescribed days, and the hygienic regulations read out aloud once every month.

Applicants for employment must not be under 16 nor over 32 years of age for men and 28 for women, must show a certificate of birth and one of good moral character. They are examined by a doctor and required to be free from bodily infirmity, contagious disease, to have been vaccinated or had small pox, and to have teeth in good order. The hours are from 6.30 to 11.30 a.m. and 1 to 6 p.m. and can be lengthened if necessary. In case of illness a doctor's certificate is necessary before returning to work. At Aubervilliers and Pantin the workpeople are medically examined once in every six months, and those with carious teeth are suspended or relegated to the splint department. In case of symptoms of poisoning without necrosis, such as anæmia, malnutrition, loss of flesh, and albuminuria, 1 litre of milk may be ordered daily, but for the last 18 months this last measure has not been required, and whereas in August 1897, 15 men and 35 women were found to be suffering from simple dental caries, in February 1898 there were only 4 cases of men and 12 of women. The Commission appointed by the Academy of Medicine reports that since these regulations have been in vogue, *i.e.*, from December 1st, 1896, to December 1st, 1897, there has not been a single fresh case of necrosis nor of general illness attributable to phosphorus poisoning in a population of 575.

According to Dr. Arnaud of Marseilles, phosphorus poisoning arises from absorption of the phosphorus vapour by the respiratory organs and probably too by the skin, and elimination by the various emunctories after a more or less lengthened sojourn in the organism. Those who are subject to glandular enlargement, or are ill-developed, scrofulous, of weak constitution, and tending to tubercular disease, rickety, suffering from cough, heart disease, indigestion or albuminuria, and anæmics and alcoholics are specially liable to the disease. Dr. Arnaud knows of several people, however, who have been exposed to phosphorus for 10, 20, and 30 years, who have been free from the disease and kept the appearance of good health. From 1884 to 1896 there were 4 cases of confirmed necrosis, one individual died, 13 of incipient necrosis and 57 cases (1890–95) of inflamed gums, dental abscesses, penetrating dental caries, and fistulous openings.

The wooden matches contain 11, and the vestas 5·7 per cent. of phosphorus. The latter are tipped cold, and there is less odour of phosphorus in the boxing and a more general appearance of health among the hands.

The French Government since March 1898 have succeeded in making a match free from white phosphorus and capable of striking anywhere. It is said to contain a modified form of red phosphorus, but as yet the unreliability of the chemical composition and the difficulty of making the paste in large quantities has prevented its becoming a general industry.—E. W. W.

### PATENTS.

*Blasting, Impts. relating to.* Kynoch, Ltd., and A. T. Cocking, Witton. Eng. Pat. 19,650, Aug. 25, 1897.

Wads, impregnated with ammonium chloride, are placed in the shot-hole above and below the cartridge with the object of preventing the ignition of any explosive gas which may be present.—W. M.

*Explosive Compounds, Impts. in the Manufacture of.* G. Beneke, Southport. Eng. Pat. 23,340, Oct. 11, 1897.

The inventor finds that a good explosive of the kind described in his previous patent, 28,220, 1896 (this Journal, 1898, 272) may be made without chromate or bichromate of sodium or potassium, and he now manufactures his explosive by first incorporating carbonate or bicarbonate of soda with resin in the melted state, and then incorporating the mixture in a dry and ground state with ammonium nitrate.—W. M.

*Waterproofing of Blasting Cartridges and new Waterproofing Compounds suitable therefore, Impts. in the.* G. Beneke, Southport. Eng. Pat. 24,385. Oct. 21, 1897.

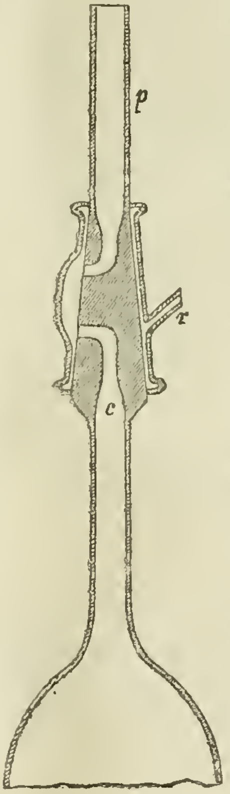
Carbonate, or bicarbonate of sodium, or other "flame reducing salt" is added to any of the waterproofing mixtures hitherto used.—W. M.

# XXIII.—ANALYTICAL CHEMISTRY.

## APPARATUS, ETC.

*Mineral Oil, Use of Acetylene in Flashing-point Tests.* P. Wolff. Chem. Rev. Fett- u. Harz-Ind. 5, [7], 139.

The difficulty in the way of obtaining a constant flame for flashing-point tests in open-cup apparatus, in works not supplied with gas, may be overcome by the aid of acetylene. A pure gas and uniform pressure are, however, essential to success—conditions which appear to be satisfactorily fulfilled in the apparatus supplied by several eminent firms in Berlin. The apparatus should, naturally, be capable of furnishing both the test- and heating-flames of the Pensky-Martens' tester.—C. S.



*Pipette with Stop-Cock.* C. Sander. Zeits. Anal. Chemie, 1898, 37, [8], 497.

The construction of the pipette is clear from the illustration. When in use, a rubber tube carrying a pinch-cock is attached to the upper end, and the liquid is sucked up to the point *p*. When the glass collar is turned round till the tube *r* is opposite the mouth of the passage *c*, the liquid at once flows out, the pipette being of such capacity as to deliver the correct amount of liquid in this way, and the excess of liquid remains in the upper part *p*.—H. B.

*Water Samples, Rapid Alteration of, on Preservation in Zinc Vessels.* F. Hundeshagen. Zeits. für öffentl. Chem. 4, 493; Chem. Centr. 1898, 2, [9], 582.

If water samples, destined for analysis, are preserved in zinc vessels, the zinc is very easily oxidised by the simultaneous action of the oxygen of the air absorbed by the water, and by the free and "half-combined" carbonic acid. The zinc oxide formed is gradually converted first into bicarbonate, and then into monocarbonate, which separates out, thus considerably reducing the carbonate-hardness of the water.—A. S.

### PATENT.

*Acidity of Milk and other Liquids, Impts. in Apparatus for Testing the.* A. W. Stokes, London, and R. A. Lister and Co., Dursley, Gloucester. Eng. Pat. 22,027, Sept. 25, 1897.

According to this process, the milk to be tested is placed in a suitably graduated cylinder, and to it is added drop by drop a solution of caustic soda tinted with phenolphthaleïn, until a permanent pink colour is obtained. The amount of soda solution added is then read off, and the amount of acidity in the milk directly ascertained, as each division of the graduated scale corresponds to 0·01 per cent. of acidity. —W. P. S.

## *INORGANIC CHEMISTRY.—QUALITATIVE.*

*Sodium Bicarbonate, Test for Monocarbonate in.* M. Kubli. Chem. Zeit. 1898, **22**, 228. (From Arch. Pharm. 1898, **236**, 321.)

An aqueous solution of quinine hydrochloride of a certain concentration is not precipitated by a solution of sodium bicarbonate which contains less than 2 per cent. of monocarbonate. To apply the test 0·4 grm. of quinine hydrochloride is dissolved in 400 c.c. of water: and 3 grms. of sodium bicarbonate in 50 c.c. of water. In making up the latter solution, water at 5° to 6° is used, and the salt brought into solution in a closed vessel without shaking. On mixing equal volumes of the two liquids a clear mixture should result.—T. E.

*Metallic Fluorides and Oxyfluorides, Some Reactions of.* E. Paterno and U. Alvisi. Atti. R. Accad. dei Lincei Roma, **7**, [1], 327—332 and [2], 15—19; Chem. Centr. 1898, **2**, [7], 168.

Metallic oxalates which are slightly soluble in water are less soluble in water containing hydrofluoric acid; thus, from hydrofluoric acid solutions of the fluorides of copper, manganese, and mercury, oxalic acid solution precipitates the salts, copper oxalate, $2CuC_2O_4.H_2O$; manganese oxalate, $MnC_2O_4H_2O$; and mercury oxalate, $HgC_2O_4$, respectively. If dry powdered fluorides, such as fluorspar, magnesium fluoride and the fluorides of the rare earths, are warmed on the water bath with saturated oxalic acid solution, hydrofluoric acid is set free. Sulphurous acid precipitates the compound $Cu_2SO_3CuSO_4.2H_2O$ from copper fluoride solutions. Silver fluoride is precipitated from its hydrofluoric acid solution by arseniates, chromates and nitrites. If the fluorine or oxy-fluorine salts of potassium be treated in weak hydrofluoric acid solution with an equivalent amount of oxalic acid, hydrofluoric acid or "fluor-oxy" acid is set free and potassium fluor-tetroxalate, $KFC_2O_4.H_2C_2O_4.2H_2O(KF.C_2O_4.H_2C_2O_42H_2O\,?)$ formed. This reaction also takes place with hydrofluoride, fluosilicate, fluoroxyuranate, fluoroxy-tungstate, and fluoroxymolybdate of potassium and with the fluorides of the rare earths. With cryolite, the aluminium fluoride is first decomposed and then the sodium fluoride. Oxalic acid also sets free hydrofluoric acid from the triplite of "Schlackenwald," but not from mica containing fluorine. Tartaric acid acts in a similar manner to oxalic acid in all the above reactions, but more slowly.—A. S.

*Bismuth, A Flame Reaction for.* G. L. Heath. Chem. Zeit. 1898, **22**, 229. (From Gaz. chim. ital. 1898, **28**, 393.)

When a small quantity of bismuth, or of a bismuth compound moistened with bromine, is brought on a platinum wire into the lower part of the flame of a Bunsen burner, it imparts a beautiful green coloration to the flame, similar to that produced by copper, boron, or thallium compounds. Concentrated hydrochloric acid may be used instead of bromine. T. E.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Sulphur and Iodine, Estimation of, in Special Cases.* A. Longi and L. Bonavia. Gaz. chim. ital. **28**, [1], 336—341; Chem. Centr. 1898, **2**, [4], 310.

The following methods are proposed for the estimation of sulphur (in the form of sulphites, thiosulphates, trithionates, tetrathionates, sulphides, and polysulphides), together with iodine (in the form of iodides and iodates):—

*First Method.* *a.* Neutralise with potassium carbonate, oxidise with excess of permanganate, decolorise with alcohol, acidify with acetic acid, and filter. In an aliquot part of the filtrate, determine the sulphuric acid with barium chloride. Treat another part of the filtrate with potassium iodide and hydrochloric acid, and titrate with thiosulphate the iodine separated from the iodate. *b.* Treat the oxidised filtered liquid at 50—60° C. with pure nitric acid, precipitate the sulphuric acid with barium nitrate, and determine the iodate in the filtrate in the same manner as above.

*Second Method.*—Oxidise with sodium peroxide, thus converting all the sulphur into sulphate, decompose the excess of peroxide, and treat with zinc dust, which reduces the iodate to iodide. Now either acidify with acetic acid, precipitate the sulphuric acid with barium acetate, and determine the iodine in the filtrate by means of nitric acid and silver nitrate, or precipitate first with nitric acid and silver nitrate, and treat the filtrate with barium nitrate.

—A. S.

*Perchlorate in Chili Saltpetre, Estimation of.* C. Ahrens and P. Hett. Zeits. für öffentl. Chem. **4**, 415—418; Chem. Centr. 1898, **2**, [8], 558.

The following method is stated to be sufficiently rapid and exact for technical purposes:—20 grms. of the suitably-pulverised sifted sample are moistened in a platinum dish with 2—3 c.c. of cold saturated soda solution, then about 1 grm. of manganese dioxide free from chlorine added, and the mixture evaporated to dryness. The mass is melted, and the covered dish heated to redness for 15 minutes. The cooled melt is dissolved in about 100 c.c. of hot water, the solution cooled and made up to 250 c.c. 50 c.c. (= 4 grms. of saltpetre) of the clear filtered solution are acidified with 10—15 c.c. of nitric acid (sp. gr. 1·2), and a 1 per cent. solution of permanganate added, with stirring, until the red colour lasts one minute. Iron alum is then added, and the solution titrated by Volhard's method. The chlorine is also determined in 4 grms. of the original sample by Volhard's method, and from the difference in the number of c.c. of $\frac{1}{10}$N silver solution used before and after fusion, the perchlorate is calculated as $KClO_4$. The addition of soda before fusing hinders the volatilisation of hydrochloric acid; that of manganese peroxide should facilitate the decomposition of the perchlorate; whilst the treatment with permanganate removes the nitrous acid, and converts the iodide present in the melt (formed from the small amount of potassium iodate present in ordinary Chili saltpetre) into iodate. The authors give results obtained with mixtures of pure $NaNO_3$ admixed with varying quantities of NaCl, $KClO_4$, and $KIO_3$. (See also this Journal, 1898, 795.)

—A. S.

*Calcium, A Rapid Method of Determining.* M. Passon. Zeits. angew. Chem. 1898, [31], 776—777.

The substance is dissolved in boiling dilute *aqua regia*, the solution made up to 500 c.c., and an aliquot portion transferred to a beaker. After the addition of phenolphthalein, dilute ammonia (1 part of the ordinary solution with 2 of water) is added until there is a perceptible pink colour. A 10 per cent. solution of citric acid is then added until the pink colour disappears, and the precipitate is redissolved, after which an additional 10 c.c. of the citric acid solution are added. The contents of the beaker are made up to 200 c.c., and the calcium precipitated directly from the boiling solution by means of ammonium oxalate in excess, without having regard to the presence of iron, alumina, magnesia, or phosphoric acid. The precipitate, which is crystalline, rapidly subsides and is readily filtered, is collected, washed, dried on the filter, ignited, and weighed as calcium oxide.

The long table of the examples given in the paper shows that the results obtained by this method are in very close agreement with those of the older and more tedious method, in which the iron, &c. are removed before the precipitation of the calcium.—C. A. M.

*Dithionic Acid from Other Sulphur Acids, Separation of.* A. Longi and L. Bonavia. Gaz. chim. ital. **28**, [1], 341—341; Chem. Centr. 1898, **2**, [4], 310.

The method depends on the fact that all lower oxidation products of sulphur, with the exception of dithionic acid, are oxidised in alkaline solution to sulphuric acid by means of permanganate (or potassium hypobromite). After this treatment, the sulphuric acid is precipitated from the solution by a small excess of barium chloride, and the filtrate, after addition of aqua regia or potassium chlorate and hydrochloric acid to oxidise the dithionic acid, is treated with barium chloride and allowed to stand several hours, whereby all the sulphur of the dithionic acid is precipitated as barium sulphate.—A. S.

*Phosphoric Acid in Phosphates, A New Solvent for.* W. Hoffmeister. Landw. Versuchsstat. 1898, **50**, 363.

The author rejects citric acid as a solvent for the phosphoric acid of bone-meal, and indeed of all phosphates save Thomas slag. He proposes as a substitute an ammoniacal solution of humic acid, in which the finely-divided phosphate is suspended and kept well stirred, while a current of carbon dioxide is passed through. Into a two-litre flask containing a little sand, is run a solution of 15 grms. of humic acid in dilute ammonia, 5 grms. of the phosphate to be examined added, and at least a litre of water. A stream of carbon dioxide is passed through for 12 days, ammonia being added occasionally. The liquid is then run into another flask, the sand washed, and the washings added to the original liquid, the whole being made up to a known volume, and filtered. Four-fifths of the whole is evaporated to dryness on the water-bath with a little hydrochloric acid, the residue taken up with water and hydrochloric acid, filtered and washed, the filtrate being made up to 100 c.c. In fractions of this the phosphoric acid, iron, and calcium are determined, while any silica is determined in the residue.—J. T. D.

*Lead in Lead Ores, Determination of.* L. Schneider. Oesterr. Zeits. Berg u. Hütt. **46**, 431—434; Chem. Centr. 1898, **2**, [8], 559.

The author has investigated the sources of error in the different methods for the estimation of lead, and he shows in five tables, the influence of the different reagents used. Nitric acid, and in a smaller degree, tartaric acid, dissociate lead sulphate, but if the amount of nitric acid do not exceed one per cent., precipitation can be effected by sulphuric acid and ammonium sulphate as accurately as in a solution free from nitric acid. The following method is recommended:—1 grm. of the ore is boiled in a covered beaker with concentrated HCl for 10—15 mins., and then after addition of 5 c.c. of concentrated $HNO_3$, heated till brown fumes cease to be evolved. 25 c.c. of a tartaric acid solution containing 10 grms. of tartaric acid are then added, the solution warmed for a short time, cooled, 25 c.c. of strong ammonia added and the whole again warmed for a short time. With pure ores, the residue consists of quartz or $BaSO_4$, and washing the filter thrice with weak ammonia suffices to completely free it from lead. The filtrate is made up to 300 c.c., 50 c.c. of $H_2SO_4$ (one of acid to one of water) added, and the whole heated till the precipitate settles to the bottom. After cooling, the precipitate is separated by decantation or filtration, washed with 1 per cent. $H_2SO_4$, dried, ignited, and weighed. The precipitation of the lead by $H_2SO_4$ is incomplete, as is always the case; the filtrate, about 500 c.c., containing, according to comparative tests, 0·003 grm. of $PbSO_4$. Under the conditions described above, therefore, the error due to the slight solubility of lead sulphate, is eliminated by a correction of 3 mgrms.—A. S.

*Telluride Ores [Gold], Assay of.* C. H. Fulton. School of Mines Quarterly, **19**, 419—426.

Owing to the growing importance of telluride gold ores and the difficulty of their assay as compared with ordinary ores, the author has conducted a series of assays under varying conditions and has arrived at the following conclusions. A large excess of litharge is necessary to obtain good results; in the case of rich tellurides the quantity of ore being decreased rather than the litharge increased so that the lead button may not be too large to cupel directly (20 to 28 grms.). The crucible assay with direct cupellation is by far the best. The fire should be moderately hot and the length of time in the furnace should be from 40 to 50 minutes. The cupellation should be at a low heat with litharge crystals forming on the cupel, whilst for accurate assays the crucible slag should be remelted and the button cupelled, since the loss of gold in the slag is much greater than in the case of ordinary ores. The loss of gold in cupellation which is recoverable is of small account.

Scorification of any kind is bad for telluride ores, whether direct on the ore or to reduce large or brittle buttons. The gold lost in the slag when scorifying the ore direct is very great and there is also loss by volatilisation, the proportions increasing with the richness of the telluride. These results agree with those of F. C. Smith, who found that the loss of gold was very great if the charge was not properly made up as regards the amount of litharge.—A. W.

*Platinum, Precipitation of, from its Solutions.* A. Atterberg. Chem. Zeit. 1898, **22**, 538: Analyst, 1898, **23**, [270], 247.

The author states that thiacetic acid, mercury, and magnesium are the most useful of the numerous substances which may be employed to precipitate metallic platinum from its solutions. The first named is recommended when only traces of the metal are present, as it yields a particularly voluminous precipitate. Mercury is the best reagent for the determination of platinum in the alcoholic filtrate from a potash determination. The platinum solution is concentrated to a small volume, excess of mercury added, and the mixture stirred until the reaction is complete. The by-product is mercuric (not mercurous) chloride, and can easily be removed by heat. The reduction only takes place in the warm; the platinum is deposited as a compact powder, but the operation must never be carried out in a platinum vessel, as the metal adheres firmly to the sides. If the platinum which is in solution has to be weighed, the best precipitant is magnesium ribbon. The solution should be neutral, or, preferably, acidified with a little hydrochloric acid. The precipitation proceeds slowly in the cold, more quickly on warming or on agitation, but a trace of the metal remains in solution for some time giving it a grayish colour, so that the liquid should not be filtered until it is clear and colourless. The platinum is heated with hydrochloric acid, washed very thoroughly, and gently ignited. The use of magnesium ribbon is also preferable in working up alcoholic platinum residues. The metal is completely precipitated in 24 hours, and can be decanted off, purified with acid, and washed.—A. S.

*Iron and Manganese in Ores, Determination of.* P. Lehnkering. Zeits. für öffentl. Chem. **4**, 459—464; Chem. Centr. 1898, **2**, [7], 507.

In the analysis of ores that contain among other impurities, organic matter, insoluble sulphur compounds, insoluble oxides and silicates, and titanium, arsenic and copper compounds, difficulties may arise in the determination of iron by the usual shortened methods. The three most common methods of titration—with $SnCl_2$, and with $KMnO_4$ in the $H_2SO_4$ solution after reduction with zinc, and in the solution prepared according to Reinhardt—only give good results if all the iron present in the ore occur in a suitable form in the solution, and if no other bodies than iron influence the titrating solution. The total iron may be brought into solution by digesting the ore with hydrochloric acid (sp. gr. 1·19)—with the addition of a concentrated solution of $SnCl_2$ for the stannous chloride method—fusing the insoluble residue with potassium and sodium carbonate, and adding the hydrochloric acid solution of the melt to the main solution. It is still essential, however, that the following points be noted. Solutions containing arsenic may only be titrated after the removal of the arsenic; solutions containing copper, without removing the latter, only by the zinc method; solutions containing titanium, never by the zinc method. Nitro compounds formed by the use of nitre for the oxidation of pyrites by fusion of the residue, influence all three methods: organic matter, the zinc and Reinhardt's method. The author proposes the following method:—About 5 grms. of the very finely divided ore is dried for one hour at 100° C., and the organic matter destroyed by heating to redness in a porcelain crucible. The contents of the crucible are digested on the water bath for four hours with 60 c.c. of hydrochloric acid (sp. gr. 1·19) in a 300 c.c. Erlenmeyer flask, the whole evaporated to dryness, taken up with hydrochloric acid, diluted with 100 c.c. of hot water, filtered, and the residue washed with hydrochloric acid and hot water. The residue, after incineration of the filter, is fused in a platinum crucible with 5 grms. of the mixed carbonates of potassium and sodium together with some potassium nitrate. The cooled melt is dissolved in hydrochloric acid, the solution evaporated to dryness with the addition of some sulphuric acid, the residue taken up with dilute hydrochloric acid, filtered, and the iron precipitated with ammonia. The precipitate, after washing well with hot water, is dissolved on the filter in hot dilute hydrochloric acid, and the solution added to the main solution of the ore. From the combined solutions any arsenic and copper is precipitated with sulphuretted hydrogen, the latter removed from the filtrate by boiling, and the liquid oxidised with potassium chlorate. The solution freed from excess of chlorine and cooled to 15° C., is made up to 500 c.c. and the iron estimated volumetrically, but not by the zinc method.

The author considers that the determination of manganese in the volumetric way, especially by Volhard-Wolff's and Hampe's methods, is not free from objections, and he therefore recommends the gravimetric estimation as sulphide. In this case also, the treatment of the insoluble residue is absolutely necessary. The manganese ore may be sufficiently dried at 120° C. In the determination of the silica, titanic acid must be taken into account as well as the insoluble sulphate, and the author therefore proposes the following method:—1 grm. of the dry ore is digested with 15 c.c. of hydrochloric acid (sp. gr. 1·19), the whole evaporated to dryness with the addition of sulphuric acid, the residue heated to 150° C. for half an hour, then taken up with dilute hydrochloric acid, filtered off, and ignited. The ignited residue is fused with 3 grms. of potassium bisulphate, and the titanium compounds removed from the cooled melt by means of cold water. The residue remaining on the filter after washing with cold water and hot dilute hydrochloric acid, consists of silica with insoluble sulphate, and after ignition, is treated in a platinum crucible with hydrofluoric and sulphuric acids.—A. S.

*Cobalt, The Oxidation of Salts of, in Alkaline Liquids, and Method of Determining in presence of Nickel and Iron.* A. Job. Comptes Rend. 1898, **127**, [2], 100—103.

The reaction first noted by Field (J. Chem. Soc. 1862) in which on adding hydrogen peroxide or bromine water to a solution containing potassium bicarbonate and a cobalt salt, a green coloration is obtained, is extremely sensitive, and is suitable for the detection of as little as 1 mgrms. of cobalt in a litre. Many unsuccessful attempts have been made to isolate this green compound. If the solution is evaporated at a low temperature after saturation with cobalt, only a brown powder of oxide is obtained. Durrant (Proc. Chem. Soc., April 1896) came to the conclusion that the cobalt is present in the solution in the cobaltic state, an opinion based on the analysis of the green gelatinous unstable precipitate obtained by adding to the green solution an excess of a cobalt salt mixed with an excess of hydrogen peroxide. MacConnell and Hanes (J. Chem. Soc., May 1897) determined the quantity of carbon dioxide liberated during the reaction, and arrived at the conclusion, which the author considers as unreliable as that of Durrant, that the cobalt is present in combination with the potassium as $CoO_4K_2$. By means of a ferrous reagent (ferrous pyrophosphate) described in a former communication (no reference given), the author considers that he has solved this problem. On adding the green solution to the ferrous reagent, the former is instantly reduced and becomes rose coloured. By producing the green solution from known quantities of cobalt, adding a known amount of the ferrous reagent, and determining the excess of the latter with iodine, the amount of oxygen taken up in the oxidation is said to be in accordance with the equation—

$$2CoO + O = Co_2O_3.$$

The only difficulty is to be certain of the complete oxidation of the cobalt, but it was found that by adding a large excess of hydrogen peroxide to the alkaline solution, and leaving it in the cold, it continues to lose oxygen for several days, and that when the composition becomes constant, the amount of oxygen still remaining corresponds to the formula $Co_2O_3$. The same result is arrived at more rapidly by adding a neutral salt of cobalt to a mixture of hydrogen peroxide and sodium bicarbonate, the excess of oxygen being then given off in a few minutes. It is possible to boil the solution for several minutes without causing its composition to vary, but prolonged boiling causes the decolorisation of the liquid and a deposit of sesquioxide. It is noted that the cobalt thus brought into the cobaltic state is ten times more soluble than in the cobaltous state. The green solution is decomposed by soda even in very dilute solution, a fact which appears to lend some support to the hypothesis that an acid of cobalt is present.

The author has also determined the amount of carbon dioxide liberated in the reaction and finds that each atom of cobalt oxidised corresponds to one molecule of liberated carbon dioxide. He, therefore, concludes that the formula given by MacConnell and Hanes, which would require 4 molecules, is incorrect. Taking into account the readiness with which cobalt forms hydrocarbonates he considers that the results can be readily interpreted by the reaction expressed in the equation—

$$2[(CO_3H)_2Co] + H_2O_2 = Co_2(OH)_4.(CO_3H)_2 + 2CO_2.$$

The method here employed for determining the oxygen taken up by means of the ferrous reagent gives a rapid process for determining cobalt in the presence of nickel and iron, for the former is not oxidised by the hydrogen peroxide, and the latter is not reduced by the ferrous pyrophosphate. The liquid containing the cobalt is added to a solution of potassium bicarbonate containing an excess of hydrogen peroxide, the liquid warmed for a few minutes, the oxygen taken up determined by means of the ferrous reagent and iodine, and the amount of cobalt calculated from the result. It is stated that the results thus obtained are as concordant as those given by electrolysis.—C. A. M.

*Copper, Estimation of Oxygen in.* M. Lucas. Bull. Soc. Chim. 1898, **19**, [16—17], 795—799.

The author modifies Hampe's method as follows:—The silver nitrate solution is made of 5 per cent. strength from the pure salt, allowed to stand, and decanted into a bottle of red glass. The copper, in sheets of about 0·15 mm. thickness, is carefully cleaned with glass paper, and 1 grm. is subjected to the action of 120 c.c. of silver nitrate solution *in vacuo* for 15 hours without shaking. The residue is collected on an asbestos filter and washed with cold water, and is again treated for 2 hours *in vacuo* to ensure the absence of copper in a soluble form. After again filtering and washing, the precipitate is dissolved in nitric acid, the silver precipitated by hydrochloric acid, and the copper determined by electrolysis. Its weight multiplied by 1·689 gives the weight of cuprous oxide in the sample. The copper may also be colorimetrically estimated, and this is to be preferred for the small amounts generally found. This process, as applied to a commercial sample and to a metal prepared by the addition of 3 per cent. of cuprous oxide to molten copper, gives results which agree among themselves, and with the estimation by reduction in hydrogen by Hampe's method. The process gives a negative result with electrolytic copper prepared according to Riche's method, gives an accurate result when applied to cuprous oxide alone, and the same result whether copper and cuprous oxide are attacked separately or simultaneously. By varying one condition of the experiment at a time, the author has arrived at the following conclusions, as to the conditions necessary for accuracy. The results of Hampe's method increase as the amount of copper employed is diminished; the modified method does not exhibit this irregularity. The silver nitrate solution is reduced by the action of light and becomes acid; this acidity cannot be corrected by the addition of silver, and the addition of caustic soda produces irregularities; the use of 50 per cent. solutions gives irregular results, the action is too rapid. If metal shavings be employed instead of foil, the results are generally the same, but it is impossible to be certain the surface is free from oxide. If the copper be attacked in the presence of air, the results are too high; neutral copper nitrate and mixtures with neutral silver nitrate exposed to the air and filtered, leave no copper on the filter, but if a solution of neutral copper nitrate be allowed to stand with 3 grms. of precipitated silver, on filtering, 0·003 grm. of copper remains after washing. Too low results are obtained in an atmosphere of carbon dioxide or hydrogen. In a current of nitrogen the results are satisfactory, and the reaction is quickened by the agitation. If the solution be filtered through paper instead of asbestos, copper is always retained by the paper and cannot be removed by washing. The basic nitrate may be dissolved by hydrochloric and sulphuric acids or by ammonia; 5 per cent. hydrochloric acid dissolves the least silver; it should be used twice.—A. C. W.

*Red Lead, Assay of.* Baucher. Petit Monit. de la Pharm. **47**, 2651; Pharm. J. 1898, **61**, [1469], 238.

*Foreign Impurities.*—Ten grms. of the sample are treated with 10 per cent. nitric acid until all the monoxide is dissolved, and then invert sugar is added to reduce any dioxide present. The insoluble residue is allowed to settle, the liquid decanted off, and more nitric acid added. The residue is then washed, first by decantation and then on a tared filter, and finally dried at 105° C., and weighed. The insoluble matter should not amount to more than 1·5 per cent.

*Lead Dioxide.*—Ten grms. of the sample are heated on a sand bath for four hours with 500 c.c. of 10 per cent. nitric acid. The dioxide is allowed to settle, washed by decantation, collected on a filter and dried. The amount present should be from 22 to 24 per cent.

*Free Litharge.*—Ten grms. are digested on the water bath with 20 grms. of normal lead acetate and 200 c.c. of water. After settling, the clear liquid is decanted and the residue collected, washed and dried. The loss of weight indicates the amount of free litharge. Theoretically, red lead should contain no litharge, but commercial samples usually contain 56—40 per cent. Red lead containing a large proportion of free litharge attacks zinc with great rapidity in the presence of sea-water, or fresh-water rich in ammoniacal salts.

*Combined Monoxide.*—This is determined by difference from the previously obtained figures. Barium sulphate, which is often used to adulterate red lead, should be tested for in the insoluble residue; and lime and the salts of other metals in the nitric acid solution.—A. S.

*Chrome Yellow, Commercial, Analysis of.* Willenz. Bull. Assoc. Belge des Chim. **12**, [4], 163—167.

The method of analysis proposed by Wittstein (Dingler's polyt. J. **210**, 280) for estimating the adulterants in chrome yellow, contains a source of error in that the digestion with sodium carbonate does not completely decompose the lead chromate. Consequently the residue cannot be correctly assumed to consist solely of barium sulphate; in fact, pure lead chromate will leave as much as 49 per cent. of such a residue.

The author finds that the following method gives satisfactory results, even in presence of the most complex form of adulteration, *viz.*, by lead sulphate, barium sulphate, calcium sulphate and carbonate, and clay.

One grm. of powdered substance is treated with 100 c.c. of dilute (1:20) hydrochloric acid at a gentle heat, the liquid being decanted on to a filter, which, together with the residue, is then washed with hot water. The total *calcium* is estimated in the filtrate, as oxalate, and the sulphuric acid of the *calcium sulphate* is thrown down by barium chloride, the amount of *calcium* present as *carbonate* being taken by difference. The residue is digested at the ordinary temperature with 50 c.c. of (neutral or slightly alkaline) ammonium acetate (sp. gr. 1·04) to dissolve the *lead sulphate*, which is then estimated by evaporating the filtered solution to dryness in presence of an excess of sulphuric acid.

The residual substance is then mixed with 50 c.c. of water and boiled for 10 minutes with 25 c.c. of potassium hydroxide solution (112 grms. of KHO per litre) to convert the *lead chromate* into soluble potassium plumbite, leaving *barium sulphate* and *clay* behind to be separated by the ordinary methods.

The *chromic acid* is determined in a separate sample, by the Bunsen iodometric method.—C. S.

*Nitric Acid [in Waters], A New Method of Determining.* E. Bohlig. Zeits. Anal. Chem. 1898, **37**, [8], 498—501.

The method is based on the double decomposition of $HNO_3$ and HCl into chlorine, &c., and the determination of the chlorine by titration with $K_4FeCy_6$, according to the following equations:—

$$2HNO_3 + 6HCl = 4H_2O + 3Cl_2 + 2NO.$$

$$2(K_4FeCy_6 + 3H_2O) + Cl_2 = 2KCl + K_6Fe_2Cy_{12} + 6H_2O.$$

In testing water, for example, 100 c.c. are evaporated to dryness in an Erlenmeyer flask placed on a hot plate. After cooling, a few c.c. of pure concentrated $H_2SO_4$ are poured down the inside of the flask, and to prevent loss of Cl, a rubber stopper, fitted with two glass tubes and a second stopper as shown, is at once inserted. The flask is moved about till all evolution of gas has ceased and a uniform solution is obtained. The flask is now held obliquely, and the upper stopper is tightly pressed into a second flask, holding 80—100 c.c. of pure water, after which it is inverted as shown, so that the water pours into the sulphuric acid solution. By pouring the liquid backwards and forwards and shaking thoroughly, the gases are completely absorbed, and the solution will now give the iodide of starch reaction strongly if any $HNO_3$ was present in the water. When a quantitative determination is to be made, the second flask should contain instead of pure water a measured quantity of standard solution of $K_4FeCy_6.3H_2O$, the excess of unoxidised ferrocyanide being determined after the reaction by titration with permanganate. The calculations are obvious: $3Cl = HNO_3$. Organic matter need not interfere with the titration with $KMnO_4$, as the colour of the latter disappears instantaneously when $K_4FeCy_6$ is present, whereas organic matter decolorises it comparatively slowly. Test analyses are stated to have been satisfactory.

Fig. 1.

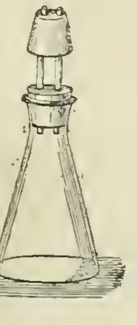
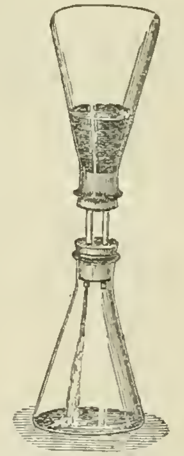


Fig. 2.



If the liquid to be tested does not contain sufficient HCl for the first reaction to occur completely, enough alkali chloride must be added.—H. B.

*Boric Acid, Estimation of, as Potassium Borofluoride.* Thaddeeff. Rev. intern. Falsific. **11**, 98; Chem. Centr. 1898, **2**, [4], 314.

The boric acid or its potassium salt (about 1 grm. of substance) is dissolved in a little water containing 2 grms. of caustic potash and an excess of hydrofluoric acid. The mixture is evaporated over the water bath, allowed to cool, 50 c.c. of a solution of potassium acetate (sp. gr. 1·14) added, the whole shaken, and allowed to stand a few hours in order to dissolve the potassium fluoride formed. The solution is then treated with 100 c.c. of alcohol and left for 12—14 hours. The precipitated potassium borofluoride is washed with alcohol and dried at 100° C. Other borates must first be converted into the potassium salt by treatment with sulphuric acid and methyl alcohol, and leading the vapour into a platinum receiver containing 2 grms. of caustic

potash and 200 grms. of water (3 c.c. of sulphuric acid and 50 c.c. of methyl alcohol, added 2—3 times, is sufficient).
—A. S.

*Boric Acid, Quantitative Determination of.* C. Montemartini. Gaz. chim. ital. **28**, [1], 344—348; Chem. Centr. 1898, **2**, [4], 314.

In an article published in 1889 in the Atti R. Accad. dei Lincei Roma, the author recognised the method of Gooch as the best for the estimation of boric acid. In this method the boric acid is set free by nitric or acetic acid, and converted into its methylester. The latter is led over a weighed quantity of lime, which is then calcined and weighed again. The author has already rejected the method of Rosenbladt, and he also objects to Thaddeeff's process. He states that the latter is complicated and inconvenient and does not yield such good results as Gooch's method.—A. S.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

*Acetylene, Toxicological Detection of.* D. Vitali. Boll. Chim. Farm. **37**, 440—454; Chem. Centr. 1898, **2**, [9], 586.

In all cases of acetylene poisoning, the acetylene should be sought for in the blood and the lungs. In both cases it may be isolated by reason of its great solubility in acetone. The lungs are cautiously evacuated and the gases passed through acetone. In the acetone solution, the acetylene may be detected by ammoniacal silver nitrate, ammoniacal copper solution, &c. The blood is agitated with acetone and the mixture heated, when the acetylene passes over with the acetone vapour, and may be detected in the distillate. If the blood is no longer fresh, carbonates and sulphuretted hydrogen are precipitated by iron sulphate, so that they may not interfere with the reactions. As acetylene is contaminated with phosphorus and sulphur compounds, these also occur in the acetone solution, and may, after oxidation with chlorine water, be detected as phosphoric and sulphuric acids.—A. S.

*Coal Tar Dyestuffs, Detection of Arsenic in, Use of the Microbiological Reaction for the.* G. Morpurgo and A. Brunner. Oesterr. Chem. Zeit. **1**, 167—169; Chem. Centr. 1898, **2**, [7], 505.

The authors recommend the method of Gosio (Rivista d'igiene e sanitá publ. Turin 1892) for the detection of very small quantities of arsenic. A slice of potato is fixed in a glass tube by means of a thin glass rod, so that it can be withdrawn at will. The whole is then sterilised and the potato inoculated with *penicillium brevicaule.* After 48 hours at 35° C., the culture has developed luxuriantly. In a second tube 5 grms. of the dyestuff to be examined is mixed with 2 grms. of tartar, triturated to a paste with water and sterilised. The culture of the *penicillium* is then brought into the tube containing the dyestuff, so that the potato touches the paste. In presence of arsenic a more or less intense garlic odour is developed in the "incubator." This occurs after 24 hours with 0·001 per cent. of arsenic, and after 4 hours with 0·01 per cent. This test is superior in sensitiveness to Marsh's test. With other fungi moulds than the one named, some give the reaction slightly and some not at all.—A. S.

*Sumac, Recognition of Adulterations of.* M. Spica. Chem. Zeit. Rep. 1898, **22**, [69], 212.

To detect *Pistacia lentiscus* (Sicilian, *Stuco*), 0·5 grm. of the powdered sumac is mixed with 5 c.c. of an 18 per cent. solution of potassium hydroxide (sp. gr. 1·155). Pure sumac gives only a brown yellow, which becomes lighter on dilution with water; but, if Lentiscus be present, the solution becomes brown with a shade of violet, and after dilution it passes into a chestnut brown. The ash of *Tamarix Africana* (Sicilian, *Bruca*), wherewith sumac is often adulterated, contains 20·129 per cent. of $SO_3$. Hence, if 1 grm. of sumac is boiled for half an hour with 100 c.c. of water, and the solution is filtered, acidified with pure nitric acid, and mixed with barium nitrate solution, the presence of Tamarix will be indicated by the turbidity of the solution.—W. G. M.

*Lard, Detection of Cotton-seed Oil in, by Means of the Phytosterol Test.* A. Bömer. Zeits. f. Untersuchung d. Nahr.- u. Genussmittel, 1898, [8], 544—552.

In previous communications (Zeits. f. Untersuch. d. Nahr.- u. Genussmittel, 1898, 1. [i.], 21—49) the author described a method of extracting the cholesterol or phytosterol from animal or vegetable fats, and showed how, by means of the form of crystallisation and melting points of the purified substance, it was possible to detect a very small percentage of cotton-seed oil in lard. The present paper deals principally with the question whether a determination of the melting point or of the form of crystallisation is the more reliable means of identifying phytosterol. E. von Raumer (this Journal, 1898, 774) suggested an improved method of extraction, in which the quantity of ether required was reduced to a very small bulk, but a second saponification was necessary. According to the author, if only 10 grms. of the fat are taken, and the crystalline form of the cholesterol, &c. determined, the quantity of ether required is not excessive, and a second saponification of the crude cholesterol is not necessary, since the presence of appreciable quantities of unsaponified fat is without influence on the crystalline form. Moreover, as the author showed before and amplifies in the present case, it is possible to detect by the shape of the crystals, the presence of minute quantities of phytosterol, when the melting point of the substance is too near to that of pure cholesterol to permit of any differentiation. On the other hand, the melting-point is considerably affected by the presence of traces of impurities. Hence the conclusion is arrived at, that a determination of the crystalline form of the product is more reliable than a determination of its melting point. This conclusion is borne out by the results of a series of experiments on mixtures of known composition, from which it appears that under favourable conditions as little as 1 or 2 per cent. of cotton-seed oil can be detected in this way in lard and 3 to 4 per cent. in any case; and hence the author asserts that the test is more sensitive and generally applicable than any other yet proposed.—C. A. M.

*Aldehydes in Alcohol, Detection of Different, by Means of Phenols.* Barbet and Jandrier. Ann. Chim. anal. appl. 1896, [1], 325; Chem. Centr. 1897, **2**, [3], 226.

Two c.c. of strong alcohol are added to a few centigrams of phenol in a test tube, and 1 c.c. of pure sulphuric acid allowed to flow down the side of the tube. Different colour reactions are formed, according to the kind of aldehyde present, in both the alcoholic and acid layers. The colour also often changes on mixing the layers. Hydroquinone and β-naphthol are especially suitable as reagents for aldehydes, the former giving an orange coloration; and the latter a carmine red with benzaldehyde, and a yellow colour with greenish fluorescence with other aldehydes (limit of sensitiveness for acetaldehyde, 1 in 200,000). The author recommends phloroglucinol as group reagent. These reactions are serviceable for the colorimetric estimation of aldehydes. By applying the reactions inversely, phenols may be detected by means of aldehydes, phenol giving a heliotrope colour with acrolein, α-naphthol a red violet with furfural, and β-naphthol a carmine red with benzaldehyde. If the sulphuric acid used contain nitrous compounds, a colour reaction is obtained directly with phenol.—A. S.

*Aldehydes in Alcohols, a New Reaction for Detecting.* C. Istrati. Bul. Soc. Sciinte din Bucuresci, 1898, **7**, 163.

The author uses Barbet and Jandrier's (see previous abstract) reaction. He mixes 2 c.c. of the alcohol with 0·2 c.c. of the reagent, and adds gradually down the side of the sloped test-tube 1 c.c. of sulphuric acid. Besides the phenols mentioned by Barbet and Jandrier the author recommends the following reagents: *Orcin*, with acetaldehyde yellow, with furfural yellow-brown; *Phenylhydrazine hydrochloride*, with formaldehyde green, afterwards bright brown, with furfural green, with benzaldehyde brown-red; *Antipyrine*, weak yellowish rose with all aldehydes save formaldehyde and acetaldehyde; *Vanillin*, brown with pure alcohol, with benzaldehyde a brown-grey, vanishing on addition of water, with formaldehyde and acetaldehyde a splendid blue precipitate; *Codeïne* (one drop), with formaldehyde yellowish violet, with acroleïn green; *Brucine*, with benzaldehyde violet, appearing gradually; *Morphine hydrochloride*, with formaldehyde violet, with methylal heliotrope; *o-Monochlorophenol*, with formaldehyde or acetaldehyde a fine rose, with furfural a splendid violet; o-*Naphthoic acid*, with formaldehyde yellow-brown, with valeraldehyde (after half an hour) yellow, with acroleïn fine yellow with green fluorescence.—J. T. D.

### *Furfural in Beer, Occurrence and Detection of.* C. Heim. Zeits. für d. ges. Brauwesen, **21**, [21], 258—260.

Referring to the test proposed by Windisch (this Journal, 1898, 593), the author states that its delicacy may be increased by omitting the first addition of hydrochloric acid and by using less acid than is necessary to dissolve the aniline, the drops of which then become stained from rose- to deep cherry-red according to the proportion of furfural present, whilst the rest of the aqueous liquid remains colourless. In this case the aldehyde, being more soluble in aniline than in water, is extracted by the former, and is converted into furaniline chloride by the added acid. This reaction will reveal furfural when in as small a proportion as 1 : 1,000,000.

That distillation or the action of yeast is unnecessary for the elimination of furfural is shown by the fact that aqueous solutions of 1 : 100,000 and 1 : 1,000,000 entirely lose their furfural content on exposure to the air for six to ten days, probably by oxidation into pyromucic acid.—C. S.

### *Formaldehyde, The Recognition of.* D. Vitali. Boll. Chem. Farmac. 1898, **37**, 321.

If to a weak solution of formaldehyde an equal bulk of a 4 per cent. solution of a phenylhydrazine salt be added, a white cloudiness occurs, gradually becoming yellow, and after some hours yellowish-red. The turbidity occurs at once in a 1 per cent. solution of formaldehyde, while in a solution containing 1 part in 100,000, it appears after two or three hours. The amorphous solid which settles out is soluble in alcohol, and can be obtained in rhombic tables by evaporating this solution. If the mixture of formaldehyde and phenylhydrazine solution be allowed to evaporate to dryness, and the washed residue taken up with alcohol, the yellow solution reduces a drop of gold chloride and gives a greenish liquid, which is reddened by sodium hydroxide solution. If the mixture of formaldehyde and phenylhydrazine solution be mixed, immediately on the appearance of the turbidity, with sodium nitroprusside and sodium hydroxide, a splendid blue colour is struck. This reaction can be used to detect formaldehyde in the presence of acetaldehyde, if the latter be not present in too great quantity, as acetaldehyde gives no colour under the circumstances.—J. T. D.

### *Quinine, Detection of.* E. Pollacci. Gaz. chim. ital. **28**, [1], 391—394; Chem. Centr. 1898, **2**, [5], 386.

In the detection of quinine by the "thalleoquinine" test, the author uses lead peroxide, instead of other oxidising agents. 0·01 grm. of the sample is treated with 1 c.c. of water and 2 drops of sulphuric acid, when the characteristic fluorescence is produced in presence of quinine. If a piece of lead peroxide is added, the mixture warmed with agitation, then 2—3 c.c. of water added, allowed to settle, and then an upper layer of ammonia added, an emerald green ring is produced in presence of quinine.—A. S.

### *Orange Oil and Bergamot Oil, Adulteration of, with Oil of Turpentine; New Method for the Detection of.* G. Mancuso-Lima. Staz. sperim. agric. ital. **31**, 244—248; Chem. Centr. 1898, **2**, [9], 585.

The author disputes the criticism of T. Leone, who has stated that his method for the examination of lemon oil for adulteration with oil of turpentine (this Journal, 1898, 382), is useless and erroneous.

It is stated that the same method may be used for the detection of adulteration of orange oil with oil of turpentine, except that in this case the increase of the polarimetric reading only amounts to about + 6°. For detecting the adulteration of bergamot oil with oil of turpentine, the oil is saponified in the cold with a limited quantity of alcoholic potash, poured into water, the unsaponifiable oil collected, dried, and distilled. A comparison of the rotating powers of the unsaponified oil, and the saponified and distilled oils, furnishes data for the detection of adulteration.—A. S.

### *Edible Oils, Adulteration of; Examination for Cotton-seed and Sesamé Oils.* F. Jean. Ann. Chim. anal. appl. **3**, 217—219; Chem. Centr. 1898, **2**, [9], 584.

The silver nitrate reaction discovered by Becchi (this Journal, 1889, 63), and modified by Tortelli and Ruggeri (this Journal, 1898, 607), is stated not to be so certain for the detection of cotton-seed oil as the method of Halphen (this Journal, 1897, 1045). The author considers that with the modern methods of oil-adulteration, in which mixtures of oils are frequently used, the only possible way of detecting adulteration is by means of the oleorefractometer and by specific gravity determinations. It is shown that neither Tortelli and Ruggeri's nor Halphen's methods meet with success in concentrating the reducing and chromogenic bodies in the liquid fatty acids; and the methods of detecting cotton-seed and sesamé oils by means of the total acids set free in presence of petroleum ether is stated to be simpler, quicker, and sufficiently accurate.—A. S.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Carburetted Water Gas, The Chemical Composition and Technical Analysis of.* E. H. Earnshaw. J. Franklin Inst. 1898, **146**, [3], 161—176.

The constituent gases of coal gas and water gas are the same, but their relative proportions in the two mixtures vary. The following are the results of actual analysis of representative samples:—

| —— | Benzene Vapour. | Heavy Hydrocarbons. | Carbonic Oxide. | Hydrogen. | Marsh Gas. |
|---|---|---|---|---|---|
| Coal gas, per cent. | 0·50 | 4·25 | 8·04 | 47·04 | 36·02 |
| Water gas „ | 0·6 | 12·8 | 30·7 | 32·4 | 13·9 |

| —— | Higher Paraffins. | Carbonic Acid. | Oxygen. | Nitrogen. |
|---|---|---|---|---|
| Coal gas, per cent. | 0·00 | 1·60 | 0·39 | 2·16 |
| Water gas „ | 2·4 | 2·7 | 0·7 | 3·8 |

The presence of notable quantities of paraffins other than marsh gas in water gas involves modification of the ordinary methods of analysis, which presuppose that marsh gas is the only paraffin present. Hempel's scheme is followed so far as absorbable constituents are concerned. The vapours of benzene are first absorbed by agitating the gas over mercury with 1 c.c. of alcohol, previously saturated for less easily absorbable constituents. The alcohol vapour is then absorbed by 1 c.c. of water. Carbon dioxide is absorbed by potassium hydrate, and the heavy hydrocarbons by a saturated aqueous solution of bromine, the vapours of which are afterwards absorbed by potassium hydrate. Oxygen is absorbed by phosphorus, which, however, does not act in the presence of heavy hydrocarbons. If, therefore, white vapours of phosphoric acid are not observed, the gas should be passed again into the bromine solution. Carbonic oxide is absorbed by ammoniacal or hydrochloric acid solution of cuprous chloride, of which that last used should be a perfectly fresh solution. Even with this precaution about 0·5 per cent. of carbonic oxide usually remains unabsorbed and is determined later. The residual gas contains hydrogen, carbonic oxide, nitrogen, marsh gas, and higher paraffins, of which the only one may be assumed to be ethane. A portion of this mixture is exploded with a measured volume of air, the contraction is ascertained, and the carbonic acid formed and the excess of oxygen are determined by absorption, and the volume of residual nitrogen is observed. The equations are :—

(1) Contraction .........$= \frac{3}{2}(H_2) + \frac{1}{2}(CO) + 2(CH_4) + 2\frac{1}{2}(C_2H_6)$
(2) Carbonic acid formed$= (CO) + (CH_4) + 2(C_2H_6)$
(3) Residual nitrogen ... $= (N_2)$ + (0·792 × the volume of air used)

Equation (3) therefore gives the volume of nitrogen at once, but two more equations are needed for the solution of equations (1) and (2), which contain four unknown quantities. To obtain these a second portion of the gaseous mixture is mixed with air and burned over palladium black, and the contraction and the carbonic acid formed are measured. This method of fractional combustion burns the hydrogen and carbonic oxide, but leaves the marsh gas and other paraffins unaltered. Therefore—

(4) Contraction....................$= \frac{3}{2}(H_2) + \frac{1}{2}(CO)$
(5) Carbonic acid formed.........$= (CO)$.

These two equations therefore give the figures for carbonic oxide and hydrogen. Assuming that the same quantity of the gaseous mixture was used in both the explosion and combustion, the difference in the contraction in the two cases will be found by subtracting (4) from (1), and will be $2(CH_4) + 2\frac{1}{2}(C_2H_6)$, which may be designated ($a$). Similarly, the difference in the carbonic acid formed will be $(CH_4) + 2(C_2H_6)$, and may be designated ($b$). Then the ethane will be equal to $\frac{4b - 2a}{3}$, and the marsh gas to $\frac{4a - 5b}{3}$. The results may be checked by the following equation :—

$$(\text{Vol. of gaseous mixture taken}) - (H_2 + N_2 + CO) = \frac{2a - b}{3}$$

Even if higher paraffins than ethane are present, the total volume of paraffins found by this method of analysis will be correct. A comparison of the results of an analysis by the above method with those which would be obtained for the same sample of gas by the more common methods shows its superiority. The common methods are: (1) the combustion over palladium is omitted, and the hydrogen is taken as equal to the difference between twice the volume of carbonic acid formed and the amount of the contraction; and (2) Hempel's method, in which the hydrogen is determined by combustion over palladium, and the marsh gas is taken as equal to twice the difference between the total contraction on explosion and the contraction due to the hydrogen. Both these methods assume that marsh gas is the only paraffin present, and that the carbonic oxide has been completely absorbed. The results for the same sample would be :—

| — | Benzene Vapours. | Fixed Illuminants. | Carbonic Oxide. | Hydrogen. | Ethane. |
|---|---|---|---|---|---|
| By the above method .. | 0·40 | 11·70 | 32·40 | 35·57 | 1·71 |
| By the method in which the combustion over palladium is omitted ........... | 0·40 | 11·70 | 31·70 | 30·31 | .. |
| By Hempel's method .. | 0·40 | 11·70 | 31·70 | 35·84 | .. |

| — | Marsh Gas | Carbonic Acid. | Oxygen. | Nitrogen (by difference). |
|---|---|---|---|---|
| By the above method........... | 9·84 | 2·70 | 0·50 | 2·18 |
| By the method in which the combustion over palladium is omitted .......................... | 20·10 | 2·70 | 0·50 | 2·08 |
| By Hempel's method........... | 16·30 | 2·70 | 0·50 | 0·83 |

The heating value of illuminating gas could be calculated accurately from its analysis were the nature of the "fixed illuminants," or hydrocarbons removed by bromine, known with certainty. The average composition of these hydrocarbons may be found thus:—A sample of the gas from which the benzene vapours and carbonic acid have been abstracted is mixed with excess of air and exploded, and the contraction and the amount of carbonic acid formed are observed. If the gas is then analysed by the method already described, the contraction due to, and the carbonic acid formed by, the fixed illuminants can be readily found by difference. Then, if $a$ = the contraction due to, and $b$ = the carbonic acid formed by, $x$ volumes of the fixed illuminants, of which the average composition is represented by the formula $C_n H_m$, then the following equations give the values of $n$ and $m$ :—

$$m = \frac{4(a - x)}{x}$$

$$n = \frac{b}{x}$$

By this means the fixed illuminants, which formed 11·70 per cent. of the sample of gas of which the analysis is given above, were found to have the average composition $C_{2 \cdot 86}H_{5 \cdot 82}$, which approaches closely to the composition of olefins ($C_nH_{2n}$) and therefore for practical purposes the fixed illuminants in this gas might be regarded as olefins. From the formulæ by which the proportions of marsh gas and ethane were calculated it was computed that the average composition of the paraffins was $C_{1 \cdot 32}H_{4 \cdot 64}$. From Thomsen's formulæ for the calculation of the heat of combustion of hydrocarbons, the heat of combustion per cubic foot of olefins may be found to be [$n$ (757) + 70] B.T.U., and per cubic foot of paraffins [$n$ (757) + 251] B.T.U. The average heating value of olefins, $C_{2 \cdot 86}H_{5 \cdot 72}$, would therefore be = 2,235 B.T.U. per cubic foot, and that of paraffins, $C_{1 \cdot 32}H_{4 \cdot 64}$ = 1,250 B.T.U. per cubic foot. Translating Thomsen's figures for the heating values of the constituents of illuminating gas into English terms, the heating value of one cubic foot of water gas of the composition shown in the last analysis may be deduced thus (the temperature of the products of combustion is assumed to be 18° C.) :—

| —— | Volume. | | Heating Value in B.T.U.'s per Cubic Foot at 60° F and 30 ins. | | B.T.U. |
|---|---|---|---|---|---|
| Benzene, $C_6H_6$ .......... | 0·0040 | × | 3807·4 | = | 15·2 |
| Olefines, $C_{2 \cdot 86}H_{5 \cdot 72}$ ...... | 0·1170 | × | 2235·0 | = | 261·5 |
| Carbonic Oxide, CO ..... | 0·3240 | × | 321·5 | = | 104·8 |
| Hydrogen, $H_2$ ............ | 0·3557 | × | 326·2 | = | 116·0 |
| Paraffins, $C_{1 \cdot 32}H_{4 \cdot 64}$ ...... | 0·1455 | × | 1250·0 | = | 182·3 |
| Carbonic acid, $CO_2$...... | 0·0270 | | .. | | .. |
| Oxygen, $O_2$................ | 0·0050 | | .. | | .. |
| Nitrogen, $N_2$............ | 0·0218 | | .. | | .. |
| | 1·0000 | | | | 679·8 |

The heating value of the gas was therefore 679·8 B.T.U.'s per cubic foot.—J. A. B.

*Semi-Woollen Textile Fabrics, Examination of.* S. Kapff. M. Text. 1898, 193; Analyst, 1898, **23**, [270], 234.

The author states that all the methods now in use for determining the proportion of cotton and wool in a mixture are inaccurate, but he therefore recommends the following process:—The fat is determined by extracting 5 grms. of the sample with ether. The residue is introduced into boiling dilute hydrochloric acid (3 of acid to 100 of water) and the mixture well stirred for 30 minutes. The material is then boiled with water for 15 minutes, washed free from acid, dried for two hours at 100° C., and left exposed to the air for about 12 hours before weighing, in order that the fibres may absorb their original content of moisture. The loss in weight gives the amount of weighting material colouring matter, &c. This treatment also removes certain natural constituents of the wool and cotton, whilst the fibres themselves are slightly attacked. The residual fibres are separated by boiling with 250 c.c. of water containing 5 grms. of caustic soda. The cotton which is left unattacked is filtered off, washed with a litre of water, then with half a litre of water acidulated with hydrochloric acid, and finally with water until free from acid. It is dried for two or three hours at 100° C., exposed to the air for about 12 hours, and weighed. The amount of cotton fibre thus found is corrected by the addition of 4·5 per cent., this correction being based on the action of caustic soda on pure cotton wool.—A. S.

### *Tannin, Estimation of.* L. Vignon. Comptes Rend. 127, [8], 369.

The process is based on the fact that tannin is completely absorbed from an aqueous solution by boiled-off silk (*soie décreusée*) at a temperature of 50° C. in four or five hours: 5 grms. of silk are used per 100 c.c. of solution, which should only contain 0·1 grm. of tannin.

The estimation is made by difference, weighing the extract before and after the absorption by silk, in a similar manner to the present method with hide powder. The author states that titration by permanganate forms the most exact method; in this case the difference is converted into tannin (gallo-tannic acid) by applying Neubauer's equivalent (1 c.c. of permanganate solution (3,164 grms. per litre) $\frac{K_2M_2O_8}{100}$ = 0·004155 grm. of pure tannin.

Comparative results of the following methods are given:—

(1.) New method (absorption by boiled-off silk, titration by permanganate).

(2.) Aimé Girard's method (absoption by fiddle-string, titration by permanganate).

(3.) Sisley's method (precipitation of tannin by ammoniacal acetate of zinc).

(4.) New method (absorption by silk; extracts weighed).

(5.) Aimé Girard's (absorption by fiddle-strings, extracts weighed).

| — | Percentage of Tannin. | |
|---|---|---|
| | Sumac. 20 grms. leaves per 1,000 c.c. | Galls. 10 grms. per 1,000 c.c. |
| Method 1 | 16·34 | 52·42 |
| „ 2 | 16·34 | 52·42 |
| „ 3 | 16·27 | 52·03 |
| „ 4 | 16·40 | 52·65 |
| „ 5 | 16·50 | 53·10 |

For the permanganate titration the author uses 1 litre of distilled water, 50 c.c. of indigo carmine (20 grms. per 1,000 c.c.), 50 c.c. of tannin solution, and 10 c.c. of $H_2SO_4$.

The method presents the following advantages:—The absorbent used—*viz.*, boiled-off silk—is easily obtained in a state of purity; it is homogeneous, regular, unalterable, and is easily handled and weighed. Fiddle-strings, on the contrary, require a long preparation; hide-powder contains soluble organic matter, and its purity cannot be guaranteed.

The colours of the silk after the absorption may be used to compare the degree of coloration of different tanning materials.*—J. T. W.

### *Tannic Acid, A Practical Method for the Analysis of.* P. Wilhelm. Rev. Gén. des Mat. Col. 1898, 2, [20], 307—308.

This process is designed for the purpose of ascertaining the practical value of tannic acid from the point of view of its application in dyeing and printing. The following solutions are necessary:—

(*a*) Solution of Methylene Blue containing 12·5 grms. of Methylene Blue, free from zinc, dissolved in distilled water, the solution being made up to one litre and filtered.

(*b*) Solution of tannic acid containing 19·6 grms. of tannic acid dissolved in distilled water, the solution being made up to one litre.

*Mode of Working.*—To 20 c.c. of the Methylene Blue solution, contained in a beaker, are added 10 c.c. of ammonia solution (containing 100 grms. of 12·5 per cent. ammonia per litre). The solution of tannic acid is then run into this mixture from a burette until all the Methylene Blue has been precipitated, which is ascertained by spotting a piece of filter paper with a drop of this solution and noticing when the blue halo which surrounds the deep central nucleus, due to the precipitate formed, entirely disappears and gives no further colour with a drop or two of tannic acid solution. The mean of a few trials gave 5 c.c. of pure tannic acid solution (19·6 grms. per litre) as the amount necessary to precipitate all the Methylene Blue; that is, 10 grms. of pure Methylene Blue (the Methylene Blue employed contains about 80 per cent. of the pure product) are precipitated by 4·9 grms. of pure tannic acid, which is in accordance with theory.

Having thus determined the amount of pure tannic acid necessary to precipitate a certain amount of Methylene Blue, we can easily find the value of any sample of tannic acid in a similar manner. However, when the solution of tannic acid is very dilute, it is better to concentrate it, in order to obtain more exact results.—J. E. H.

### *Indigo on Cloth, Estimation of.* A. Binz and F. Rung. Zeits. angew. Chem. 1898, [39], 904—905.

In order to establish the applicability of the glacial acetic acid method, a comparison of this with a modification of Renard's hydrosulphite method was made.

*The Hydrosulphite Method.*—The sample was heated with hydrosulphite solution on the water-bath until the cloth was completely decolorised. The solution was then washed out with hot water and the cloth left behind in the vessel. As the indigo-white solution is retained tenaciously by the cloth, 2·5 to 3 litres of hot water are required to wash out even a few decigrams of the dyestuff. By conducting a current of air through the solution the indigo-white was oxidised and the indigo was collected on a weighed filter.

*The Glacial Acetic Acid Method.*—Brylinski's method (this Journal, 1898, 609) was followed, but instead of filtering after the extraction, 150 c.c. of ether was added to the extract; the indigotin remains suspended in the upper layer of ether, and this was separated from the lower layer of aqueous acetic acid. The ethereal suspension was found to filter well, and was then washed with alcohol and ether. (Suitable quantities for extraction are: 10 grms. of fabric, 150 c.c. of glacial acetic acid, and wash out with 300 c.c. of water.)

From the table of results given it can be seen that the glacial acetic acid method leads to higher results than does the other method. This may be due to the reduction by hydrosulphite going too far; and experiments have been made which prove that it is not due to the indigo being mixed with an acetyl-cellulose (produced by the action of the acetic acid on the fabric), for this is easily soluble in ether, and would therefore be washed out.

In the opinion of the authors the acetic acid method is the best yet proposed.—J. McC.

### *Formic Acid, Determination of, in Presence of Acetic Acid and of Easily Oxidisable Organic Substances.* A Leys. Monit. Scient. 12, Sept. 1898, 632—635. (See this Journal, 1898, 804.)

The author finds that mercuric acetate is reduced to mercurous acetate by the action of formic acid, 1 mol. of acid being required for 1 mol. of acetate. Mercuric formate is probably first produced with liberation of acetic acid; the former then decomposes into mercurous formate, formic acid, and carbon dioxide, and finally the mercurous formate reacts with the acetic acid to form mercurous acetate. By allowing mixtures of solutions of mercuric acetate and formic acid to remain for some time, or by boiling until crystals appear on the surface and then cooling, mercurous acetate crystallises out in lustrous white scales; in presence of acetic acid, alcohol, or aldehyde the crystals are denser and more granular. According to the author there are probably two forms of crystals, of which one contains a certain quantity of water. This reaction may be used to determine formic acid in presence of acetic acid as follows. The acidity of the solution to be tested is first determined. If it contain only a small quantity of formic and a large quantity of acetic acid, it is diluted with water until only about 20 to 30 per cent. of acid is present; if, however, the proportion of formic acid amount to 1 in 20 of acetic acid or more, it is diluted so as to contain only about 2 per cent. of acid. To 10 c.c. of the solution, 20 to 30 c.c. of a 20 per cent. solution of mercuric acetate are then added, the mixture made up to 100 c.c., and boiled for seven to eight minutes. Unless a large excess of the acetate is used, the mercurous acetate begins to decompose. After cooling and standing over night, the crystalline precipitate is filtered off on to glass wool, the filtrate being used for washing the last portions from the vessel, drained, washed four or five times with a mixture of alcohol (95°) and 2 per cent. by volume of glacial acetic acid, then two or three times with alcohol alone, and finally with anhydrous ether. After drying in the air—or, better, in a vacuum—the mass is dissolved in nitric acid, diluted with an equal volume of water as rapidly as possible, to avoid a slight precipitation of mercury, which disappears, however, on standing. The solution is made up to a suitable volume; 10 c.c. are diluted largely and then precipitated by a slight excess of sodium chloride. The mercurous chloride is filtered, washed, and dried at 100°, care being taken not to prolong the drying beyond the minimum time necessary, as even at 100° there is a slight loss by volatilisation. Finally the weight of mercurous chloride × 0·0976 gives the corresponding amount of formic acid.

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*Note.—Boiled-off silk may be prepared from raw silk by boiling 20 grms. of the latter in 500 c.c. of water containing 50 grms. of white soap, for 30 minutes; rinse well and wring out, and repeat the operation in a fresh bath: after the second boiling, wring and rinse thoroughly in distilled water to get rid of all soap.

Accurate results are obtaining even with a mixture of 99 per cent. of acetic acid and 1 per cent. of formic acid.

When alcohol is present with formic acid, the solution is diluted so as to contain 1 to 2 per cent. of acid, and before adding mercuric acetate, 2 c.c. of glacial acetic acid are added to prevent precipitation.

Methylic and ethylic alcohols and their aldehydes do not interfere with the reaction. The method is suitable for the determination of small quantities of formic acid in certain natural spirits, for the detection of adulteration, for the determination of formic acid in the "formol" of commerce, and for the estimation of formaldehyde after oxidation.

—E. W. W.

*Fats, Determination of the Ash of.* A. Delecœuillerie. Rev. Pharm. de Flandres, **5**, 65; Pharm. J. 1898, **61**, [1469], 217.

The following simple method is recommended for burning off fats, previous to incineration, in the ash determination. A weighed quantity of the fat is melted in a platinum dish, and a small filter-paper, free from ash, is folded in four and placed upright in the melted fat. The paper is then lighted and serves as a wick, the fat being quickly and completely burnt off.—A. S.

*Fatty Acids, Liquid; Separation of, in the Form of Zinc Salts.* A. Bömer. Zeits. für Untersuch. d. Nahr.- u. Genussmittel, 1898, [8], 541—543.

The author gives a table of comparative results, showing that the iodine values of liquid fatty acids obtained by Jean's method (treatment of the zinc soaps with ether) agree well with those of the ordinary method, in which the lead soaps are treated with ether.

He has also tried whether it is possible to determine the iodine value of the zinc salts before converting them into fatty acids and thus to calculate the iodine value of the latter. It is pointed out that since the molecular equivalents of the higher unsaturated fatty acids differ but slightly, a variation in the percentage of these acids in a mixture would not have a very great influence on the result. Thus, 100 parts of oleic acid correspond to 111·19 of zinc oleate; 100 of linolic acid to 111·27 of zinc linoleate, and 100 of linolenic acid to 111·35 of zinc linolenate. Hence, without risk of a considerable error, the molecular equivalent of the zinc salts of mixed liquid fatty acids may be taken as that of oleic acid salt (627·1); and since 100 parts of zinc oleate correspond to 89·94 parts of oleic acid, the iodine value of the liquid fatty acids (taken as oleic acid) may be calculated by dividing that of the zinc soap by 0·8994 or multiplying it by 1·112.—C. A. M.

*Fatty Acids, Liquid; The Application of Benzene in the Determination of the Iodine Value of.* K. Farnsteiner. Zeits. für Untersuch. d. Nahr.- u. Genussmittel, 1898, [8], 529—532.

In a former communication (this Journal, 1898, 801) the author described a method of separating the liquid and solid fatty acids of a fat by means of benzene, and he has now made further experiments with the object of determining whether the Hübl value of the liquid acids can be directly determined in the benzene solution. He finds that commercial benzene absorbs a considerable amount of iodine, even after treating it with dilute acids and alkalis, repeatedly distilling, and rejecting the last fractions of the distillate. When, however, the benzene is completely freed from thiophen (this Journal, 1896, 584 and 706; and 1897, 131), not a trace of iodine is absorbed on treatment with Hübl's solution. On this fact the author bases the following method. From 1 to 2 grms. of the fat are converted into lead salts of the fatty acids in the usual manner, and dissolved in 100 c.c. of benzene (free from thiophen) at a gentle heat. After being left from 10 to 15 minutes, until a precipitate commences to crystallise, the flask is allowed to stand for about two hours at a temperature of from 8° to 12° C., and the liquid then filtered without subsequent washing of the precipitate. After shaking the filtrate with about 100 c.c. of 10 per cent. hydrochloric acid until the fatty acids are liberated, the benzene solution is twice washed with water and filtered. Two portions of 25 c.c. are taken from the filtrate and treated with the Hübl solution in the usual manner, whilst a similar third portion is evaporated in a current of hydrogen and the residue weighed, in order to determine the quantity present in the other fractions.

—C. A. M.

*Cotton-seed Oil, Oxidised; Notes on; with a Method for the Analysis of Oxidised Oils in General.* W. Fahrion. Zeits angew. Chem. 1898, [31], 781—785.

The author's experiments were primarily made with the object of determining whether the oxidation of cotton-seed oil caused any increase in the amount of unsaponifiable matter. Small quantities of the oil were distributed on strips of wash-leather and exposed to the air for 8 and 12 days respectively, after which the leather was cut into small fragments, extracted with cold petroleum spirit, the solvent evaporated, and the residue dried and examined. Two oxidation products, A and B, were thus obtained, which were considerably thicker than the unoxidised oil. Since the oxidation products became more insoluble in petroleum spirit as the oxidation proceeded, the fragments of leather which had been extracted with that solvent were further treated, and yielded in the first case a trifling amount, and in the second (12 days' oxidation), a considerable quantity of a thick yellow syrup. This syrup (B′) was completely soluble in hot alcohol—a property which the author regards as characteristic of the glycerides of oxy-fatty acids.

Since this syrup was insoluble in petroleum spirit, it could not be saponified by Henriques' method of cold saponification. The author therefore made use of the following process, which he considers applicable to the analysis of oxidised oils in general:—From 2 to 3 grms. of the oxidised oil are saponified with 10 c.c. of 8 per cent. alcoholic soda on the water-bath. The alcohol is evaporated, the soap dissolved in hot water, and the solution decomposed with hydrochloric acid in a separating funnel, shaken with 25 c.c. of petroleum spirit, and allowed to stand over night. The non-volatile acids are all contained in the petroleum layer, and it is not necessary to again shake out the aqueous layer with petroleum spirit. After running off the lower layer, the petroleum layer is withdrawn, leaving the "oxy-fatty" acids in the funnel. If their quantity is considerable they may enclose unoxidised fatty acids, and it is therefore advisable to dissolve them in a dilute solution of soda or ammonia, and repeat the treatment with petroleum spirit after acidifying with hydrochloric acid.

The united petroleum spirit extracts are evaporated, and the residue, consisting of the unoxidised fatty acids and the unsaponifiable matter dried to constant weight (1). It is then dissolved in 25 c.c. of 90 per cent. alcohol and titrated with seminormal alkali, the mgrms. of KOH required being calculated on 1 grm. of the original oil. The number thus obtained, which the author terms the "inner saponification value," furnishes a measure of the non-volatile and unoxidised fatty acids.

The neutral alcoholic solution is extracted with petroleum spirit, the extracts washed with alcohol, the petroleum spirit evaporated, and the residue of unsaponifiable matter dried and weighed (2). The difference between 1 and 2 gives the quantity of non-volatile fatty acids, the molecular weight of which can be calculated from the inner saponification value.

The "oxy-fatty" acids left in the separating funnel are dissolved in hot alcohol, the solvent evaporated, the residue dried to constant weight, ignited, the ash deducted, and the difference taken as the "oxy-fatty" acids (3). The sum of 1 + 3 gives the Hehner value.

In this way the author obtained the following results :—

| ——— | Cotton-seed Oil. | A. | B. | B′. |
|---|---|---|---|---|
| Iodine value | 108·8 | 55 4 | 46·3 | 29·1 |
| Acid value | 2·2 | 13·3 | 13·8 | 33·4 |
| Saponification value | 190·4 | 223·1 | 227·5 | 271·3 |
| Inner saponification value | 186·9 | 128·8 | 128·9 | 74·4 |
| Hehner value | 94·22 | 85·34 | 83·62 | 74·20 |
| Unsaponifiable matter Per Cent. | 1·10 | 1·11 | 1·28 | 0·72 |
| "Oxy-fatty" acids „ | 0·27 | 20·70 | 19·43 | 37·72 |
| Non-volatile fatty acids „ | 92·85 | 33·53 | 62·91 | 35·76 |
| Their molecular weight | 278·1 | 276·2 | 273·2 | 269·1 |
| Their melting point | 35—36° | 45—46° | 46° | 51° |

Volatile acids are produced during the oxidation. The author also shows that butyric acid is one of the volatile products formed on oxidising the soap of cotton-seed oil with alkali permanganate.

The oxy-fatty acids of cotton-seed oil are completely soluble in ether, whereas those of liver oils are only partially soluble. (Zeits. angew. Chem. 1891, 634.)

In the author's opinion the foregoing method of analysis affords a valuable means of following the course of the oxidation during the drying of linseed oil, and can also be applied to the examination of unoxidised fats and oils, as is shown in the following examples :—

| ——— | Beef Tallow. | Olive Oil. | Butter Fat. |
|---|---|---|---|
| Saponification value | 193·9 | 188·4 | 225·9 |
| Inner saponification value | 193·8 | 188·1 | 185·2 |
| Hehner value | 95·58 | 95·25 | 87·60 |
| Unsaponifiable matter... Per Cent. | 0·11 | 0·98 | 0·24 |
| "Oxy-fatty" acids „ | 0·13 | 0·18 | 0·14 |
| Non-volatile fatty acids „ | 95·34 | 94·07 | 87·22 |
| Their molecular weight | 275·0 | 280·1 | 263·7 |

From these results it is manifest that when, as in the case of tallow and olive oil, the total saponification and inner saponification values are practically the same, the amount of volatile or of oxy-fatty acids must be insignificant. On the other hand, butter, by reason of its volatile acids, shows a considerable difference (40·7) between the two values, and from this difference the amount of alkali required to neutralise all the volatile acids from 5 grms. of bulls' fat can be found (36 c.c. decinormal KOH), (36·3 for 5 grms.) can be readily calculated from this difference. This calculated value is higher than the Reichert-Meissl, owing to the fact that the latter only represents a portion of the total volatile acids.—C. A. M.

*Pentoses: Revised Method of Determination.* A. Grègoire and E. Carpiaux. Bull. Assoc. Belge des Chim. **12**, [4], 143—151.

In the present paper the authors give the results of their investigations into the estimation of the furfural produced by the decomposition of the carbohydrates.

Finding that the phenylhydrazine method proposed by Tollens contains a source of error in the solubility of the hydrazone in water, the authors have devised a gasometric method wherein the difference between the volume of nitrogen eliminated from equal amounts of phenylhydrazine, before and after the precipitation of furfural, forms a measure of the quantity of the latter present.

In the first case (estimation of nitrogen evolved by the phenylhydrazine reagent), cupric sulphate is employed, 4 or 4·5 grms. of phenylhydrazine being dissolved in 250 c.c. of water, 25 c.c. of this solution are mixed with 20 c.c. of concentrated hydrochloric acid, and boiled for several minutes for the expulsion of air, the acid used preventing the decomposition of the phenylhydrazine. The hot liquid is transferred to a Schloesing (nitric acid) apparatus, already charged with 25 c.c. of a 20 per cent. solution of cupric sulphate and a little hydrochloric acid and boiled to completely expel the air. The gas disengaged by the ensuing reaction is collected, cooled, and measured, corrections being made for temperature and for the tension of the vapour of water and of chlorobenzene.

In the typical analysis cited, 94 8 c.c. of nitrogen at 19° C. and at a pressure of 748 mm. were obtained. The corrected pressure was 721 mm., and the weight of 1 c.c. at that pressure being 0·00108929 grm., the above volume, when multiplied by the factor 3·857, gave 0·3982919 grm. of phenylhydrazine, or 88·51 per cent. of the quantity taken.

For the precipitation of furfural, 81·5 grms. of sodium chloride—corresponding to the hydrochloric acid distilling over in the estimation of pentoses—were dissolved in 400 c.c. of water, and two drops of acetic acid added, followed by 0·6 grm. (maximum) of furfural and 50 c.c. of the phenylhydrazine solution already examined, the liquid being then made up to 500 c.c. and agitated for an hour in the Witt agitator.

The filtrate is then treated with an excess of hydrochloric acid to prevent decomposition, and is freed from air by boiling.

To determine the residual free phenylhydrazine, 200 c.c. of the filtrate and 10 per cent. of hydrochloric acid are treated in the Schloesing apparatus, as already described, and the gas is measured.

In the typical example, 50 c.c. of phenylhydrazine yielded 189 c.c. of nitrogen and 500 c.c. of the filtrate, 44·5 c.c., the nitrogen in the hydrazone being thus equivalent to 144·5 c.c.; temperature 18°·5 C., pressure 748 mm.

After the necessary corrections, the result, multiplied by 3·429 (furfural factor), gave 0·541816 grm., against 0·5445 grm. of furfural weighed out; *i.e.*, 99·51 per cent. of the total was recovered.—C. S.

*Milk Sugar, Examination of.* C. Beuttner. Schweiz. Wochenschr. f. Chem. Pharm. 1897, 589; Pharm. J. 1898, **61**, [1469], 238.

The following method is recommended for detecting adulteration of milk-sugar, an indication of the amount of the adulterant being also obtained. 1·2 grm. of the sample is treated with 12 c.c. of 60 per cent. alcohol, and 10 c.c. of the filtered solution (equivalent to 1 grm. of milk sugar) are evaporated to dryness on the water-bath. The amount of residue should not exceed 0·03 grm., pure milk sugar yielding an average of 0·027 grm. Milk sugar mixed with 5 per cent. of cane sugar gave 0·076 grm. of residue, which, after deducting 0·027 for milk sugar, indicates 0·049 grm., or 4·9 per cent. of cane sugar. In the same way, adulteration with 5 per cent. of glucose was indicated to the extent of 4·7 and 4·8 per cent.; with 5 per cent. of glucose and 5 per cent. of cane sugar, to the extent of 9·6 and 9·7 per cent.; and with 20 per cent. of glucose, to the extent of 18·7 per cent.—A. S.

*Optical Determination of Honey by the Polarimeter.* R. Frühling. Zeits. für öffentl. Chem. **4**, 410—412; Chem. Centr. 1898, **2**, [4], 305.

It has apparently not been observed that the polarisation of aqueous solutions of honey, freshly prepared in the cold way, such as are generally used for this purpose, is by no means constant at first, and only becomes fixed after the lapse of 5—6 hours. This phenomenon is the consequence of birotation, such as is shown by aqueous solutions of crystalline dextrose freshly prepared in the cold way, and also by concentrated aqueous solutions of levulose after dilution with cold water, and must therefore always take place with honey, the dextrose of which, wholly or partly crystalline, has separated out. The birotation diminishes very slowly at the ordinary temperature, and, with dextrose, first attains the normal condition after several hours; with concentrated levulose solutions, on the other hand, after dilution with cold water, the constant value is attained within half an hour. On warming, the birotation goes back rapidly to the normal point; on boiling, instantaneously. An addition of 0·01 to 0·1 per cent. of ammonia immediately destroys the birotation and brings back the normal value. As compared with the polarisation at the beginning, five honeys showed within five hours, or immediately; after addition of 1—2 drops of ammonia, an increase of levorotation of 12·7, 9·0, 8·2, 11·8, and 14·5 Ventzke-Soleil (13·024 grms. of honey dissolved in 50 c.c. of water; 200 mm.); with an "acacia-honey" the increase only amounted to 2·3; this was of a thin fluid consistence, and showed very little crystalline separation. If, as is apparently the case, the birotation of freshly-prepared honey sugar solutions has not been previously observed, the correctness of the numbers which have been published up to the present must be regarded as questionable. The author proposes the following method of examination:—Prepare the aqueous solution in the hot way, treat with one or two drops of ammonia, avoiding the use of lead acetate, then add "alumina paste," fill up to the mark, mix, and polarise.—A. S.

*Dextrin containing Starch, Analysis of Commercial.* R. Hefelmann and W. Schmitz-Dumont. Zeits. für offentl. Chem. **4**, 448—450; Chem. Centr. 1898, **2**, [8], 561.

1. *Dry Substance.*—2 grms. of the dextrin are weighed on to a layer of glass rod and ignited sand contained in a small porcelain dish, a little water added, the solution evaporated to dryness on the water-bath with stirring, and the residue dried for four hours at 105° C.

2. *Ash.*—5 grms. are cautiously incinerated.

3. *Soluble and Insoluble.*—5 grms. are dissolved in cold water, the solution made up to 250 c.c., 100 c.c. pipetted off into a 110-c.c. flask, 7 c.c. of ether D.A.B. added, and the flask stoppered and well agitated. The starch collects as a flocculent precipitate, while all the dextrin remains dissolved. After standing a short time, the liquid is filtered through a covered double filter, and 50 c.c. of the filtrate (= 0·915 grm. of dextrin) evaporated as described under 1. This gives the soluble matter (dextrin, sugar, and soluble salts), whilst the insoluble is the difference between the soluble and the dry substance.

4. *Sugar* (calculated as maltose).—25 c.c. of the ethereal aqueous solution are treated according to Allihn's method, the separated cuprous oxide collected on a Gooch filter and converted into CuO by Hefelmann's method of simple ignition over a Teclu burner.

5. *Dextrin* is calculated from the difference between the soluble matter and the sugar, taking into consideration the ash of the soluble matter.—A. S.

*Wines, Sweet; Determination of Phosphoric Acid in.* F. Glaser. Zeits. f. Untersuch. d. Nahr.- und Genussmittel, 1898 [8], 553—555.

Various objections have been brought against the author's method of determining phosphoric acid in sweet wines (this Journal, 1896, 716), which, in brief, consists in cautiously evaporating the wine over wire gauze, oxidising the greater part of the sugar with nitric acid, completing the decomposition with sulphuric acid and mercury, neutralising and determining the phosphoric acid by the molybdate or citrate method. The author here defends his process from these criticisms. While admitting that the citrate method gives results from 3 to 6 mgrms. per 100 c.c. of wine too low, he contends that if the oxidation be properly carried out, the results obtained by the molybdate method are absolutely accurate. Any oxalic acid formed by the action of the nitric acid on the sugar, which would have a disturbing influence on the subsequent determination of the phosphoric acid, is completely decomposed by the treatment with sulphuric acid and mercury. By adding 10 c.c. of concentrated nitric acid to the wine before the evaporation, and warming gently over the wire gauze at first, an evolution of gas soon occurs, which causes the boiling to proceed regularly and prevents loss by spurting. The addition of mercury is not always essential. If after adding the sulphuric acid the mass is dark coloured, a few drops of nitric acid are added, and the heating continued until it again becomes dark, when more nitric acid is added, and so on, until on continued heating the liquid remains clear. Besides being of value in the analysis of wines, the author considers that this method is generally applicable in all cases in which the course of the analysis would be interfered with by the presence of a large amount of organic matter.—C. A. M.

*Iodoform, Estimation of.* G. Meillère. Ann. Chim. Analyt. **3**, [5], 153—154; Analyst, 1898, **23**, [270], 233.

The following method is stated to overcome the difficulties encountered in the estimation of iodoform in presence of organic substances. The sample is treated in a flask with 25 c.c. of pure nitric acid (free from chlorine), and a slight excess of silver nitrate added (1·75 grms. to 1 grm. of iodoform), the explosion which results from the contact of iodoform and the silver salt in the dry state being prevented by this arrangement. The flask is connected with a Liebig bulb-tube containing silver nitrate solution, and is then slowly heated, without actually boiling, for 10 minutes, after which the temperature is raised to effect the decomposition of the iodoform. When nitrous fumes cease to be evolved, the liquid, after dilution to 150 c.c., is heated until clear, the precipitate collected on a tared filter, washed, and dried at 100° C. If the nitrate solution in the bulb-tube become turbid, it must be added to the contents of the flask. In two determinations made with 1 grm. of iodoform, an average yield of 1·7835 grms. of silver iodide was obtained; theoretical yield, 1·789 grms.—A. S.

*Tartaric Acid in Presence of Citric Acid, Determination of.* A. Borntraeger. Zeits. Anal. Chem. 1898, **37**, [8], 477—485.

The author has shown that a solution containing 10 grms. of KCl and 2·5 grms. of anhydrous, neutral potassium citrate in 100 c.c. of water will dissolve completely 1 grm. of potassium bitartrate at the room temperature, whereas the solubility of the bitartrate is much less if the neutral citrate be replaced by 2·5 grms. of citric acid. If the 10 per cent. KCl solution contain both 2·5 per cent. of neutral citrate and 2·5 per cent. of citric acid, the solubility of the bitartrate is still less. In analysing "lemon syrups" 20 grms. of the sample (containing 16—17 per cent. of the mixed acids) were dissolved in water, neutralised with KOH, 5 grms. of KCl added, and the whole diluted to 50 c.c. 5 grms. of citric acid in 50 per cent. solution were then added, the solution was stirred till potassium bitartrate began to precipitate, and was allowed to stand over-night; the bitartrate was filtered off, washed first with 10 per cent. KCl solution which had been freshly saturated with $KHC_4H_4O_6$, and finally with pure 10 per cent. KCl solution, after which it was titrated hot.

To determine the effect of varying the proportions of the citric and tartaric acids, the following experiments were made. Solutions of 0·5 to 5 grms. of citric acid, 0·5 to 4 grms. of pure bitartrate, and 5 grms. of KCl were neutralised hot with KOH, cooled, diluted to about 50 c.c., 3 grms. of citric acid (as 50 per cent. solution) were added and the solution was stirred, settled, &c., as above described. It was found that if the solution before neutralisation contained 5 grms. of citric acid and 0·5 grm. of bitartrate, the precipitation of the latter from the neutralised solution by 3 grms. of citric acid was very incomplete, whereas 5 grms. precipitated it almost completely. The greater the proportion of tartaric acid to citric acid in the neutralised liquid, the more rapid is the subsequent precipitation of the bitartrate; thus, with 5 grms. citric acid (as potassium citrate) and 0·5 grm. of bitartrate, precipitation took 1½—3 hours, whereas with 4 grms. of bitartrate it took 15—30 seconds. A large number of experiments are quoted, and the following conclusions are drawn therefrom:—

(1) Using 5 grms. of citric acid as precipitant, satisfactory results (97·3 to 101·3 per cent.) are obtained when the neutralised solution contains 1 grm. of bitartrate *plus* 0·5—2 grms. of citric acid; 2 grms. of bitartrate *plus* 0·5—4 grms. of citric acid; 3 grms. of bitartrate *plus* 3—4 grms. of citric acid; or 4 grms. of bitartrate *plus* 3—5 grms. of citric acid.

(2) Using 3 grms. of citric acid as precipitant, satisfactory results (97·3 to 101·4 per cent.) are obtained with 1 grm. of bitartrate *plus* 0·5—1 grm. of citric acid; 2 grms. of bitartrate *plus* 0·5—2 grms. of citric acid; or 3—4 grms. of bitartrate *plus* 1—2 grms. of citric acid.

With a higher proportion of bitartrate to citrate than those stated, the results are too high, owing to the precipitation of a difficulty-soluble acid citrate. In testing a solution containing tartaric and citric acids one should take a quantity the acidity of which corresponds with 3 grms. of citric acid, add 5 grms. KCl, neutralise with potash, dilute to about 50 c.c. and precipitate as above with 3 or 5 grms. of citric acid. If the amounts of the two acids found do not fall within the above limits, a new test must be made, adjusting the proportions by adding either citric acid or an accurately weighed quantity of tartaric acid, as required.
—H. B.

*Caffeine and Theobromine, Separation and Quantitative Determination of.* H. Brunner and H. Leins. Schweiz. Wochenschr. Pharm. **36**, 301—303; Chem. Centr. 1898, **2**, [7], 512.

The authors show that the different methods proposed for extracting both alkaloids from the kola nut yield different results, and they therefore describe the following process:— The pulverised drug (coffee, kola, cocoa, maté, &c.) is boiled for half an hour with 500 c.c. of water, fresh water being added to replace that lost by evaporation. The solution is then treated with small portions of freshly-precipitated lead hydrate until it is completely decolorised, then boiled for a quarter of an hour, filtered, the residue extracted twice with 500 c.c. of water, the solution concentrated to half a litre, and carbon dioxide led into the boiling liquid till the lead is completely precipitated. After filtering, the liquid is mixed with washed and ignited quartz sand, evaporated to dryness on the water-bath, and the residue treated with ether for 6—8 hours in a Soxhlet's extractor. The ether is then expelled, the residue thrice extracted with 50 c.c. of water, the solution let cool to 50° C., filtered, evaporated to crystallisation, and dried at 80° C., a white ash-free product being obtained. In order to separate the alkaloids, the product is dissolved in hot water, silver nitrate added, then ammonia to again dissolve the precipitate, the solution heated in a porcelain dish away from light and dust, till the ammonia is completely removed, and then allowed to cool to 30° C. The precipitated theobromine silver is filtered off, washed, dried at 100° C. on a tared filter, and weighed. The theobromine is calculated by means of the formula $C_7H_7AgN_4O_2$. The silver content may be determined by ignition.

The filtrate from the theobromine silver precipitate is treated with common salt, filtered, evaporated to dryness on the water-bath, the caffeine extracted from the residue by ether, dried at 100° C., and weighed.

The weight of material taken does not largely influence the results; 10 grms. is recommended. The authors show the accuracy of their methods by analytical data.—A. S.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Phosphorescent Sulphides of Strontium, Composition of.* J. R. Mourelo. Comptes Rend. **127**, [8], 372.

The decomposition of strontium thiosulphate and sulphite at a red heat gives rise to products which have been previously examined.

The precipitate obtained by adding a saturated solution of sodium thiosulphate to a saturated solution of strontium chloride, is shown to contain, besides strontium sulphide and sulphate, a large proportion of sodium chloride, which cannot be washed away. This precipitate exhibits a yellowish-green phosphorescence, quite different from that of strontium sulphide obtained by other processes, and is more stable, no doubt owing to the trace of sodium chloride which covers the particles and protects them from the oxidising action of the air.

An impure strontium sulphide prepared by Verneuil's method for obtaining an extremely phosphorescent calcium sulphide, gave very good results, the phosphorescence being splendid, even after keeping for two years. This product contains strontium sulphide, sodium sulphide, bismuth sulphide, sodium chloride, and varying quantities of strontium sulphate, besides a small quantity of bismuth existing as subnitrate (basic bismuth nitrate); the latter body is the active substance which causes the strontium sulphide to become intensely phosphorescent, but its activity is only developed in the presence of the alkaline constituents.

The best results were obtained by fusing for four hours at a red heat—

| | Grms. |
|---|---|
| Strontium carbonate | 100 |
| Sulphur | 100 |
| Basic bismuth nitrate | 2 |
| Sodium carbonate | 2 |
| Sodium chloride | 0·12 |

Neither basic bismuth nitrate nor strontium sulphide are of themselves phosphorescent, but their mixture—or, rather, the solution of subnitrate and sulphate in the sulphide, and in the proportions given—exhibits a maximum of phosphorescence.—J. T. W.

*Sodium, Action of Sulphur upon.* J. Locke and A. Austell. Amer. Chem. J. **20**, 592—594.

In a series of experiments in which a solution of sulphur in toluene was boiled with metallic sodium, the proportions of the elements being varied to correspond with the different sulphides, the authors found that no monosulphide was formed, but greenish-yellow porous cakes were obtained, which in the case of mixtures corresponding to $Na_2S_4$ and $Na_2S_5$ were free from residual metallic sodium, and contained 28·89 and 28·57 per cent. of sodium respectively, thus probably consisting of $Na_2S_3$ and $Na_2S_4$. Even when naphthalene boiling at 218° C, is substituted for toluene, the sodium still fails to reduce the polysulphides, for when the proportion of sodium and sulphur used, corresponded to $Na_2S_2$, the product apparently contained as much residual free sodium as with another mixture of the same proportions in boiling toluene at 110° C. Thus, whether at 110° or 218° C., metallic sodium fails to reduce a sodium polysulphide to monosulphide.—E. W. W.

*Silicide of Tungsten.* E. Vigouroux. Comptes Rend. **127**, 393—395.

Silicide of tungsten is prepared by heating in an electric furnace, 100 parts of silicon and 230 parts of tungstic oxide. There is first formed a whitish, heavy, brittle button with a metallic appearance and a distinct crystalline structure. There are also present greenish crystals of silicide of carbon.

To isolate the silicide of tungsten, the mass is suspended in dilute hydrochloric acid (1 in 10), and is attached to the positive pole of a two- or three-cells battery, a negative pole of carbon being placed in the same liquid. By this means the metal itself is dissolved and the silicide is left almost intact. It falls in crystals to the bottom, whence it is removed and treated repeatedly with aqua regia, and afterwards with ammonia, whereby the last traces of free tungsten are removed. Lastly, the silicide of tungsten is separated from any silicide of carbon by gravity in methyl iodide. The final product thus obtained gives the formula $Si_3W_2$.

Silicide of tungsten forms fine laminated crystals, of a metallic appearance and steel-grey colour, with a density of 10·9 and fusible in a strongly heated reverberatory furnace. Dry chlorine attacks it, when heated to from 200° to 300° C., with bright incandescence forming chlorides of silicon and tungsten. Bromine and iodine separately produce a bromide and iodide without incandescence when heated. It burns in pure oxygen with incandescence, and without incandescence in air. Nitrogen has no action at any temperature. Hydrochloric, hydrofluoric, and nitric acids have no action, either hot or cold. Gaseous hydrochloric acid at the temperature of the gas flame does not attack it, and hot aqua regia produces an inappreciable effect. A mixture of nitric and hydrofluoric acids, even in the cold, produces a violent action. Alkaline solutions attack it feebly, whilst fused alkalis act quickly upon it, the fused alkaline carbonates producing an alkaline silico-tungstate. Potassium nitrate has a slow action.

The analysis is conducted by heating in chlorine gas in a glass tube, when the chloride of tungsten condenses first, and the chloride of silicon is carried further away. The latter is converted into silica and weighed, and the former is treated with nitric acid to form tungstic acid, which is then ignited and weighed. There are indications of another silicide of tungsten besides the $Si_4W_2$—A. W.

### *Purple of Cassius, the Chemical Nature of.* R. Zsigmondy. Annalen, **301**, [2 and 3], 361—387.

Preliminary experiments showed that precipitates containing more than 40 per cent. of gold dissolve in ammonia, but to cloudy solutions which soon deposit part of the gold. Precipitates richer in tin dissolve to almost clear solutions, which may be boiled and kept for months without alteration. A mixture of 200 c.c. of gold chloride (3 grms. Au per litre), 250 c.c. of stannous chloride (3 grms. Sn per litre), and 4 litres of water with a slight excess of hydrochloric acid, was made. After three days the purple had deposited and left a solution free from gold and tin. The precipitate, after well washing and ignition, contained 10·3 per cent. of gold and 59·7 per cent. of stannic oxide. The dry precipitate is insoluble in strong and dilute alkalis, but when moist, the purple dissolves in water in the presence of small quantities of acids or alkalis, though it is insoluble in strong alkalis. Traces of alkalis are sufficient to effect solution. Salts and excess of alkalis and acids precipitate the purple from this solution. The purple does not pass through the membrane of a dialyser.

Colloidal stannic acid was prepared by washing the precipitate which forms in very dilute stannic chloride solutions. This colloid shows practically the same solution phenomena as the purple of Cassius.

By precipitating mixtures of solutions of colloidal stannic acid and colloidal gold with dilute acids or salts solutions, substances are obtained of the same colour as the gold solution (red, violet, and blue) and possessing all the properties of the purple. Purple of Cassius is thus to be regarded as a mixture of colloidal gold and colloidal stannic acid. The gold acquires its property of dissolving in acids through the presence of the stannic acid which is soluble in them.

"A mixture of colloids may behave as a chemical individual; the properties of one body will be hidden by the properties of the other."—A. C. W.

### *Sulphur, The alleged Decomposition of.* H. Alexander. Zeits. f. Elektrochem. 1898, **5**, [8], 95—98.

In connection with certain researches on the electrolysis of fused and solid salts, the author had occasion to repeat an experiment which has been held by Gross (Elektrochem. Zeits. **4**, 1) to indicate that sulphur is a compound containing a new element, *Bythium*. Gross maintains that when a fused mixture of the chloride and sulphide of silver is electrolysed, the new element is set free, but the author, on carefully repeating the experiment with known quantities of the pure substances, and subsequently analysing the product, was able to account for the whole of the silver, chlorine, and sulphur, and could obtain no indication of the presence of any unknown element.—J. S.

### *Alkali-Copper Thiosulphates.* Rosenheim and Steinhäuser. Ber. **31**, 1876—1877.

In consequence of publications by Bhaduri and by Muthmann and Stützel, the authors make a preliminary announcement of some results obtained in an extended research on the constitution of sulphites and thiosulphates. Sodium-cuprous thiosulphates vary greatly in composition; the cause of this lying in the formation of mixed crystals of isomorphous compounds. Muthmann and Stützel have also come to this conclusion. The authors have prepared the following double salts:—

| | |
|---|---|
| (1.) $Cu_2S_2O_3.K_2S_2O_3.1\frac{1}{3}H_2O$ ....... | Yellow: crystalline. |
| (2.) $Cu_2S_2O_3.2K_2S_2O_3.1\frac{1}{2}H_2O$ ...... | Yellow prisms. |
| (3.) $Cu_2S_2O_3.2K_2S_2O_3$ ............ | White needles. |
| (4.) $Cu_2S_2O_3.(NH_4)_2S_2O_3.1\frac{1}{3}H_2O$ .. | Yellow prisms. |
| (5.) $Cu_2S_2O_2.2(NH_4)_2S_2O_3.1\frac{1}{2}H_2O$ . | White needles. |
| (6.) $Cu_2S_2O_3.2BaS_2O_3.4H_2O$ ...... | Crystalline white precipitate. |

Of these, the first three seem to have been obtained before; (1) by Rammelsberg, and (2) by Muthmann and Stützel, in each case with slightly different water-content; and (3) by Muthmann and Stützel, who regard it as a cupric, not a cuprous, derivative.—J. T. D.

### *Phosphorus and Nitric Acid, Reaction between.* C. Montemartini. Chem. Zeit. (Rep.) 1898, **22**, 226. (From Gazz. chim. ital. 1898, **28**, 397.)

Nitric acid was allowed to act on red phosphorus at the ordinary temperature. The phosphorus was scarcely attacked by 2·4 per cent. acid in six months; acids containing 17·3 and 28·3 per cent. dissolved it in times varying from 16 to 40 days. With 68 per cent. acid, the reaction, beginning slowly, soon became violent, heat being evolved. In all cases ammonia was formed in quantities varying from 0·0039 to 0·0856 grm. for 1 grm. of phosphorus. The largest quantity was observed with the 28·3 per cent. acid.—T. E.

### *Phosphoric Acid with Magnesia, Notes on Compounds of.* H. Struve. Zeits. Anal. Chem. 1898, **37**, [8], 485—496.

When strong ammonia is poured upon the salt $2MgO.P_2O_5 + 7H_2O$ (J. Chem. Soc. 1897, ii, 372), the crystals gradually swell up and change into basic ammonium magnesium phosphate $(NH_4)_2Mg_2P_2O_8 + 12H_2O$. The latter salt, when prepared, as usual, by precipitation from solutions of alkaline phosphate and of a magnesium salt and then air-dried, loses 10 molecules or 36·75 per cent. of water at about 100° C. By heating the air-dried salt in a current of dry air, it was found to suffer no change till 60° was reached, when the first traces of ammonia were liberated. At 80° water began to come off; after several hours at 100° C. the residue amounted to 51·08 per cent and 5·13 per cent. of ammonia had been lost; on gradually raising the temperature more water and ammonia were driven off, and on gentle ignition the last portions of ammonia were driven off, a partial oxidation to nitrous acid and water taking place. Towards the end the gases evolved always showed an acid reaction, due to nitrous acid. When ignited strongly the characteristic sudden glow of the residue took place, with separation of traces of carbonaceous matter, and by igniting this residue in oxygen, carbon dioxide was developed. The carbonaceous matter is probably derived from volatile organic bases present in the ammonia. The residue left after the strongest ignition amounted to 14·86—15·08 per cent. When the air-dried salt was heated in a stream of hydrogen, the changes were similar to those just described. On distillation with water, the salt lost 3·05 per cent. of $(NH_4)_2O$, the remainder being liberated on addition of dilute KOH solution. The air-dried salt loses both water and ammonia when kept even at the room temperature in a desiccator over sulphuric acid; thus, a quantity of 3·408 grms. lost 16·67 per cent. in one month, and in four months had lost 48·52 per cent., the composition of the residue corresponding with the formula $(NH_4)_2Mg_4P_4O_{15} + H_2O$. Over glacial phosphoric acid there was, however, no loss, and the author states that although sulphuric acid is regarded as non-volatile at ordinary temperatures, when it was used, a minute film containing sulphate of ammonium was perceptible on the interior surfaces of the desiccator.

When acted upon by glycerin, the air-dried salt loses both ammonia and water, changing slowly at ordinary temperatures, but rapidly when warmed, into 2MgO, $P_2O_5 + 7H_2O$.—H. B.

*Phosphine, Action of, on Copper Sulphate.* E. Rubénovitch. Comptes Rend. **127**, [5], 270—273.

Pure phosphine prepared from phosphonium chloride was allowed to act on copper sulphate solution, air being carefully excluded throughout the whole of the operations. A black precipitate was formed, which, after careful washing, gave figures agreeing with the formula $P_2Cu_5H_2O$. Two molecules of copper sulphate were found to act on one of phosphine, and one-fifth of the total phosphorus appeared as phosphoric acid; hence the equation—

$$10CuSO_4 + 5PH_3 + 6H_2O =$$
$$2(P_2Cu_5.H_2O) + H_3PO_4 + 10H_2SO_4 \quad .$$

The black phosphide begins to lose water at 80° C., and is completely dehydrated at 150° C., becoming reddish-brown. When hydrated it oxidises in the air but slowly; but dehydrated it oxidises rapidly.—J. T. D.

*Plants, Occurrence of Simple Carbon Compounds in.* A. Lieben. Monatsh. für Chem. 1898, **19**, [6], 333—353.

The author has observed that the sole product of the reduction of carbonic acid by nascent hydrogen is formic acid. If this were also the case in green plants, formic acid should always be found in them, at least in small quantity.

To test this view, grass and the leaves of different trees were distilled with 0·6 per cent. sulphuric acid in a current of steam, and finally over the naked flame, care being taken, however, that the material never became dry. Both distillates contained the same compounds. In many experiments the first part of the distillation was carried out with a fractionating column, in order to isolate any volatile products present. In all cases the distillate contained formic and acetic acid, the latter in the larger quantity, and very small quantities of a solid acid, sparingly soluble in water. This acid was so readily oxidised and decomposed that it could not be recognised. Indications of the presence of propionic acid were also noticed. The total amount of acid obtained was always small; 62 kilos of grass, for example, yielded 11 grms. of the barium salts of the mixed acids. When the grass or leaves were distilled immediately after gathering, methyl alcohol was invariably found, whereas if they had remained over-night in contact with the dilute sulphuric acid, ethyl alcohol would have predominated. The acids were not affected in this way. Formaldehyde and acetaldehyde were not found in the distillates. A series of experiments was now made in which sugar, filter-paper, and starch were treated in the same way as the leaves and grass. Neither methyl nor ethyl alcohol was formed, but formic acid was always present in the distillate with minute quantities of an oil probably containing furfurol, and a solid acid probably identical with that produced from the leaves. No acetic acid was formed.

It follows, therefore, that the formic acid and the solid acid are formed by the action of the dilute sulphuric acid on carbohydrates contained in the leaves, and are probably not themselves present in the leaves, whilst ethyl and methyl alcohols and acetic acid are present in the leaves, the ethyl alcohol proably in the form of an ester.—T. E.

*Phenols, Action of Bromine upon, in the Presence of Aluminium Bromide.* F. Bodroux. Bull. Soc. Chim. 1898, **19**, [16—17], 756—760.

The reactions were effected by adding the phenol in small quantities to bromine containing 1 per cent. of aluminium bromide in solution. Under these conditions bromine replaces all the hydrogen atoms of the nucleus, leaving the hydroxyl group unattacked. If a lateral chain is connected to the nucleus by the group CH⋮ or C, it is destroyed and a bromine atom takes its place. The lateral chain is not removed if union to the nucleus takes place through the group $CH_2$. Similar results had previously been obtained with the hydrocarbons.—A. C. W.

## New Books.

Inorganic Chemistry according to the Periodic Law. By F. P. Venable and Jas. Lavis Howe. The Chemical Publishing Co., Easten, Pa. 1898. Price 3 dols. H. Grevel and Co., 33, King Street, Covent Garden, W.C.

It is stated by the authors that they claim on behalf of this book, that it takes the periodic system for its guiding principle throughout, following the advice of Lothar Meyer in his address to the German Chemical Society. It is a volume of small 8vo size, comprising preface, table of contents, and 260 pages of subject-matter. There is an alphabetical index of subjects. The work is illustrated with 35 woodcuts. The text is divided into seven parts, apportioned as follows:—I. Definitions. Nomenclature. II. The Elements. III. Compounds of the Elements with Hydrogen. IV. Compounds of the Elements with the Halogens. V. Compounds of the Elements with Oxygen, Sulphur, Selenium, and Tellurium. VI. The Nitrides, Phosphides, Carbides, Silicides, and the Alloys. VII. Metric System. Thermometry.

Laboratory Text Book for Brewers. By Lawrence Briant. Second Edition; entirely re-written. Fell and Briant, 16, Glasshouse Yard, London, E.C. 1898. Price 10*s*. 6*d*.

Small 8vo volume containing prefaces to first and second editions, table of contents, 327 pages of subject-matter, an appendix of 21 pages, and an alphabetical index of subjects. The work is well illustrated with woodcuts, plates representing microscopic figures, and diagrams. The appendix is composed of tables, and a list of apparatus and chemicals necessary for fitting up a small brewer's laboratory. The subject-matter is subdivided into chapters under the following headings:—I. Carbohydrates and Allied Substances. Nitrogenous Matters and Enzymes. II. Apparatus and Manipulation. III. Analysis. IV. The Polarimeter. V. Interpretation of Analyses. VI. Standard Solutions and Reagents. VII. Typical Analyses. VIII. Microscopical Work.

Chemisch-technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr. Emil Jacobsen. 37. Jahrgang. 1898. Erstes Halbjahr. Erste Hälfte. Mit in den Text gedruckten Illustrationen. R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder, Schönebergerstr. 26, Berlin, S.W. H. Greve and Co., 35, King Street, Covent Garden, London.

This number for the first half-year of 1898, and the first half thereof, treats of the following subjects of Applied Chemistry:—Building Materials, Cements, Artificial Stone. Dyestuffs, Dyeing and Calico Printing. Coal-tar Products. Fats, Oils, Illuminating and Heating Materials. Fermented Liquors. Tanning, Leather and Glue Manufacture. Textiles. Glass and Earthenware. Wood and Horn. India-rubber and Gutta-percha, Cements, and Adhesives. Lakes, Varnishes, and Paints. Metals.

Commercial Organic Analysis. A Treatise on the Properties, Proximate Analytical Examination, and Modes of Assaying the Various Organic Chemicals and Products employed in the Arts, Manufactures, Medicine, &c. With concise methods for the Detection and Determination of their Impurities, Adulterations, and Products of Decomposition. By Alfred H. Allen, Past President of the Society of Public Analysts, Public Analyst for the West Riding of Yorkshire, the City of Sheffield, &c.

Second Edition, Revised and Enlarged. Volume IV. Proteids and Albuminous Principles, Proteoids or Albuminoids. J. and A. Churchill, 7, Great Marlborough Street. London, 1898. Price 18s.

This is the concluding volume of the second edition of the author's Commercial Organic Analysis, an 8vo volume, containing 580 pages, comprising preface, table of contents, subject-matter, appendix, addenda, alphabetical index of subjects, and list of errata. The text is illustrated with 35 wood engravings. The chapters of the work are devoted to the following subjects:—Proteids and Albuminous Principles. General Characters of Proteids. Classification of Proteids. Analytical Reactions. Proteids of Eggs. Of Blood Plasma. Proteids of Urine. Proteids of Plants. Proteids of Milk. Milk. Milk Products. Meat and Meat Products. Proteids of Digestion. Hæmoglobin and its Allies. Proteoids or Albuminoïds. Characters and Classification. Collagenes or Gelatoids. Fibroids. Chitinoids. Keratin Substances or Keratoids Appendix. Analyses of Proteids, &c. Addenda.

Petroleum Report. (299.) (July 13, 1898.)
Price 1s. 3*d.*

This is the Report of the Select Committee of the House of Commons, appointed on the 14th March 1898, to inquire into and report upon the sufficiency of the law relating to the keeping, selling, using, and conveying of petroleum and other inflammable liquids, and the precautions to be adopted for the prevention of accidents with petroleum lamps.

The following is a summary of the recommendations of the Select Committee:—

1. That the present law affecting petroleum spirit, not being adequate for public safety, should be amended.
2. To adopt a flash-point of 100° (Abel close test) as the dividing line between petroleum oil and petroleum spirit.
3. Legislation to control the storage, transport, and sale of petroleum generally, and admixtures of the same with other substances, certain heavy oils being exempted.
4. To provide for an efficient system of testing.
5. To provide for adequate supervision and administration by local authorities.
6. That official inquiries be made into the causes of accidents arising from the storage, transport, or use of inflammable liquids.
7. Statutory powers to enable the Secretary of State to issue orders affecting the manufacture and sale of lamps.
8. To spread information among the public as to the nature of petroleum, and the management of petroleum lamps.

# Trade Report.

## *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

(*From the Board of Trade Journal.*)

### Netherlands.

*Exemption of Salt from Excise Duty.*

Her Majesty's Minister at the Hague transmits a copy and translation of a Royal Decree of the 19th August, in accordance with which it has been resolved to exempt the payment of excise duty on salt when used in manufactories and trading establishments.

### United States.

*Customs Decisions.*

*Electric carbons* are dutiable at the rate of 90 cents per 100, under paragraph 98 of the Act of 1897.

*Ground sulphur* in sacks is exempt from duty under paragraph 674, Act of 1897, as sulphur not otherwise provided for; and sulphur in barrels, free of duty, under the same paragraph as crude sulphur.

### Russia—Siberia.

*Exemption from Duty of Machinery for Siberia and Ural Mining Districts.*

Her Majesty's Ambassador at St. Petersburg transmits the following list of machines and their parts that may be imported under certain conditions free of duty during the course of 10 years, to the 1st January 1909, across all the frontiers of the Empire for the requirements of Siberian and Ural gold mining.

1. Machines required exclusively for gold mining for working gold ore:—

(*a*) Concentrators of all systems for working gold-bearing sands.

(*b*) Concentrating appliances for the reduction of slimes and the extraction therefrom of slick and pyrites.

(*c*) Centrifugal concentrators for the dry process of concentration of gold-bearing stuff.

(*d*) Apparatus for amalgamating gold-bearing ores, including amalgamators of every description, mills, pans, metallic sheets and other apparatus destined for the extraction of gold by means of amalgamation.

(*e*) Apparatus for the extraction of gold by wet process, namely, ore-smelting rotating furnaces of every kind and other apparatus for chlorination, *viz.*, generators for obtaining chlorine (chlorinators), cylindrical pans of boiler and half-boiler iron, and all appurtenances belonging to them (bolts, distributors, siphons, &c.), and serving for the extraction of gold by means of cyanide of potassium and other chemical reagents, as also dynamo machines for electrical deposit of gold from cyanide solutions.

For the free importation of the machines specified on Part 1 a certificate of the District Mining Engineer of the Crown must be presented to the Custom House at which the goods are clearable to the effect that the machines and their accompanying parts which have arrived at the Custom House are in conformity with the specifications and drawings furnished to him, the engineer, and that they are really required for the working of gold and are imported by a certain person or company to be named.

In case of the establishment of depôts of gold mining machines in the provinces of Yenissei, Irkutsk, Yakutsk, and the Trans-Baikal, Amur and maritime regions, the owners of these depôts, in order to obtain permission for receiving foreign machines duty free, must present certificates from the District Mining Engineer of the Crown to the effect that the machines and their parts imported are applicable to gold mining, and come under the class of those enumerated in the published list, and that their owners really possess depôts of gold-mining machines.

### Duty on Zinc Pigments.

*Eng. and Mining J., Sept.* 24, 1898, 364.

In a recent case an importation of "lithophone," which is a pigment containing about 30 per cent. of sulphate of zinc, and 70 per cent. of barytes, was assessed by the Collector of the port of New York for duty at $1\frac{1}{4}$ cents a lb. The importers, Gabriel and Schall, of New York, protested, and the Board of General Assessors now decides that the assessment was not correct, and the pigment should be admitted at the rate of 1 cent per lb. as a "paint containing zinc."

### Export of Methylated Spirit.

*Chemist and Druggist, Oct.* 22, 1898, 675.

By a new General Order, 15 1898, just issued by the Board of Inland Revenue, permission is given for the export of methylated spirit in vessels containing 1, 2, 3, or any number of even gallons, provided the vessels are packed in cases containing not less than 10 gall's. each.

## *EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.*

### THE EXPORT OF SAL AMMONIAC FROM THE UNITED KINGDOM TO RUSSIA.

*Board of Trade J., Oct.* 1898, 408.

It having come to the knowledge of the Board of Trade that large quantities of sal ammoniac are exported from this country to Russia, in excess of probable Russian requirements, for electric batteries and similar uses, inquiries have been made into the subject through the Foreign Office, by H.M. Consular Officers at St. Petersburg, Odessa, Nicolaieff, Sebastopol, Kieff, Batoum, and Riga, with the following result :—

*St. Petersburg.*—The Consul-General at St. Petersburg reports that there is undoubtedly a considerable quantity of sal ammoniac imported annually into St. Petersburg, partly for the interior and partly for the Tentileff works near St. Petersburg, at which smokeless powder is manufactured. He further states that he has been informed that a portion of this chemical product, which figures in the import lists as sal ammoniac, consists really of soda, the competition in the sale of the latter being so keen on the part of the local agents, that they endeavour to conceal its identity under another appellation. He adds, that it is exceedingly difficult to obtain precise information on the subject, so much secrecy being observed in the trade in connection with the matter in question.

*Odessa.*—The Acting Consul-General at Odessa reports that he has been unable to discover to what use the sal ammoniac imported at that port is put, other than that connected with electric batteries and similar apparatus. He adds, that according to statistics obtained from the Custom House, the quantity of sal ammoniac imported into Odessa in 1897 was 306 tons, or somewhat less than half that imported during the previous year.

*Nicolaieff.*—The Vice-Consul an Nicolaieff states that the local demand for sal ammoniac is supplied through Odessa, the drug being used for tinning and soldering, besides being employed by chemists. He is further given to understand that it is imported in large quantities into the north of Russia for dyeing, and also for being converted into spirits of ammonia, which is extensively used for household purposes,

*Sebastopol.*—No sal ammoniac, or next to none, is imported at this port,

*Kieff.*—The Consul at Kieff states that sal ammoniac is imported into this district in two sorts—first, in crystallised powder, used for medicinal and electric battery purposes ; second, used in mechanical works, &c., principally for soldering purposes. Of the first sort, about 300 poods (nearly 5 tons) are used in the Kieff district, and are received from Germany ; whilst of the second, close on 100 poods (about $1\frac{1}{3}$ tons) is imported from England.

*Batoum.*—The Consul at Batoum reports that inquiries made by him at Batoum, Tiflis, and Baku have elicited the fact that the quantity of sal ammoniac imported into the Caucasus is merely what is required by chemists for electric and other similar uses, and that the total amount is therefore quite insignificant.

*Riga.*—The Consul at Riga states that besides being used for electrical purposes, sal ammoniac is extensively employed in woollen mills for. among other purposes, cleaning cloth and yarns. It also enters very largely into the manufacture of tin-plates, and tinners and coppersmiths use it for tinning, soldering, and brazing. He further remarks that it is generally from this salt that liquid ammonia is manufactured, and that a certain quantity is consumed in the manufacture of snuff. He adds, that the quantity of sal ammoniac imported into Riga is small.

### HENEQUEN IN YUCATAN.

*Board of Trade J., Oct.* 1898, 423.

Yucatan produces henequen fibre, chile, dyewoods, cedar, and mahogany for export ; the sugar and aquardiente produced is for home consumption. There are extensive salt pans on the coast, from which a considerable amount of salt is collected.

Henequen, which during the year averaged 1·25 dols. Mexican per aroba of 25 lb., has now risen to over 4 dols., the aroba in consequence of the scarcity of manilla fibre in the market, owing to the war. Many of the producers of henequen are under contract to supply the fibre at 1·50 dols. for the year, and are not producing more than sufficient to comply with their contracts, whilst the few that are not under contract are obtaining high prices. Thus shipments of henequen have not as yet greatly increased.

A company has been formed with local capital in Merida, which is now manufacturing rope, cordage, matting, and bagging of henequen fibre. Should this company be able to turn out bagging that can take the place of gunny bags, they will command a good business.

### THE GERMAN COKE INDUSTRY.

*Board of Trade J., Oct.* 1898, 456.

The Board of Trade have received, through the Foreign Office, copy of a memorandum drawn up by the Commercial Attaché to Her Majesty's Embassy at Berlin, on the present condition of the German coke industry.

The memorandum states that an interesting article on the development of the German coke trade has appeared in the *Stahl und Eisen* for July, of which the following is a short summary :—

The production of coke in Germany, since 1890, has increased by more than $3\frac{1}{2}$ million tons, or by 55 per cent. ; and this does not include coke produced in North German and Rhenish Westphalian works, nor in various cement factories. The districts here included are the following :—Ruhr, Upper and Lower Silesia, Saar, Berg neighbourhood near Aachen, Obern Kirchen, and the Kingdom of Saxony. If all other coke were included, the coke production of Germany would for 1897 be certainly about 12 million tons. But from the above-named districts only, the advance—from 1890 to 1897—has been from 6,411,520 to 9,960,740 tons. It is in the Ruhr district that the chief coke production occurs, which shows a development from 341,033 tons in 1870 to 5,562,503 in 1895. From a table attached to the report it appears that the value has risen from 21 million marks in 1885 to 76 millions in 1897, or by 262 per cent.

Of the estimated 12 millions of coke turned out in 1897, about 9 remained in the country and only 8 went abroad, which, however, includes Luxemburg. The German article goes on to give other data on the subject of methods of production ; and, finally, two other diagrams are given of the number of tar-ovens and of the price-curves of by-products.

### THE MANUFACTURE OF SAKAI CARPETS.

*Bd. of Trade J., Oct.* 1898, 425.

The carpet-making industry of Japan is of comparatively recent growth. The chief seat of the industry at the present day is Sakai, a town about eight miles south of Osaka, whilst a few are made at the neighbouring village of Surnivoshi.

The progress of the trade is shown in the following table :—

| Year. | Carpets Exported. | Value. |
|---|---|---|
| | No. | Yen. |
| 1880 | 1,595 | 2,043 |
| 1890 | 27,593 | 51,048 |
| 1891 | 60,811 | 94.731 |
| 1892 | 112,279 | 177,446 |
| 1893 | 203,050 | 391,989 |
| 1894 | 546,091 | 1,134.073 |
| 1895 | 685,536 | 1.635,902 |
| 1896 | 661,732 | 1,152,177 |
| 1897 | 555,878 | 973,871 |

The materials employed in carpet making are cotton wool, silk and yamamai or wild silkworms' thread, waste silk and jute.

The silk used is only that which comes from Joshiu, and the dyeing of the thread is all done in Tokio.

A great improvement has taken place lately in the dyes used, but as the large dealers are of opinion that still further improvements are necessary and desirable, a meeting of those interested is being called to try and do away with the practice now in vogue of buying cheap dyes from the native merchants in Osaka, and in lieu thereof to purchase them direct from the foreign importers at Kobe. The imported dyes now in use come principally from England and Germany. These dyes last better than those formerly used, and are now almost invariably employed in the making of wool rugs and jute rugs of the better qualities; while for cotton rugs—except such few as are dyed on what is termed the "old Japanese system"—very inferior dyes are used even now, and the colours quickly fade.

Provided that strict attention be paid to the quality of the dyes employed, and earnest endeavours made to maintain a certain uniformity of standard, there is no reason why the future of the trade should not be assured.

### Sicilian Sulphur Trade.

*Bd. of Trade J., Oct.* 1898, 459.

According to information received from Her Majesty's Consul at Palermo, the following table shows the condition of the Sicilian sulphor trade on 31st July in each of the years 1894—1898 inclusive:—

| Years. | Amount Exported during the Month ended 31st July. | Amount Exported during the Seven Months ended 31st July. |
|---|---|---|
| | Cantals. | Cantals. |
| 1894 | 322,759 | 3,052,514 |
| 1895 | 249,978 | 2,857,152 |
| 1896 | 273,981 | 3,221,918 |
| 1897 | 373,580 | 3,540,745 |
| 1898 | 319,135 | 4,018,478 |

*Note.*—13 cantals = 1 ton.

On 31st July 1898 the stocks in hand in the whole of Sicily amounted in the aggregate to 1,918,391 cantals.

### Exports of German Aniline Colours.

*U.S. Cons. Reps., Oct.* 1898, 226.

The United States commercial agent at Bamberg reports that the German chemical industry has largely increased in trans-Atlantic exportations during the last few years. In 1889 German chemicals sent abroad were valued at 53,800,000 dols.; in 1896, they were worth 77,150,000 dols., an increase of nearly 24,000,000 dols., or more than 43 per cent., within seven years.

One-third of the total increase is due to the heavy exportation of German aniline colours. They rose from 9,140,000 dols. in 1889 to 15,947,900 dols. in 1897, the imports remaining stationary at about 905,000 dols. This result is all the more remarkable, because it was achieved, as in almost every other case, in spite of lower prices; for, in 1889, the weight of the aniline colours exported amounted only to 6,975 tons, while in 1897 it reached 17,639 tons. The quantity exported in 1897 was therefore 2½ times that of 1889, while the value was far from being doubled.

The heaviest consumer is America. The United States imported last year 39,008 double centners of aniline colours; Great Britain's imports rose to 35,750 double centners; Austria-Hungary bought 16,595 double centners; China, 13,855 double centners; Japan, 5,077 double centners; Belgium, 8,281 double centners; British East Indies, 8,066 double centners; and Italy, 9,823 double centners. France, Denmark, Holland, Russia, Sweden, and Switzerland are also good customers for German aniline colours, and Greece, Norway, Portugal, Roumania, Spain, Turkey, Brazil, Canada, Mexico, and British Australia buy their aniline colours in the German market.

### Chemicals and Paints in Mexico in 1897.

*Foreign Office Annual Series, Sept.* 1898, *No.* 2184.

There was scarcely any variation in the total value of the imports of chemical products. There was, however, a very considerable increase, amounting to nearly 40 per cent. of the imports of caustic soda and potash and sulphates of iron and copper, which was set off by a decrease in paints and colours (probably due to over-importation last year), and in miscellaneous drugs and chemicals. The values of the imports according to these sub-divisions were as follows:—Paints and colours, 54,119*l.*; caustic soda and potash and sulphates of iron and copper, 98,735*l.*; and miscellaneous drugs and chemicals, 191,655*l.*

Paints are of inferior quality and very dear, and English paints ought to be able to compete on very favourable terms, whereas they are not imported at all. All the colours sold are coarsely ground, and no Japan size nor first quality paint or tinting brushes are obtainable.

### Copper and Lead Exports from Mexico in 1897.

*Foreign Office Annual Series, Sept.* 1898, *No.* 2184.

A falling-off, both in quantity and value, has occurred in the exports of copper. As a large part of the copper exported from Mexico is the produce of one particular mine, owned by the Boleo Company of Santa Rosalia, Lower California, this is probably due to local and temporary causes.

It is publicly announced that the same group of French capitalists which controls the Boleo Company has acquired a very extensive copper property in the state of Michoacan, known as Inguaran, which in former times was very productive, but of late years has been practically abandoned. If this property proves to be anything like as good as is reported, a large increase in the exports of copper may be expected as soon as the preliminary work is finished and the extraction of the ore begins.

No clean lead is exported from Mexico, and by far the greater part of the lead which figures in the export statistics is shipped in the form of argentiferous bars, as they come out of the smelting furnaces, as it is found that the separation of the silver from the lead can be more economically performed abroad. The increase, therefore, in the exports of lead, from 960,909 cwts. in 1896, to 1,177,868 cwts. in 1897, affords a proof of the activity of the smelting industry in Mexico.

### The Present and Prospective Supply of Calcium Carbide.

*U.S. Cons. Reps., Sept.* 1898, 17.

The U.S. Consul-General at Frankfort reports as follows:—

The rapidly increasing use of acetylene has pushed the demand for calcium carbide so close to the present limit of available supply, that the questions of increased productive capacity, the future output, and probable price of this new and important material, assume a definite international interest.

It is understood that the use of acetylene, and consequently the consumption of carbide, has increased somewhat more rapidly in America than in Europe, and that, notwithstanding the large output at Niagara Falls, the home demand has rather more than kept pace with the supply. The latter may practically be also said of Europe, at least of Germany, which country, notwithstanding its pre-eminence in all kinds of chemical manufacture, and all that one hears and reads of new plants projected or under construction, still obtains almost its entire supply of carbide from Switzerland, notably from the great works at Neuhausen.

A year ago about the only manufacturer of carbide in Germany was a company at Bitterfeld, in Prussian Saxony, which smelted the lime and carbon by electrical heat generated by steam power, and found itself, for that reason, unable to compete, and so suspended operations and removed its plant to Neuhausen, where it has reopened with water-power generators and a largely increased capacity.

The whole carbide industry, so far as Central Europe is concerned, is now in a transition stage, and the conditions of supply and price are likely to be greatly modified by the increased production of next year. The Compagnie Genevoise, at Vernier, on the Rhone, near Geneva, has greatly enlarged its plant, and is making contracts for 1899 at a considerable reduction from present rates.

But the most important accession will come through the operations of the great combination headed by the Schückert Electrical Manufacturing Company, at Nuremberg, which, as is stated on published authority, will increase its capital to 28,000,000 marks (6,664,000 dols.), and expects to have in operation during the course of next year the following carbide factories :—

| | Horse-power. |
|---|---|
| Electrical Works Lonza, at Gampel (Wallis) ..... | 2,500 |
| Electrical Works Hofsland, at Sarpborg, Norway. | 5,000 |
| Bosnian Electrical Company, Iajce, Bosnia....... | 8,000 |
| Société Espagnole, Brega (Catalonia) ............ | 2,500 |

The aggregate annual product of these four plants is estimated at 20,000,000 kilos. (20,000 metric tons), capable of generating 6,000,000 cubic metres (211,896,000 cubic feet) of acetylene. Besides these, the Aluminium Industry Company, of Neuhausen-on-Rhine, has this year increased its capital to 16,000,000 francs (3,088,000 dols.), for the purpose of constructing a 7,500 horse-power plant at Land-Gastein, in Austria. All these establishments will employ the original process, by which the lime and carbon are smelted together by the heat of an electric current.

The Walther process, briefly described in a former report of this series,* smelts the same materials by the heat of darting acetylene flames intensified by mechanical pressure, and is employed by a company at Leipsic, which has works at Cassel, and now advertises its product in the special organ of the carbide industry. Although this company is said to have exhibited at Berlin carbide samples of very high quality, there seems to prevail among business men some doubt as to whether this process can be made to compete economically with electrical heat generated by water power under favourable conditions. So far as can be ascertained, a favourably located and well-managed water-power plant in Germany or Switzerland ought to produce carbide of standard quality at a net cost of 160 marks (38 dols.) per metric ton, and it is doubted whether this minimum figure can be much reduced by any process now in practice. The consumption of acetylene will have to increase rapidly after next winter to keep pace with the great augmentation in the carbide supply that will then occur.

Already the effect of this future increase in production exerts a definite influence upon contract prices, and a large purchase for next year's delivery has recently been made at a reduction of 20 marks (4·76 dols.) per 100 kilos. (220 lb.) from the market price of the same firm for 1898. Carbide is a form of merchandise which varies exceedingly in price according to the quantity involved in the transaction. So far as can be ascertained, manufacturers now receive from railways and other consumers or wholesale dealers who purchase in large quantities from 34 to 35 marks (8·09 dols. to 8·33 dols.) per 100 kilos., packing charges not included. Consumers of small quantities have to pay to dealers from 10·25 dols. to 11·90 dols. per 100 kilos., exclusive of packing, the price between these two figures being governed by the quantity purchased.

The standard guaranteed strength is 300 litres of gas per kilo. of carbide, and the present situation may be summarised as follows:—

There is now in the European market no apparent surplus of carbide, and the current supply is taken up as fast as it is turned out.

There is even a demand that would justify some importation, provided the foreign-made carbide were of standard quality and could be furnished at prices within the local market rate and under conditions of delivery and payment that prevail here.

This situation can probably not continue beyond the close of the present year. The several large new carbide plants that will be started up next winter and spring will so increase the output as to probably reduce the market price to a fair profit above net cost of production, and, unless the use of acetylene increases phenomenally, leave a surplus that will be available for export.

Calcium carbide is, however, a somewhat critical kind of merchandise for international traffic. The requirements of railways and steamship companies in respect to packing, &c., are so costly and difficult of fulfilment that in one recent case a large order of carbide for China had to be cancelled because it was impossible to comply with the specifications for its transportation.

### Calcium Carbide and Acetylene in France.

*U.S. Cons. Reps., Oct.* 1898, 279.

Consul Thackara, writing from Havre, 4th Aug. 1898, says :—

There are no official statistics bearing upon the annual output of calcium carbide, so that the amount furnished yearly by the different manufacturers has to be estimated. These estimates vary from 1,000 to 5,000 tons per year from each factory, depending upon the demand and the number of hours each establishment works.

There are 10 factories at present engaged in the manufacture of carbide of calcium in France. Two are at La Bathie and one at each of the following places: Séchilienne, Froges, Chapareillan, Lancey, Notre Dame de Briançon, St. Béron, Bellegarde, and Crampagna. Four factories are undergoing construction. They will be situated at St. Etienne de Maurienne, Epierre, Serres, and Chute du Giffre. They will be able to produce, when running at their full capacity, from 2,500 to 3,000 tons per year. The wholesale price of carbide of calcium in France is from 350 to 400 frs. (67·55 to 77·20 dols.) per ton, exclusive of cost of packing. The cost of packing is 4·50 frs. (87 cents) per iron drum containing 50 kilos. (110 lb.), 6 frs. (1·19 dols.) per drum of 100 kilos. (220 lb.), and 11·50 frs. (2·22 dols. per drum of 200 kilos. (441 lb.).

The carbide of calcium manufactured in France is guaranteed to give 300 cubic litres of gas per kilo. The output is constantly increasing, and the supply is fully equal to the demand. The retail price of the article is from 55 to 60 frs. per 100 kilos. (10·615 to 11·58 dols. per 220·46 lb.), not including packing.

There are two villages in France completely lighted by acetylene by the Société Franco-Espagnole du Gaz Acétylène, of No. 81, Rue St. Lazare, Paris. They are Alzonne, in the department of Aude, a town of 1,506 inhabitants, and Saurat-par-Tarascon, in the department of Ariege, a place of 3,024 inhabitants.

The ordinary method of introducing acetylene gas into towns, up to the present time, has been the same as that used by the coal-gas companies. For the privilege of furnishing gas to consumers at certain rates, the municipality usually demands of the contracting company that the lighting of the city itself shall be virtually free. It is claimed by the French acetylene-gas companies that the installation of a gas plant in each particular house has become so simple that the consumers will not pay an increased price for gas on account of the free city lighting, but prefer to have their own plants, thus having their light at a reduced cost and independent of the city plant. It is estimated by the gas companies that a cubic metre of acetylene gas costs 38·6 cents, but the lighting power being 15 times that of coal gas, the same unit of light is obtained with a consumption of acetylene gas 15 times less than that of ordinary gas. Hence, taking 30 centimes (5·79 cents) as the price of a cubic metre of coal gas, there results an economy of 50 per cent. in the lighting power in favour of acetylene gas.

### Utilisation of Indian-corn Stalks in Russia.

*U.S. Cons. Reps., October* 1898, 215.

On the 19th of July, a most interesting experiment was made by the Russian Admiralty at the naval proving grounds at Poligon, near St. Petersburg.

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* See *Consular Reports*, No. 206 (November 1897), 337.

A coffer dam, 6 ft. long, 6 ft. deep, and 3 ft. broad, was packed with blocks of cellulose made from the pith of Indian-corn stalks. The material was supplied by the Marsden Company, of Philadelphia, and the dam was packed under the supervision of the experts of that Company. The cellulose within the dam was compressed until it weighed 8 lb. for each cubic foot. A 6-in. solid shot was fired through the dam, striking it a distance of about 20 in. from the bottom. The shot struck the dam at a velocity of 1,000 ft. a second, and passed clear through both the iron walls and the cellulose packing. Less than half a pound of the cellulose was carried out by the projectile. The water compartment of the dam was filled, giving a pressure of nearly 5 ft. of water on the perforated surface. In just half an hour a moist spot began to show on the outer surface of the dam, but it was evident that the moisture had come along the bottom of the packing and not through the path of the shot. In four hours no water had come through the shot's path, and only 6 galls. had passed under the packing. The experiment conclusively demonstrated that a ship provided with a coffer dam packed as was the one used in the experiment, could be perforated 5 ft. below the water line without the least danger of the entrance of water. This demonstration opens up an immense market for Indian-corn pith, and will prove of great advantage to American agriculture.

After extracting the cellulose for packing coffer dams and other purposes, the outer envelope of the stalk is ground into an excellent cattle food. This property of the maize stalk is of the greatest importance. It is evident that this waste product of the vast maize fields of America is destined to interest European nations and find, eventually, a wide market.

Similar trails of maize pith as an obturating material for battle-ships have been made in England with favourable results.

### QUININE IN JAVA.

*U.S. Cons. Reps., Oct.* 1898, 266.

Although for some years Java has been the principal country for producing cinchona bark, yet the actual manufacture of sulphate of quinine there has only just begun.

The first shipment to the United States of 10,000 oz. of sulphate of quinine took place last January, and up to the present date there have been invoiced for shipment to the United States 48,300 oz. of sulphate of quinine, valued at 11,395·55 dols.

There is at present only one factory for working the cinchona bark in Java, and it has been running a very short time; but the success of the enterprise is probable.

Just now things look especially bright, as the war has resulted in a rise in quinine quotations. Quinine from this factory (Bandoengsche Kinine-fabriek), which the latter part of April was at 6·15 dols. per unit,* had risen by the middle of May to 7·54 dols.

The Consul encloses a letter written by Mr. F. W. Sijthoff, the manager of the factory above mentioned, describing the industry, its history and its prospects.

As regards the cultivation of the cinchona bark, the result of the Government plantations in Java does not bear out the common opinion that commercial ventures are better managed by private enterprise. An official report just published showed that the Government in 1896 cleared a profit of over 38,500 dols. from its cinchona estates, while the same year was, as a rule, a very poor one for planters.

The Consul considers the planting of cinchona in Java a good investment, as the shortage in production in a few years will be large. Concessions of land are not hard to get.

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* The unit of quinine means the percentage of sulphate of quinine in half a kilo. (1·10 lb.) of cinchona bark. Thus, bark yielding 1 per cent. with the unit price 4 cents would bring 4 cents per half kilo., while bark yielding 5 per cent. of sulphate of quinine would bring 20 cents per half kilo.

### BEET-SUGAR EXPORTS FROM GERMANY.

*U.S. Cons. Reps., Sept.* 1898, 57.

As a supplement to a report on the German beet-sugar industry by Consul-General Goldschmidt, of Berlin,* the Consul-General adds that the total exports of sugar from Germany in 1896-97 (counting all grades, as well as stocks previously carried over) reached the enormous amount of 2,616,776,425 lb. The amount, continues Mr. Goldschmidt, was unprecedented and will probably never be reached again, since the prices, especially for raw sugar, paid the producers no profits. This amount included 660,000,000 lb. of refined and about 530,000,000 lb. of raw sugar sent to England. The Consul-General transmits the following table, with additional details :—

*Exports of Sugar (net) from Germany to Countries of Destination during the Year* 1896-97.

| Country of Destination. | Sugar of Class† a. | b. | c. | Syrup and Molasses. |
|---|---|---|---|---|
| | 100 kilos.‡ | 100 kilos.‡ | 100 kilos.‡ | 100 kilos.‡ |
| Hamburg, free port..... | 20,291 | 85,974 | 11,840 | 77 |
| Belgium.............. | 1,000 | 6,278 | 1 | 2,872 |
| Denmark ............... | 41,827 | 22,208 | 786 | 4,410 |
| France ................ | .. | 188 | .. | 246,712 |
| Great Britain........... | 2,377,862 | 2,999,272 | 68,088 | 1,956 |
| Italy .................. | 900 | 1,021 | 6 | 2 |
| Netherlands ............ | 118,210 | 86,600 | 2,639 | 9 |
| Norway ................ | 175 | 83,049 | 61,778 | 358 |
| Portugal................ | 24,360 | 6,795 | 20,947 | .. |
| Russia.................. | 2 | 82,024 | 1,465 | 597 |
| Sweden.................. | 235 | 3,058 | 188 | 281 |
| Switzerland............. | 1,221 | 103,697 | 120 | 2,443 |
| Cape Colony ............ | 10 | 8,809 | 318 | 3 |
| Other African countries and colonies. | 745 | 11,889 | 150 | .. |
| Japan .................. | 1,484 | 246,849 | 34,440 | .. |
| British East Indies ..... | 5 | 87,988 | .. | 1 |
| British North America.. | 139,218 | 274 | .. | .. |
| United States........... | 4,877,292 | 112,056 | 758 | 420 |
| Chile .................. | 35 | 73,915 | 1,278 | 92 |
| Colombia ............... | 11 | 10,329 | .. | .. |
| Other American countries. | 315 | 10,592 | 3,579 | 438 |
| British Australia ....... | 10 | 13,756 | .. | 10 |
| Other countries......... | 1,363 | 18,122 | 2,935 | 719 |
| Total in 1896-97... | 7,606,571 | 4,051,143 | 211,906 | 260,459 |
| In 1895-96 ........ | 5,044,447 | 3,986,304 | 38,168 | 400,131 |

* See *Consular Reports*, No. 211 (April 1898), p. 494.

† The kinds of sugar belonging to the different classes are, since 1st August 1896, the following :—(*a*) Raw sugar, containing at least 90 per cent. of sugar, and refined sugar containing under 98 per cent., but at least 90 per cent. of sugar; (*b*) rock candy and sugar in white, full, hard cakes, blocks, plates, sticks, or squares, or in white, hard, translucent crystals, containing at least 99½ per cent. of sugar; all these sugars come under Custom-house supervision; (*c*) all other sugars containing at least 98 per cent. of sugar.

‡ 100 kilos. = 220·46 lb.

## *BOARD OF TRADE RETURNS.*

### SUMMARY OF IMPORTS.

| Articles. | Month ending 30th Sept. 1897. | 1898. |
|---|---|---|
| | £ | £ |
| Metals................................ | 1,651,108 | 1,508,337 |
| Chemicals and dyestuffs ............ | 347,745 | 347,426 |
| Oils.................................. | 705,731 | 813,115 |
| Raw materials for non-textile industries. | 5,493,168 | 5,706,649 |
| Total value of all imports .... | 35,179,633 | 35,605,281 |

## Summary of Exports.

| Articles. | Month ending 30th Sept. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) .... | 2,825,884 | 2,567,238 |
| Chemicals and medicines........... | 636,378 | 672,238 |
| Miscellaneous articles.............. | 2,525,220 | 2,790,342 |
| Total value of all exports..... | 18,305,275 | 19,945,085 |

## Imports of Metals for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| Copper :— | | | £ | £ |
| Ore ............. Tons | 12,596 | 15,454 | 48,158 | 65,826 |
| Regulus ........ „ | 5,774 | 7,831 | 128,621 | 192,309 |
| Unwrought ..... „ | 4,433 | 5,250 | 218,643 | 275,376 |
| Iron :— | | | | |
| Pig and puddled bar. „ | 5,082 | 10,775 | 18,509 | 45,541 |
| Ore.............. „ | 420,823 | 387,331 | 306,830 | 271,980 |
| Bolt, bar, &c. ... „ | 6,149 | 6,895 | 48,469 | 55,750 |
| Steel, unwrought.. „ | 2,181 | 3,365 | 15,269 | 20,388 |
| Lead, pig and sheet „ | 15,409 | 10,921 | 196,209 | 140,352 |
| Pyrites ........... „ | 49,518 | 39,293 | 78,320 | 60,361 |
| Quicksilver ....... Lb. | 81,974 | 8,960 | 7,660 | 896 |
| Silver ore....... Value £ | .. | .. | 167,242 | 81,205 |
| Tin ............... Cwt. | 41,876 | 12,968 | 125,816 | 46,630 |
| Zinc .............. Tons | 6,386 | 6,010 | 108,554 | 116,659 |
| Other articles ...Value £ | .. | .. | 182,808 | 195,058 |
| Total value of metals | .. | .. | 1,651,108 | 1,568,331 |

## Imports of Chemicals and Dyestuffs for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............. Cwt. | 14,483 | 19,363 | 8,698 | 10,275 |
| Bark (tanners', &c.) „ | 25,685 | 28,772 | 8,004 | 8,297 |
| Borax ............ „ | .. | 137 | .. | 139 |
| Brimstone ........ „ | 16,760 | 17,530 | 3,777 | 4,453 |
| Chemicals...... Value £ | .. | .. | 96,625 | 90,653 |
| Cochineal ......... Cwt. | 369 | 213 | 2,282 | 1,202 |
| Cutch and gambier Tons | 1,821 | 721 | 29,575 | 13,645 |
| Dyes :— | | | | |
| Alizarin.......Value £ | .. | .. | 21,035 | 19,893 |
| Anilin and other „ | .. | .. | 40,339 | 43,316 |
| Indigo ........... Cwt. | 406 | 250 | 5,494 | 4,075 |
| Nitrate of potash . „ | 22,063 | 13,460 | 16,994 | 10,852 |
| Valonia........... Tons | 2,418 | 1,693 | 24,095 | 17,269 |
| Other articles... Value £ | .. | .. | 90,827 | 93,357 |
| Total value of chemicals | .. | .. | 347,745 | 317,426 |

## Imports of Oils for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut......... Cwt. | 41,212 | 17,077 | 42,632 | 16,577 |
| Olive.............. Tuns | 2,086 | 655 | 71,882 | 22,645 |
| Palm ............. Cwt. | 65,625 | 69,927 | 68,092 | 76,332 |
| Petroleum ........ Gall. | 14,430,549 | 23,375,283 | 268,136 | 380,831 |
| Seed .............. Tons | 2,892 | 3,689 | 63,925 | 63,283 |
| Train, &c. ......... Tuns | 3,300 | 3,232 | 47,568 | 49,872 |
| Turpentine ....... Cwt. | 55,660 | 112,960 | 57,041 | 122,101 |
| Other articles .. Value £ | .. | .. | 86,455 | 81,474 |
| Total value of oils... | .. | .. | 705,731 | 813,115 |

## Imports of Raw Material for Non-Textile Industries for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,490 | 2,585 | 2,612 | 6,001 |
| Bristles........... Lb. | 488,181 | 468,216 | 63,722 | 59,146 |
| Caoutchouc ...... Cwt. | 21,700 | 33,444 | 230,193 | 459,085 |
| Gum :— | | | | |
| Arabic.......... „ | 4,282 | 2,757 | 13,341 | 5,915 |
| Lac, &c. ........ „ | 10,584 | 2,491 | 44,453 | 7,340 |
| Gutta-percha .... „ | 4,454 | 5,175 | 39,841 | 54,345 |
| Hides, raw :— | | | | |
| Dry............. „ | 35,349 | 26,864 | 86,020 | 67,369 |
| Wet ............ „ | 53,003 | 56,771 | 106,020 | 124,886 |
| Ivory............. „ | 1,020 | 930 | 46,502 | 38,177 |
| Manure :— | | | | |
| Guano .......... Tons | 3,242 | 1,774 | 14,937 | 8,125 |
| Bones .......... „ | 2,790 | 4,512 | 10,308 | 19,315 |
| Nitrate of soda... „ | 8,309 | 4,407 | 60,008 | 34,243 |
| Phosphate of lime „ | 37,941 | 39,354 | 56,320 | 53,433 |
| Paraffin........... Cwt. | 60,654 | 56,602 | 49,302 | 44,746 |
| Linen rags........ Tons | 1,925 | 1,231 | 18,378 | 12,555 |
| Esparto........... „ | 20,843 | 14,055 | 81,012 | 57,574 |
| Pulp of wood :— | | | | |
| Chemical ...... „ | 26,804 | 15,104 | 126,851 | 102,751 |
| Mechanical .... „ | | 19,627 | | 60,018 |
| Rosin............. Cwt. | 129,731 | 226,126 | 31,686 | 47,876 |
| Tallow and stearin „ | 169,397 | 111,445 | 159,202 | 118,079 |
| Tar ..............Barrels | 26,732 | 27,440 | 17,336 | 21,473 |
| Wood :— | | | | |
| Hewn ......... Loads | 275,874 | 254,983 | 596,528 | 542,209 |
| Sawn .......... „ | 1,025,668 | 1,140,695 | 2,421,915 | 2,649,484 |
| Staves.......... „ | 14,604 | 15,915 | 54,758 | 75,864 |
| Mahogany ..... Tons | 9,760 | 6,925 | 84,590 | 58,337 |
| Other articles....Value £ | .. | .. | 1,047,333 | 977,803 |
| Total value ....... | .. | .. | 5,463,168 | 5,706,649 |

Besides the above, drugs to the value of 64,180*l*. were imported, as against 55,177*l*. in September 1897.

## Exports of Metals (other than Machinery) for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 8,905 | 8,709 | 39,468 | 37,363 |
| Copper :— | | | | |
| Unwrought.... „ | 33,635 | 43,661 | 86,413 | 117,808 |
| Wrought ...... „ | 30,125 | 23,059 | 92,526 | 73,999 |
| Mixed metal... „ | 22,398 | 15,382 | 52,805 | 37,220 |
| Cutlery .........Value £ | .. | .. | 168,490 | 49,910 |
| Hardware ....... „ | | | | 171,143 |
| Implements ..... „ | .. | .. | 115,273 | 107,765 |
| Iron and steel ... Tons | 283,559 | 269,631 | 2,013,675 | 1,765,644 |
| Lead ............ „ | 3,224 | 3,883 | 43,948 | 54,481 |
| Plated wares ... Value £ | .. | .. | 35,680 | 39,788 |
| Telegraph wires . „ | .. | .. | 46,127 | 35,411 |
| Tin ............. Cwt. | 9,669 | 8,365 | 31,583 | 31,578 |
| Zinc ............. „ | 11,329 | 12,181 | 9,523 | 11,033 |
| Other articles .. Value £ | .. | .. | 90,373 | 84,005 |
| Total value ....... | .. | .. | 2,825,884 | 2,567,238 |

## Exports of Drugs and Chemicals for Month ending 30th Sept.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 388,595 | 309,733 | 99,083 | 85,987 |
| Bleaching materials „ | 110,416 | 82,092 | 34,483 | 22,418 |
| Chemical manures Tons | 38,881 | 54,108 | 162,453 | 207,591 |
| Copper sulphate .. „ | 1,148 | 377 | 18,236 | 6,134 |
| Medicines....... Value £ | .. | .. | 91,704 | 89,675 |
| Other articles... „ | .. | .. | 230,419 | 260,433 |
| Total value .......... | .. | .. | 636,378 | 672,238 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH SEPT.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Gunpowder | Lb. | 769,400 | 1,331,500 | 21,429 | 29,079 |
| Military stores | Value £ | .. | .. | 71,862 | 205,277 |
| Candles | Lb. | 1,947,000 | 2,042,100 | 26,625 | 28,005 |
| Caoutchouc | Value £ | .. | .. | 112,486 | 118,644 |
| Cement | Tons | 32,134 | 26,734 | 55,164 | 51,507 |
| Products of coal | Value £ | .. | .. | 103,119 | 101,058 |
| Earthenware | „ | .. | .. | 126,111 | 124,810 |
| Stoneware | „ | .. | .. | 13,332 | 16,542 |
| Glass:— | | | | | |
| Plate | Sq. Ft. | 133,902 | 166,681 | 7,291 | 8,817 |
| Flint | Cwt. | 7,822 | 7,933 | 19,069 | 19,916 |
| Bottles | „ | 61,473 | 67,350 | 30,213 | 31,459 |
| Other kinds | „ | 19,348 | 19,864 | 13,807 | 18,041 |
| Leather:— | | | | | |
| Unwrought | „ | 13,043 | 12,860 | 106,949 | 113,215 |
| Wrought | Value £ | .. | .. | 39,931 | 42,092 |
| Seed oil | Tons | 3,417 | 2,817 | 62,332 | 50,234 |
| Floorcloth | Sq. Yds. | 1,664,700 | 1,728,000 | 75,787 | 77,586 |
| Painters' materials | Val. £ | .. | .. | 118,041 | 138,735 |
| Paper | Cwt. | 80,354 | 75,103 | 118,751 | 115,181 |
| Rags | Tons | 4,459 | 4,435 | 23,598 | 23,134 |
| Soap | Cwt. | 60,795 | 63,175 | 61,300 | 65,733 |
| Total value | | .. | .. | 2,525,220 | 2,790,342 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

19,926. C. Hoy. Improvements in and relating to bottles for storing unstable salts or any other unstable substances or liquids. Filed Sept. 20. Post-dated July 15.

20,040. A. Flesch. Improved press for extracting liquid. Sept. 21.

20,116. T. M. Wightwick. Improvements in thermometers. Sept. 22.

20,135. R. Frentzel.—From L. Tralls, Austria-Hungary. Improvements in and connected with drying apparatus. Sept. 23.

20,203. C. D. Abel.—From M. Marquardt and E. Höffner, Germany. Improvements in apparatus for superheating steam. Sept. 23.

20,215. W. Curtlidge and W. Wade. Improvements in the construction of kilns. Sept. 24.

20,232. J. S. Lethem. Improvements in or relating to furnaces. Sept. 24.

20,509. E. W. Lucas. Improvements in or relating to condensing apparatus. Sept. 28.

20,677. A. de Doete. Apparatus for measuring the colour intensity of coloured liquids and of transparent solid matter. Sept. 30.

20,785. E. Koenig and M. L. Erlanger. New or improved aerating apparatus. Complete Specification. Oct. 3.

28,875. A. J. Boult.—From O. D. Orvis, United States. Improvements in or relating to furnaces. Complete Specification. Oct. 4.

21,003. H. M. Maltby and F. Maltby. Improved furnace for melting glass, tin, lead, and similar substances. Oct. 5.

21,049. E. Rowland. Improvements in thermometers. Oct. 6.

21,081. H. G. C. Thofehrn. Improved receiver for liquefied gas, or gas under pressure. Oct. 6.

21,085. H. G. C. Thofehrn. Improvements in the process of liquefying gases, and apparatus therefor. Oct. 6.

21,181. W. H. Barr. Improvements in the method of and means for filtering, drying, deodorising and disinfecting steam and other gases. Oct. 8.

21,274. A. Dervaux. A new process of and means for cleansing filter material. Oct. 10.

21,300. E. Shaw. Improvements in apparatus for cooking, concentrating and evaporating liquids. Oct. 10.

21,310. A. Abendroth. Improvements in apparatus for mixing liquids and gases. Oct. 10.

21,398. G. H. Zeal. Improved maximum and minimum thermometer. Oct. 11.

21,470. C. F. Hughes-Hallett. Improvements in means for heating or cooling liquids. Oct. 12.

21,499. H. Winckler. Improvements in and connected with machines for drying fish, offal, stable, town, and slaughter-house refuse, and other material and substances requiring similar treatment. Oct. 12.

21,522. J. Prince. Improvements in Newcastle kilns. Oct. 13.

21,537. C. Heap and T. Oddy. Improved apparatus for condensing gases particularly applicable for liquefying air. Oct. 13.

21,773. J. M. Munyon. Improvements in atomizers. Complete Specification. Oct. 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

22,779. W. Dalton. Improved scientific sanitary filter. Oct. 12.

23,054. H. Higgins. Maintenance and regulation of heat in distillation, refining, chemical manufacturing, processes, and the like. Oct. 5.

23,339. P. R. Allen and J. B. Chadwick. Apparatus for evaporating or concentrating liquids. Sept. 28.

25,084. P. Hawkins, H. Hawkins, and S. H. Hawkins. New or improved hydrogen gas generator. Oct. 12.

25,898. E. Beckton. A portable spout to attach to drums containing ammonia or other strong or obnoxious chemicals. Oct. 12.

26,525. J. T. Bentley. Apparatus for cooling liquids. Oct. 19.

26,615. J. Weck. Sterilising apparatus. Oct. 12.

28,908. H. H. Lake.—From Manhattan Concentrator Company, United States. Machines for separating substances of different specific gravities. Oct. 5.

29,178. J. G. Lorrain.—From C. S. Ruckstuhl, United States. A new or improved thermometer. Oct. 19.

1898.

14,783. G. W. Simpson. Maximum thermometers. Oct. 12.

15,331. H. W. Abbott. Process and apparatus for refrigerating and cooling air. Oct. 5.

17,084. C. Maquet. Heating apparatus. Oct. 19.

17,259. M. Otto. A titrating and mixing apparatus especially applicable to the distribution and regulation of mixtures of ozone and air. Oct. 19.

17,523. M. Otto. Meter for apparatus for measuring and registering liquids and gases. Sept. 28.

17,524. M. Otto. Improved colorimeter, more especially applicable for use as an ozonometer. Sept. 28.

## II.—FUEL, GAS, AND LIGHT.

### APPLICATIONS.

19,803. J. B. de Alzugaray. Improvements in and connected with the manufacture of filaments for electric lamps. Sept. 19.

19,845. C. S. McIntire. Improvements in apparatus for generating acetylene gas. Sept. 19.

20,122. B. H. Wallin and Baron R. de Wendel. Improvements in apparatus for generating acetylene gas. Sept. 22.

20,127. L. Jacot.—From E. Pellaton, Switzerland. Improvements relating to the manufacture of acetylene gas. Sept. 22.

20,206. E. Andreoli. Improvements in ozonisers. Sept. 23.

20,273. A. J. Boult.—From E. Verbeke, Belgium. Improvements in or relating to Bunsen burners. Complete Specification. Sept. 24.

20,342. C. de Ricaris. The manufacture of a carburet compound and of an inflammable gas therefrom, and apparatus therefor. Sept. 26.

20,366. C. Weygang. Improvements in or relating to apparatus for use in burning liquid fuel. Sept. 26.

20,411. W. H. Akester. Improved compositions for kindling or causing ignition. Sept. 27.

20,430. G. Dillberg. An improved acetylene gas generator. Complete Specification. Sept. 27.

20,451. J. Dymond. Improvements in or relating to gas producers. Sept. 27.

20,468. B. H. Thwaite and H. Allen. Improvements in apparatus for generating acetylene gas from calcic carbide. Sept. 28.

20,517. H. R. Romney, J. Thame, and The Fibrous Materials Syndicate, Ltd. Improvements in and relating to the manufacture of fuel. Sept. 28.

20,541. F. B. Poerschmann and H. Schnieder. Improvements in acetylene generators. Complete Specification. Sept. 28.

20,597. C. Weygang. Improvements in or relating to apparatus for use in burning liquid fuel. Complete Specification. Sept. 29.

20,626. G. W. B. Crees. An improved generator of acetylene. Sept. 30.

20,646. R. Abeles. Improvements in carbide containers and in containers for acetylene gas apparatus. Complete Specification. Sept. 30.

20,740. W. P. Thompson.—From O. Lenz, Germany. Improvements in and in the manufacture of incandescent mantles. Complete Specification. Oct. 1.

20,766. W. Forrester. An improved acetylene gas generator. Oct. 3.

20,837. A. G. Smith. An improvement on acetylene gas generators. Oct. 4.

20,839. T. Rosethorn. Improved method of heating liquids. Oct. 4.

20,851. W. J. Ginder. Improvements in acetylene lamps or generators. Oct. 4.

20,879. C. B. Forward and J. M. Davidson. A process of refining and treating crude petroleum and of producing asphalt from petroleum and its products. Complete Specification. Oct. 4.

20,936. G. Wiltshire. Improvements in apparatus for producing acetylene gas. Oct. 5.

20,995. H. Colberg, A. Thompson, and J. Lion. Improvements in or relating to apparatus for generating acetylene gas. Oct. 5.

20,996. H. H. Lake.—From R. Türr, France. Improvements in apparatus for the production of acetylene gas. Oct. 5.

21,038. J. McConechy. Improvements in apparatus for the production of acetylene gas. Oct. 6.

21,067. F. Brown. Improvements in or relating to apparatus for use in the production of oxygen gas. Oct. 6.

21,242. S. Troubel. Improvements in acetylene gas generators. Oct. 8.

21,456. F. Smith. Improvements in burners for incandescent gas lighting. Oct. 12.

21,462. W. Foster. Improvements in and relating to the stoppers used in gas retorts. Oct. 12.

21,564. F. J. Glaholm. Improvements in acetylene gas apparatus. Oct. 13.

21,601. G. Daubenspeck. Improvements in incandescent mantles. Oct. 14.

21,643. W. Doman. Improvements in apparatus for the generation of acetylene gas. Oct. 14.

21,651. G. Chelmisky. Improvements in spray producers for petroleum, naphtha, and like furnace grates. Complete Specification. Oct. 14.

21,678. E. Festner and G. Hoffmann. Improvements in or relating to coke ovens. Complete Specification. Oct. 14.

21,691. D. J. V. Praag and F. W. Harker. Improvements in acetylene generating apparatus and the like. Oct. 14.

21,760. Count A. D. de Micheroux. An improved apparatus or oven for the production of coke and lighting gas with recovery of the by-products Complete Specification. Oct. 15.

### Complete Specifications Accepted.

#### 1897.

21,833. F. S. D. Scott and T. Hawkins. Generation of gas for motive power, illuminating, heating, and other purposes. Sept. 28.

22,648. J. A. Ageron and L. P. Wirth. Apparatus for generation and combustion of acetylene gas. Oct. 5.

23,547. A. Wartenweiler and R. Spengler. Automatic apparatus for the production of acetylene gas. Oct. 19.

23,793. C. J. Lundström. Preservation of carbide and its subsequent treatment for use in the production of gas. Oct. 19.

23,977. W. Johnson. Improvements on apparatus for the production and use of acetylene gas. Oct. 19.

25,592. The Acetylene Light Syndicate, Limited, and J. Gore. Apparatus for the manufacture of acetylene gas. Oct. 12.

26,202. A. Martini. Incandescent mantles. Sept. 28.

27,300. J. Reibel. Devices for feeding the carburet in acetylene gas generating apparatus. Oct. 12.

27,360. J. Hopwood and C. Cummins. An improved fuel. Oct. 5.

27,869. J. Bouring. Manufacture of coke and apparatus therefor. Sept. 28.

28,031. E. A. Wood. Incandescent gas burners. Oct. 5.

28,264. Sir H. Grubb, Knight. Apparatus for the generation of acetylene gas. Oct. 19.

#### 1898.

11,964. C. Bollé. From P. Dreske, Germany. Apparatus for generating acetylene. Oct. 5.

14,049. K. E. Rosenthal. Generators for acetylene gas. Oct. 12.

14,050. J. Billwiller and K. E. Rosenthal. Generators for the production of acetylene gas. Sept. 28.

17,079. F. Geblert. Apparatus for producing acetylene gas. Oct. 19.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Applications.

19,849. W. Schrüder. Process for obtaining yellow prussiate of potash as a by-product in the destructive distillation of coal. Sept. 19.

20,086. J. Armour and F. Alison. An improved method of and apparatus for heating paraffin scale. Complete Specification. Sept. 22.

## IV.—COLOURING MATTERS AND DYES.

### Applications.

20,063. B. Willcox.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture and production of new colouring matters and of new intermediate products relating thereto. Sept. 21.

20,881. A. Wilkie.—From J. K. Wilkie, British North Borneo. Improvements in or relating to methods of or apparatus for the extraction of cutch and the like. Oct. 4.

20,947. W. H. Claus, A. Rée, and L. Marchlewski. Production or manufacture of new polyazo colouring matters. Oct. 5.

21,705. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of anthraquinone dyestuffs. Oct. 6.

21,157. B. Willcox.—From The Badische Anilin and Soda Fabrik, Germany. The manufacture of indigo leuco products and colouring matters and of intermediate compounds relating thereto. Oct. 7.

21,415. J. Imray.—From La Société L. Durand, Huguenin et Cie., Germany. Manufacture of leuco derivatives of gallocyanins. Oct. 11.

21,496. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture of yellow basic colouring matters. Oct. 12.

21,596. O. Imray. — From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of acid dyestuffs of the diphenylnaphthylmethane series. Oct. 13.

### Complete Specifications Accepted.

#### 1897.

21,968. I. Levinstein and Levinstein, Ltd. Production or manufacture of aromatic aldehydes and of colouring matters therefrom. Oct. 5.

25,136. S. Pitt.—From L. Cassella and Co., Germany. Producing diamido-oxydiphenyl and like bases. Oct. 19.

25,234. S. Pitt.—From L. Cassella and Co., Germany. Production of direct-dyeing black cotton dyestuffs. Oct. 12.

25,756. O. Imray.—From The Fabriques de Produits Chimiques de Thann et Mulhouse, Alsace. Manufacture of para- and meta-nitro-anisidine, and of colouring matters therefrom. Oct. 5.

27,051. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Manufacture of new colouring matters. Oct. 5.

28,604. H. H. Lake.—From Farbwerke Mülheim vormals A. Leonhardt and Co., Germany. Manufacture of colouring matters. Oct. 12.

#### 1898.

18,477. O. Imray.—From F. Fritzsche and Co. Manufacture of dyestuffs of the phthaleine series. Oct. 19.

## V.—TEXTILES : COTTON, WOOL, SILK, Etc.

### Applications.

19,935. W. Joynt and A. J. Summerfield. Improvement in the treatment of ramie plants, rhea grasses, and fibrous plants as a substitute of cotton, wool, silk, and the like. Sept. 20.

20,678. W. Erben. An improved process of extracting grease from wool by naphtha or like solvent, and apparatus for carrying out said process. Sept. 30.

### Complete Specifications Accepted.

#### 1897.

21,801. W. G. Thomson. Method of and apparatus for manufacturing inlaid linoleum and other floorcloths. Sept. 28.

25,468. W. P. Ingham. Manufacture of slag wool. Oct. 5.

29,072. J. Y. Johnson.—From W. H. Drury, United States. Manufacture of yarns, and apparatus therefor. Oct. 5.

29,842. T. B. Powell. Coating textile fabrics and other materials. Oct. 19.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

### Applications.

19,858. S. Khrènnikoff. Improvements in apparatus for boiling, bleaching, and dyeing cotton and the like fabrics. Sept. 19.

20,301. F. A. Hartmann.—From C. Dreher, Germany. *See* Class XIV.

20,562. A. Marr. Improvements in apparatus for dyeing, bleaching, drying, and similarly treating yarn in cops and the like. Sept. 29.

20,670. C. D. Abel.—From the firm of H. C. Meyer, jun., Germany. Improved process for bleaching cane. Sept. 30.

20,780. J. Tait. *See* Class XIX.

20,865. J. Grossmann. A new or improved indigo vat. Oct. 4.

21,108. F. A. Boyes. Improvements in means for conducting or guiding fabrics when under treatment in bleaching and like processes. Oct. 7.

21,575. G. W. Johnson. From Kalle and Co., Germany. Improvements in dyeing black with logwood. Oct. 13.

21,645. A. G. Brookes.—From J. F. Monaghan, United States. Improvements in kiers for bleaching or the like. Oct. 14.

### Complete Specifications Accepted.

#### 1897.

24,506. S. Musgrave, H. Musgrave, and J. T. Musgrave. Method of and process for obtaining iridescent colours upon paper or other suitable material. Oct. 5.

27,937. T. Ingham. An improvement in dyeing. Oct. 5.

28,090. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. A new process for producing black shades upon cotton. Oct. 12.

## VII.—ACIDS, ALKALIS, AND SALTS.

### Applications.

19,907. A. J. Boult.—From A. F. Izidore and M. Hiscots, France. Improvements in or relating to the recovery of nitrous gases in the manufacture of sulphuric acid. Sept. 20.

20,050. A. Gutensohn. A new or improved method of and apparatus for producing basic cupric acetate. Complete Specification. Sept. 21.

20,087. B. Piffard. Improvements in the manufacture of gaseous ammonia. Sept. 22.

20,356. S. Pitt.—From G. Laureau, Son, and Co., France. The preservation and treatment of kelp for the extraction of iodine and of gelatinous matter, and the employment of the latter for waterproofing fabrics, paper, leather, wood, and the like. Sept. 26.

20,357. S. Pitt.—From G. Laureau, Son, and Co., France. The extraction and utilisation of gelatinous matter and cellulose from marine plants, the treatment of the liquors obtained, and the apparatus employed therefor. Sept. 26.

20,415. C. Dreyfus and A. G. Green. Improvements in apparatus and vessels for the manufacture and use of acetic acid. Sept. 27.

20,681. R. W. E. MacIvor and T. F. Spencer. *See* Class X.

20,744. W. R. Ormandy. Improvements in the treatment of kaolin, sulphates of alumina and the alkalis, sand, and other substances containing iron, and in obtaining certain compounds of iron. Oct. 1.

21,126. J. Grossmann. Improvements in the preparation and manufacture of hydrosulphurous acid and hydrosulphites. Oct. 7.

21,299. F. B. Felt. Improvements in retorts and method for the subjection of liquid substances to the action of air and other reagents, more especially for the treatment of manganate of soda or other alkali. Complete Specification. Oct. 10.

21,616. A. Séhillot. An improved process of manufacturing sulphuric acid. Complete Specification. Oct. 14.

21,653. T. Macalpine. An improved method for the production of oxalic acid and of oxalates. Oct. 14.

21,657. T. C. Lowden. Improvements in or relating to the manufacture of salt, and apparatus therefor. Complete Specification. Oct. 14.

### Complete Specifications Accepted.

1897.

22,395. W. Garroway. Manufacture of lead carbonate, ammonium nitrate, silicates of sodium, and caustic soda. Sept. 14.

24,525. O. Imray.—From W. T. Gibbs, Canada. Manufacture of potash salts. Sept. 28.

26,176. J. C. Mewburn.—From Chemische Fabrik von Heyden Gesellschaft mit Beschränkter Haftung, Germany. Production of halogen compounds of silver. Oct. 12.

27,221. A. R. Scott. Improvements in obtaining chlorine. Oct. 19.

1898.

14,269. C. E. Acke. Manufacture of caustic alkalis from alloys of lead with the alkaline metals. Oct. 19.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### Applications.

20,100. C. H. Temple. A process or method of printing designs upon tiles and other clay ware from a photographic half tone process block or plate. Sept. 22.

20,616. W. P. Sauerland. An improved circular regenerative furnace for glass founding. Complete Specification. Sept. 29.

21,001. H. Sturm. Improvements in furnaces and drying apparatuses having a movable bed for ceramic and other products. Oct. 5.

21,202. W. Haley. Improvements in the method of and means for moulding seamless and other glassware. Complete Specification. Oct. 8.

21,346. C. Dörr. An improved plastic and hardenable substance and a process for its manufacture. Complete Specification. Oct. 11.

21,559. C. T. Hill and F. W. Williams. Improvements in or in connection with press cloths for use in the preparation or treatment of pottery clay. Oct. 13.

1898.

10,807. J. T. Newell. Treatment of glass for coating purposes as a backing for slabs, tiles, facing plates, and the like, and for light reflection and other purposes. Sept. 28.

17,209. T. Pfister and E. Barthels. Cloisonné work. Sept. 28.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

19,888. F. Higgarty. An improved flooring or paving composition. Sept. 20.

20,538. W. L. Allcroft. An improved floor covering. Sept. 28.

20,679. P. Davies. Improvements in and relating to the manufacture of Portland and like cement. Sept. 30.

20,831. J. Clarke, T. G. Stevens, and J. J. Hewitt. Improvements in the manufacture of cement. Oct. 3.

20,952. J. Richardson. Improvements in and relating to fireproof buildings. Oct. 5.

21,073. C. D. Abel.—From C. Westphal, Germany. Improved process for hardening artificial stone. Oct. 6.

21,231. J. Armour. Improvements in the manufacture of building materials. Oct. 8.

21,533. T. W. James. Imitation mica-marble and stone. Oct. 13.

21,584. E. Homan. Improvements in or connected with fireproof floors, ceilings, or the like. Oct. 13.

### Complete Specifications Accepted.

1897.

18,246. F. D. Cummer. Means applicable for use in the manufacture of cement. Oct. 12.

23,993. C. G. J. Frosell. Composition applicable for cement, artificial stone, and the like. Oct. 5.

29,020. F. S. Smith. Construction of bricks and other moulded building materials. Oct. 19.

29,263. C. Hannay. Improvements in firebricks, slabs, or tiles. Oct. 19.

1898.

16,173. O. Imray. — From La Société Anonyme des Ciments et Plâtres de Vilvorde à Vilvorde, Belgium. Manufacture of Portland cement. Sept. 28.

## X.—METALLURGY, MINING, Etc.

### Applications.

19,804. J. B. de Alzugaray. Improvements in extracting metals and oxides from ores and salts. Sept. 19.

19,831. W. H. Harrap. Improvements in alloys. Sept. 19.

19,964. J. L. Grant.—From W. Scott, Canada. Improvements in amalgamating apparatus for separating gold or other precious metals from auriferous or other material. Sept. 20.

20,283. C. Brand. An improvement in moulds for casting slag blocks. Complete Specification. Sept. 24.

20,328. G. Bennett. Improvements in steel ingot moulds. Complete Specification. Sept. 26.

20,450. O. Elstner. A process for the preparation of metallic oxides. Sept. 27.

20,681. R. W. E. MacIvor and T. F. Spencer. Improvements in the manufacture of sesquioxide of chromium direct from chrome ore. Sept. 30.

21,022. S. P. Bowen. Improvements in bogies for molten blast furnace slag and the like. Oct. 6.

21,138. W. T. Whiteman.—From E. Petersson, Belgium. An improved process for the treatment of sulphurous ores containing arsenic, antimony, or tellurium. Oct. 7.

21,302. H. P. Pease. Fuel-ore blocks or bricks for smelting. Oct. 10.

21,306. S. Pitt.—From The Pharmaceutisches Institut Ludwig Wilhelm Gans, Germany. Improvements in the manufacture of metal leaf. Oct. 10.

21,376. L. Pszczolka and R. M. Daelen. An improved process for effecting the preliminary refining of pig-iron. Oct. 11.

21,387. R. B. Thomas and H. S. Thomas. Improvements in the manufacture of tin and terne plates and other like metal coated plates or sheets, and in apparatus employed in the said manufacture. Oct. 11.

21,395. J. B. de Alzugaray. Improvements in or relating to the extraction of gold, silver, and other metals from ores and the like. Oct. 11.

21,418. W. Wilkinson. Improvements in the method of casting and stamping aluminium and other metal articles. Oct. 11.

21,552. The Coombes Dry Mining Syndicate, Ltd., and J. A. Coombes. Improvements in the method of and in apparatus for recovering gold from gold-bearing materials. Oct. 13.

21,671. A. K. Huntingdon and R. T. Preston. A process for superficially alloying metal. Oct. 14.

21,772. S. O. Cowper-Coles. Improvements in the treatment of refractory ores. Oct. 15.

### Complete Specifications Accepted.

1897.

23,267. J. E. Bryant. Apparatus for the electro-deposition of one metal on another. Oct. 19.

24,267. P. E. Secrétan. Alloy of aluminium, and process for making the same. Sept. 28.

27,015. J. E. Dowson. Process relating to the reduction of ores, and improvements in furnaces therefor. Oct. 19.

27,881. F. D. James and M. B. Fox. An improved metal, and method of producing it. Oct. 19.

1898.

5943 S. O. Cowper-Coles. Treatment of zinc-bearing ores for obtaining zinc and other metals therefrom. Oct. 5.

12,267. P. Rogers and J. Player. Machinery or apparatus for coating metal plates or sheets with tin, lead, or other metals or alloys. Oct. 5.

18,169. D. C. Ripley. Manufacture of glassware. Oct. 19.

## XI.—ELECTRO-CHEMISTRY and ELECTRO-METALLURGY.

### Applications.

19,855. H. H. Lake.—From La Société Roger and Ratbier, France. Improvements in the manufacture of electrodes for electric accumulators. Complete Specification. Sept. 19.

19,876. O. J. Steinhart, J. L. F. Vogel, and H. E. Fry. Improvements in the electrolytic separation of zinc from zinc oxide. Sept. 19.

19,877. O. J. Steinhart, J. L. F. Vogel, and H. E. Fry. An improved cell for the electrolysis of fused metallic chlorides. Sept. 19.

19,878. O. J. Steiohart, J. L. F. Vogel, and H. E. Fry. Improvements in the manufacture of anhydrous zinc chloride. Sept. 19.

20,029. W. S. Rawson. Improvements in electric batteries. Sept. 21.

20,919. R. J. Crowley, S. S. Bromhead, and H. Fairbrother. An improved electrolyte for primary batteries. Oct. 4.

20,956. L. Pirwitz and W. Behr. Improved non-conducting material or composition and process for the production of same. Complete Specification. Oct. 5.

21,169. R. Haddan.—From Columbus Electrizitats Gesellschaft (Limited Co.), Germany. Improvements in electric batteries. Oct. 7.

21,233. J. Thornton and T. R. Canning. Improvements in apparatus for coating metal articles by electro-deposition. Oct. 8.

21,248. S. O. Cowper-Coles. Improvements in and connected with with the electro deposition of metals. Oct. 10.

21,307. S. Pitt.—From The Pharmaceutisches Institut Ludwig Wilhelm Gans, Germany. Improvements in the production of incandescent materials suitable for use in electric lighting. Oct. 10.

21,361. H. Blumenberg, jun. Improvements in battery solutions. Oct. 11.

21,695. E. A. Stokes, E. J. Stokes, and R. J. Crowley. A circulating electric battery. Oct. 15.

### Complete Specifications Accepted.

1897.

17,316. L. G. Garcia. Improvements in accumulators Sept. 28.

24,051. R. Fabian. Electrodes for primary and secondary batteries in chemical cells. Oct. 5.

24,884. A. F. Harris. Improvements in electro-deposition apparatus. Oct. 12.

25,246. H. von der Linde. Process for the electrolytic separation of metals. Sept. 28.

28,242. P. J. R. Dujardin. Negative plates for secondary batteries. Oct. 5.

1898.

8226. L. P. Ford and T. W. Barber. Manufacturing artificial stone and concrete. Aug. 24.

11,604. E. Stassano. Electro-metallurgic production of iron, steel, and their alloys with chromium, tungsten, nickel, manganese, and the like. Oct. 12.

15,222. H. H. Lake.—From J. Burns, France. Improvements in electric accumulators. Sept. 28.

15,430. W. P. Thompson.—From E. Langguth, Germany. Diaphragm for electrolytic apparatus. Oct. 5.

16,129. O. Imray.—From Oesterreichischer verein für Chemische und Metallurgische Production, Bohemia. Electrolysis of solutions of alkali salts, especially alkali chlorides. Oct. 19.

17,583. O. T. Bugg, jun., and D. H. Darling. Electric batteries. Oct. 12.

## XII.—FATS, OILS, and SOAP.

### Applications.

20,058. H. Hampel and V. Zampis. An improved method of preparation of a rosin soap for paper sizing, containing an admissible quantity of free rosin. Complete Specification. Sept. 21.

20,649. W. R. Harrison and E. Stephenson. Improvements in the method of and means for removing superfluous moisture from meal or other residuum left after the extraction of oil from seed or other oleaginous substances by volatile solvents, and for preventing the fine particles of such meal or residuum escaping from the extractor. Sept. 30.

20,650 W. R. Harrison and E. Stephenson. Improvements in the method of and apparatus for drying, screening, cooling, and bagging the meal or other residuum left after the extraction of oil from seeds or other oleaginous substances by volatile solvents. Sept. 30.

20,967. R. E. Green. Improvements in the production of glycerine. Oct. 5.

21,369. R. Adam. An improved soap compound which can also be used as a combustible deodorizer and process for the manufacture of the same. Complete Specification. Oct. 11.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

19,869. C. D. Abel.—From H. C. Meyer, jun., Germany. Process for the treatment of resinous cane for the removal of the resin therefrom. Sept. 19.

20,037. R. Lokesch. An improved method of making imitation enamels. Complete Specification. Sept. 21.

20,044. J. Deborde. Improvements in or relating to artificial rubber. Sept. 21.

20,223. J. Bishop. Bishop's patent French polish. Sept. 24.

21,156. L. E. Josset. Improvements in and in connection with luminous paint. Oct 7.

21,326. W. G. Williams and S. Taylor. Improvements in the preparation of dry colours. Oct. 11.

### Complete Specifications Accepted.

1897.

22,792. L. Hunt. New or improved non-poisonous and permanent white paint or varnish. Oct. 12.

1898.

15,569. O. Zürcher. Processes for extracting caoutchouc. Oct. 12.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Application.

19,931. J. H. Goodwin. Improvements in material for leather and other belting. Sept. 20.

20,301. F. A. Hartmann.—From C. Dreher, Germany. Dyeing of leather a fast yellow. Sept. 26.

20,444. P. Emperanger and A. Huillard. An improved rotary barrel for use in rapid tanning. Complete Specification. Sept. 27.

20,680. A. Rainard. Improvements in tannic extracts and in apparatus for the manufacture thereof. Sept. 30.

21,064. A. J. Boult.—From P. A. D. Prost and A. Godard, France. Improved tanning and finishing process for leather and skins. Oct. 6.

21,663. J. L. Planckaert and F. Vuylsteke. A new or improved process for tanning rabbit and other skins. Complete Specification. Oct. 14.

### Complete Specifications Accepted.

1897.

25,324. P. Leyendecker. Machine for treating hides and skins. Oct. 5.

26,677. W. F. Reid and E. J. V. Earle. Manufacture of enamelled leather. Oct. 19.

1898.

5003. G. Fettweis. Tanning of skins and the like, and apparatus therefor. Oct. 12.

16,369. J. H. Curling. Tanning hides and skins. Oct. 19.

16,419. S. Thomson and J. A. Todd. Method of tanning hides and skins. Oct. 5.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

19,957. E. Shaw. Improvements in apparatus suitable for preparing syrup for use in the manufacture of sweetmeats. Sept. 20.

19,961. W. P. Thompson.—From H. A. J. Manoury, France. Improved method of refining raw sugar. Sept. 20.

20,900. W. P. Thompson.—From K. Koch, Russia. Improvements in or relating to the manufacture of cube or loaf sugar. Complete Specification. Oct. 4.

### Complete Specification Accepted.

1897.

24,3[illegible]3. H. Machin, of the firm of Batger and Co.—From E. A. Dix, Russia. Manufacture of sugar, and apparatus therefor. Oct. 19.

24,593. D. Loiseau. Processes of purifying and clarifying sugary products in sugar houses, sugar, molasses refineries, refineries, or factories of glucose, and the like. Oct. 19.

## XVII BREWING, WINES, SPIRITS, Etc.

### Applications.

20,238. J. Hall. Steam circulating arrangement and apparatus for heating brewing pans and the like, and returning the water of condensation to boiler. Sept. 24.

21,214. H. Finlay. Improvements in the manufacture or treatment of yeast or barm. Oct. 8.

21,750. M. H. C. Shann and F. M. Johnson. Improvements in or relating to the fermentation of liquids. Oct. 15.

### Complete Specifications Accepted.

1897.

22,846. A. D. Watson. An improved process for the extraction of the plasma (cell contents) and the separation of cellulose from yeast. Oct. 5.

26,532. J. T. H. Gronwald. Preserving beer. Oct. 19.

1898.

17,159. F. R. Coudert, jun. Manufacture of ethyl alcohol. Oct. 19.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

*A.—Foods.*

20,448. E. de Vevey. Improvements relating to the preservation of milk. Sept. 27.

20,992. N. S. W. K. Buchtrup. Improved method of preserving milk or cream. Complete Specification. Oct. 5.

21,284. H. Y. Castner. Improved method of purifying salt. Oct. 10.

### Complete Specifications Accepted.

1897.

21,387. F. H. W. Graeff. Preservation of organic substances. Sept. 28.

22,686. L. Wacker. Process and apparatus for preserving meat. Oct. 12.

26,292. R. R. Bell and A. C. Butler. Improvements in and relating to tea essences. Oct. 19.

1898.

12,612. F. Crognet. Process for obtaining sterilised raw meat in the condition of powder. Oct. 5.

12,869. N. Bendixen. Method of and apparatus for sterilising milk. Oct. 5.

18,500. M. Poppe. Process for the production of margarine or artificial butter. Oct. 19.

*B.—Sanitation.*

20,118. R. H. Reeves. Improved method of effecting the purification of sewage effluents and in appliances employed therewith and therefor. Sept. 22.

20,181. C. Andrews. A new or improved method of treating sewage. Sept. 23.

20,620. W. R. Hutton, jun. Process for the utilisation of precipitated sewage sludge. Sept. 30.

21,199. S. Howard. The "Howard Destructor" for the treatment of sewage. Oct. 8.

21,288. W. J. Wright. Improved water softening and purifying apparatus. Oct. 10.

21,289. W. J. Wright. New or improved water heating and purifying apparatus. Oct. 10.

21,361. A. E. H. Lozé. Improved material for purifying, filtering, clarifying, and sterilizing liquids. Complete Specification. Filed, October 11. Date applied for, March 7, 1898, being date of application in France.

### Complete Specifications Accepted.

1897.

17,257. M. Otto. Process of and apparatus for the purification of water and other liquids. Oct. 5.

*C.—Disinfectants.*

21,369. R. Adam. *See* Class XII.

21,408. W. P. Thompson. — From J. G. Brissonnet, France. Improved antiseptic and disinfectant compound. Oct. 11.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

20,058. H. Hampel and V. Zampis. *See* Class XII.

20,780. J. Tait. Improvements in bleaching and purifying paper pulp. Oct. 3.

### Complete Specifications Accepted.

1897.

22,859. C. Indruweit. Improved process for the metallic coating of paper. Oct. 12.

1898.

7906. W. K. Trotman. Manufacture of paper. Oct. 19.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, AND EXTRACTS.

### Applications.

19,823. J. T. Hewitt. The manufacture of a new product from piperidine. Sept. 19.

19,868. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. The manufacture of an aromatic ether. Sept. 19.

20,081. P. Stern. Improvements in the preparation of corrosive sublimate for sale. Sept. 22.

20,192. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of aromatic mercury compounds. Sept. 23.

20,272. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of chemical compounds from acetylene. Sept. 24.

20,447. G. W. Johnson.—From C. F. Boehringer and Soehne, Germany. The manufacture and production of formic aldehyde compounds of uric acids and alkyluric acids. Complete Specification. Sept. 27.

20,801. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of new compounds containing halogen. Oct. 3.

21,471. W. Pip. A process for the preparation of piperidine and dihydrochinoline. Oct. 12.

21,756. O. Imray. — From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of a substance for raising the pressure of the blood. Oct. 15.

### Complete Specifications Accepted.

1897.

23,227. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Manufacture or preparation of hetroeroxanthine, paraxanthine, and methylated hypoxanthines. Oct. 19.

25,942. A. Classen. Manufacture of formaldehyde compounds of the proteïn bodies soluble in water. Oct. 12.

26,442. W. G. Whiffen. Preparation of a crystalline compound of emetine. Oct. 13.

27,032. H. J. H. Fenton and E. S. St. Barbe Sladen. Process for the production of dehydroxytartaric acid. Oct. 5.

## XXI.—PHOTOGRAPHY.

### Applications.

19,956. A. Werner. Improvements in natural colour photography. Sept. 20.

20,417 H. H. Lake.—From International Colour Photo Company, United States. Improvements relating to the production of coloured pictures by photography. Complete Specification. Sept. 27.

20,433. M. Luhn. Improvements in connection with the reproduction of so-called animated photographs. Sept. 27.

20,958. T. W. Barber. Improvements in apparatus for taking and exhibiting photographs in colour. Oct. 5.

21,595. C. D. Abel.—From The Actien Gesellschaft für Anilin Fabrikation, Germany. Improvements in photographic developers. Oct. 13.

21,649. W. Friese-Greene. Improvements in taking and in projecting photographic images in means therefor and in photographic negatives. Oct. 14.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

19,806. E. Hollings. Improvements in moulding or forming into blocks or the like gun-cotton or similar materials. Sept. 19.

20,102. E. Bainbridge. An improvement in percussion fuses. Complete Specification. Sept. 22.

20,358. C. A. McEvoy. Improvements in fuses for shells. Sept. 26.

20,449. C. Kastner. Improvements in or relating to matches. Sept. 27.

20,989. G. P. Bickford Smith. A new or improved composition for electrically firing detonators. Complete Specification. Oct. 5.

20,990. G. P. Bickford-Smith. An improvement in detonators for firing explosives. Complete Specification. Oct. 5.

21,141. C. A. Allison.—From P. Hathaway, United States. Improvements in matches and in the method of and apparatus for producing same. Complete Specification. Oct. 7.

### Complete Specifications Accepted.

1897.

21,136. A. Luck and C. L. Nichols. Manufacture and production of explosives. Sept. 28.

29,389. F. L. Thomas.—From La Société des Poudres et Dynamites, France. Improvement in explosive. Oct. 5.

1898.

3863. G. H. Hosie. Explosives and their use. Oct. 19.

## PATENTS UNCLASSIFIABLE.

### Applications.

20,645. E. K. Maussner. Fire- and water-proof material, and method and apparatus for producing the same. Sept. 30.

21,149. E. Milo and E. J. Richardson. Process for the manufacture of materials having the appearance of mother-of-pearl, malachite, marble, lapis lazuli, or other ornamental objects. Oct. 7.

21,606. R. Pritt. Improvements in a vegetable compound or pulp. Oct. 14.

### Complete Specification Accepted.

1898.

15,758. C. A. Allison. — From L. Silverman, United States. Obtaining products from the pith of corn-stalks, and utilising same. Sept. 28.

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*Meeting held on Wednesday, October 26th,* 1898.

## CHAIRMAN'S ADDRESS.

## THE PRESENT POSITION AND PROSPECTS OF THE CHEMICAL MANURE INDUSTRY.

BY ALFRED SMETHAM, F.I.C.

As you are all aware, the art of agriculture is as old as civilisation, and seems to have been conducted with more or less success throughout the ages. That it was brought to a high degree of perfection in many countries is patent to every one who has given the matter any thought or attention, but up to the year 1840 or so the methods of cultivation were entirely empiric. It is true that bones had been applied as manure, but in what the fertilising properties consisted was uncertain. Liebig, who for many years waged valiant war in support of his "mineral theory" of manuring, alleges in his "Letters on Chemistry" published in 1851, that the beneficial effects observed are due entirely to the phosphate of lime, and combats with vigour the early conclusions of Lawes that ammonia salts are of value as manure to cereal or other crops. Time, with its relentless accumulation of facts, has as completely routed the theories of Liebig as were those of Bastian overthrown by the classical work of Pasteur and his followers.

But notwithstanding his now proved erroneous ideas on the fixation of nitrogen by plants, agriculture owes a debt of deep gratitude to Liebig for the impetus which he gave to agricultural science by his investigations and by the concrete forms in which he embodied his thoughts, by which to a large extent he laid the lines in this, as in other subjects, on which future investigators were to travel. But he did more than this. As the result of pure science he gave to the world a new industry, which has continued to grow, until to-day it takes an important place among the chemical industries of this and other countries. I refer to the Chemical Manure Industry.

It was, I believe, in a communication to the meeting of the British Association at Bristol in 1836, that Liebig first propounded the possibility of converting the insoluble phosphate of bones into the soluble condition by transforming it into superphosphate by treatment with sulphuric acid. Prior to that date Messrs. T. and H. Proctor had started about the year 1812 to boil bones and chop them into small pieces for sale as turnip manure. About 1825 steam engines for grinding the bones were erected, and a much finer meal was by that means obtained. About the year 1835 it occurred to Mr. Thomas Proctor, jun., that a superior glue might be manufactured by dissolving out the phosphate of lime from bones by means of hydrochloric acid, and the process was successfully carried out on the practical scale. The glue obtained was of better colour than that previously manufactured, and the dissolved phosphate was precipitated by means of carbonate of lime and used as manure. About 1837 or 1838, Mr. Proctor had communications with Boussingault and Liebig, and gained from them the idea of treating bones with sulphuric acid; the first experiments being made with spent animal charcoal from the sugar refinery at Bristol. In 1839 he began to use sulphuric acid upon raw bones, and by adding some potash salts he made a manure which was called German Compost. In 1842, Mr. Proctor, in conjunction with Mr. Ryland, started the manufacture of bone superphosphate at Birmingham, and it was to the improvements introduced into the manufacture by the latter that the rapid rise of the industry was to a large extent attributable.

The step from bone to coprolites in the manufacture of superphosphate was, from a chemical point of view, not a great one, but from an economic standpoint it proved the pivot in the history of the manufacture. To Mr. Lawes (now Sir John, of Rothamsted fame) is due the credit of first pressing coprolites into service, and it may safely be said that to him, as the originator of the finest set of agricultural experiments the world has yet seen, and one of the pioneers of an industry which has done so much for British agriculture, past, present, and future generations, not only of agriculturists, but of the toiling millions of the towns have been and will be laid under obligations which even if the modesty of this "grand old man" of agriculture would permit, could never be adequately expressed.

Up to the time to which we are referring English agriculture had been to a large extent self contained, and although leases were framed to prevent as far as possible the sale of produce of the land, the necessary exportation of grain and meat to the towns involved a loss of the fertilising constituents of the soil which, in the absence of artificial manures or purchased feeding stuffs, could not be returned, and as a consequence the land in many parts of the country was getting exhausted. With a view to remedying this defect, many expedients were resorted to, amongst them marling. The ponds, or "pits" as they are called, which are so common in the surrounding country, and which add so much to the picturesqueness of the landscape, are in most instances old marl pits, and are constant mementoes of a class of farming which has given place to the higher cultivation of a scientifically controlled agriculture.

About the same time as Liebig gave to the world the more rapidly acting phosphate, there was introduced into England a manure called guano, from Peru. This, the dried excrements of sea birds deposited during centuries in a rainless district, produced such magical effects, especially with corn crops, that its fame spread like wildfire, and thus prepared the way for the more extended use of artificial manures. The price at which it was sold was high, but as the earlier importations contained nitrogen equal to over 18 per cent. of ammonia, existing chiefly as ammonia salts, and phosphoric acid equal to about 26 per cent. of phosphate of lime, the return on the outlay, notwithstanding the high price, was usually a very profitable one. But as we now know, nitrogen applied as manure is not equally effective with all crops, root and leguminous crops often giving a full yield when dressed with phosphates alone. Hence, although guano was much in favour, there were not wanting farmers who pinned their faith to dissolved bones and artificial manures which had mineral or bone superphosphate as a basis.

The native deposits of coprolites, found chiefly in Cambridgeshire and Suffolk, contained about 60 per cent. of tribasic phosphate of lime, and were admirably suited, when finely ground, to the manufacture of superphosphate. To this was usually added varying quantities of sulphate of ammonia, or some organic nitrogenous compounds, and the manures thus produced were sold as specific manures for different crops. Those were halcyon days for the manure manufacturer, which, in the keener competition of a more educated age, are, I fear, never to return.

The supply of bones being limited, the deposits of coprolites were largely drawn upon until in the early seventies the stores, which Liebig scarcely 20 years before had predicted would, if utilised, render England independent of any importations of wheat, were exhausted, and this notwithstanding the fact that there had been large importations of bone-ash and mineral phosphates from abroad.

To give a detailed description of the various forms of phosphatic materials which have been imported in such large quantities from all parts of the globe, the deposits of which have been in many instances exhausted, would prove interesting, but would be beyond the scope of my present endeavours. French, Spanish, and Canadian phosphates, once the mainstay of the manufacturers, have now given place to those from South Carolina, Florida, and Algeria, while the huge deposits of ammoniacal guanos from Peru became poorer and poorer in quality, until about ten years ago the importation was so small, and the quality so poor, that many farmers who had proved faithful to their first love, were compelled to give up its use, and to purchase other forms of manure. The same fate was in store for the large number of phosphatic guanos, of which Sombrero, Baker Island, Mejillones, Curaçao, and Malden Island were the chief.

And yet in spite of the great drain upon the natural supplies of phosphates, phosphatic materials have never been at so low a price as they have been during the past few years.

At present the chief point which determines the source from which our supplies are drawn, seems to be the cost of freight. In Florida, for instance, enormous deposits of rock and river phosphate of high quality exist, but the margin of profit on working is so small, or is a minus quantity, that many of the mines have ceased working, and all have limited the output. Notwithstanding the pessimistic views taken some 10 or 15 years ago as to the future supplies of phosphates there seems at present little fear of a phosphate famine for many generations to come, and glancing at the history of the industry during the past 50 years or so, I incline to the belief that, in spite of a constantly growing population, and a necessarily higher cultivation, the discovery of new deposits, and the conservation of our present supplies, will meet all the requirements of the case.

In making this statement, I have in mind the fact that in addition to the very widely-distributed deposits of the more concrete forms of phosphate before alluded to, phosphoric acid is present to a greater or less extent in every soil which I have examined, and often exists in larger quantities in foreign soils than in English. The average quantity of phosphoric acid in English soils, so far as my experience goes, is about 0·13 per cent. If, however, to keep on the safe side, we assume it to be only 0·10 per cent., that quantity, small as it is, represents for a depth of 9 inches a quantity equal to over 5 tons of bone meal per acre, or a total of 160,000,000 tons or so for the whole of the cultivated land of Great Britain alone. Taking the average loss per acre at 10 lbs. of phosphoric acid, a quantity somewhat above that on arable land, and decidedly above that on pastures, on the assumption that the produce of our farms sold at market is lost to the land, the store of phosphoric acid to a depth of 9 inches represents something like 300 years' supply, provided crops were able to glean the last particle, and still yield the same crops as at present. Last year the importations of guano, bones, and phosphate of lime amounted to 399,505 tons, or an importation of about 99,000 tons of phosphoric acid, or nearly 5 lb. per acre over the whole of the cultivated area of the United Kingdom. Against this deficiency of 5 lbs. of phosphoric acid per acre, there are importations of cattle and sheep, dead and alive, equal to nearly 8,000 tons per annum; there is a reserve store of unknown amount in subsoil phosphoric acid which deep-rooted plants like wheat and clover can draw upon; there is the concentrated phosphoric acid from the iron ores obtained by the Gilchrist Thomas' process, and sold in ever-increasing quantities as basic slag; there is a return in bones from our home produced cattle and sheep, and there is the not inconsiderable importation of phosphoric acid in the form of linseed cake, maize, wheat, and other feeding stuffs. With the figures at my disposal it is impossible to make anything like an accurate balance sheet of the losses by exportation and the gains by importation of phosphoric acid over the whole of the cultivated land of the United Kingdom, but as the loss of phosphoric acid from the soil by drainage is practically *nil*, there seems to be little doubt that our importations of phosphoric acid, directly as manures, and indirectly as feeding stuffs, and our home productions as basic slag, bones and home-consumed corn and other crops used instead of purchased foods, must very nearly if not quite compensate for the annual loss of phosphoric acid. Whether the importations, even if they actually balance the waste, have reached their economical limits is, I think, open to doubt. Much depends upon the relative prices of the manure and the produce, but as it is the immediately available phosphoric acid rather than the latent reserve store which, so far as phosphates are concerned, determines the crop, it will probably pay those farmers, who farm highly to continue to purchase phosphatic manures, even although the actual store of phosphoric acid in the land is annually increasing.

The English farmer is proverbially slow to avail himself of any improvement suggested from outside, and even to-day, with an experience of over 50 years in the use of purchased manures, a very large proportion of our farmers, keen as they are in the sale of beasts or other produce, have very crude notions as to the composition and value of the manures they purchase, and unfortunately in too many instances consider that money withheld from the purchase of fertilisers is so much money gained. These barriers of ignorance are rapidly being broken down by the more extended agricultural education of the rising generation; by means of the classes and field experiments conducted under the auspices of the Technical Education Committees of the County Councils; by the more important agricultural experiments at Rothamsted, Woburn, &c., and indirectly by the Fertilizer and Feeding Stuffs Act, and it is probable that a somewhat increased demand may be looked for as the effect of this general spread of knowledge. But this notwithstanding, it seems to me pretty evident that for home consumption the importation and production of phosphoric acid-containing materials is rapidly reaching the maximum required by the exigencies of the situation. If it could be shown that the demands on the phosphatic stores of the world were as nearly reached by other nations as by us, the question of future supplies is not one which would come within the range of practical polities. As it is, these future drains on our phosphate reserves are and must be an unknown quantity, but having regard to the enormous stores distributed all over the world in small quantities, and capable of concentrating by one means or another, to the vast known deposits of rich phosphates in Florida, Carolina, Algeria, Canada, and many other countries, to probable future discoveries, and the fact that hardening prices would tend to the better conservation of our available supplies, it does not require a great stretch of imagination to see for many centuries to come a fertile land growing increasingly heavy crops without any indications of phosphatic starvation.

As before indicated, it is exceedingly difficult to draw up any accurate balance sheet of our national losses and gains in phosphoric acid, but the following table, based on the most accurate statistics I have been able to obtain, will in the main substantiate the position I have taken up:—

$P_2O_5$ in soil of Great Britain and Ireland, at 0·10 per cent. in a depth of 9 inches, about 63,000,000 tons.

Loss per annum, at 10 lb. per acre over cultivated area of United Kingdom, 214,000 tons.

Gains in 1897:—

| | Tons. | Tons. |
|---|---|---|
| Guano, 16,164 tons at 18·3 per cent. | 2,958 | |
| Bones, 58,208 tons at 23 per cent. $P_2O_5$ | 13,387 | |
| Phosphates, 325,133 tons at 25·2 per cent. | 82,923 | |
| | | 99,268 |
| Imported cattle (alive), 618,336 tons at 20 lb. $P_2O_5$ each<br>„ sheep (alive), 611,504 tons at 2½ lb. $P_2O_5$ each<br>„ beef, 3,010,387 cwt.<br>„ mutton, 3,193,276 cwt. | | 8,000 |
| Home consumption of basic slag, 100,000 tons at 15 per cent. | | 15,000 |
| Imported wheat, 62,743,280 cwt. at 0·85 per cent. $P_2O_5$ | | 26,660 |
| „ wheat meal and flour, 18,680,669 cwt. at 0·20 per cent. $P_2O_5$ | | 1,868 |
| „ barley, 18,958,720 cwt. at 0·75 per cent. $P_2O_5$ | | 7,109 |
| „ oats, 16,116,810 cwt. at 0·60 per cent. $P_2O_5$ | | 4,830 |
| „ maize, 53,785,380 cwt. at 0·60 per cent. $P_2O_5$ | | 16,134 |
| „ beans, 2,840,050 cwt. at 1·10 per cent. $P_2O_5$ | | 1,562 |
| „ peas, 2,820,135 cwt. at 0·85 per cent. $P_2O_5$ | | 1,198 |
| „ oat meal, 732,195 cwt. at 0·40 per cent. $P_2O_5$ | | 146 |
| „ maize meal, 1,029,301 cwt. at 0·60 per cent. $P_2O_5$ | | 308 |
| „ oil-cakes, 336,958 tons at 2·25 per cent. $P_2O_5$ | | 8,180 |
| „ cotton seed, 412,879 tons at 1·60 per cent. $P_2O_5$ | | 6,605 |
| „ linseed, 1,908,628 qrs. at 1·54 per cent. $P_2O_5$ | | 5,379 |
| „ rape seed, 185,332 qrs. at 1·90 per cent. $P_2O_5$ | | 653 |
| Home-produced cattle / „ „ sheep — Bones marketed, estimated at 50,000 tons at 23 per cent. $P_2O_5$ | | 11,500 |
| „ „ corn, United Kingdom — wheat, 56,295,774 bushels at 0·85 per cent. $P_2O_5$ | | 12,818 |
| barley, 72,613,455 bushels at 0·75 per cent. $P_2O_5$ | | 12,156 |
| oats, 163,556,156 bushels at 0·60 per cent. $P_2O_5$ | | 17,086 |
| „ „ beans, 6,650,425 bushels at 1·10 per cent. $P_2O_5$ | | 1,959 |
| „ „ peas, 5,249,732 bushels at 0·85 per cent. $P_2O_5$ | | 1,254 |
| | | 259,673 |

Sub-soil $P_2O_5$—unknown but large.

In explanation of this table I would remark, in the first place, that the annual average loss which I have calculated at 10 lb. of phosphoric acid per statute acre, probably exceeds the present drain on our resources by at least one-tenth. Warrington has calculated the average loss on a four course rotation, with all the corn and the fattened beasts sold off the farm at something less than 10 lb. per acre. Where a five or six course rotation is adopted, the quantity would probably be less. Moreover, more than half of the land under cultivation in Great Britain is in meadow land or permanent pasture, and although it is not easy to gauge the loss on this vast area, there can be little doubt that the loss I have assumed leaves a very wide margin of excess. On the other hand, much of the imported phosphoric acid never finds its way upon the land. A large proportion of the imported wheat, for instance, is consumed in towns, and for agricultural purposes is lost; but in this connection it must be remembered that most of the offal which contains about five-sixths of the phosphate is used directly or indirectly as cattle food. Some of the barley and maize is used for brewing, the manufacture of glucose and other industrial purposes; but by far the largest portions of these grains are used for feeding purposes, and eventually find their way on to the land in the form of manure. Even those portions retained in the animal economy will in the long run be returned, for the most part as bone manures.

Another source of error is the exportation of unknown quantities of manufactured manures; but these, being to a large extent nitrogenous, would not materially affect the results.

In treating of the available supplies of plant food, I have considered the phosphate question first, because in the manure-manufacturing industry phosphates have formed, and still form the basis of nearly all manufactured fertilisers. It must not be thought, however, that in doing so, I have been heedless of the fact that nitrogen in a combined form is of equal, if not of greater value, both from the scientific and manufacturing point of view. But in dealing with the sources of supply, and the probability of these meeting the requirements of the future, we are brought face to face with difficulties far greater than when considering the phosphate question. The reason for this is that, unlike phosphoric acid, nitrogen may be lost to the soil in other ways than by removal in crops, and on the other hand it is absorbed by the soil and by plants by other means than by a direct application as manure. These sources of loss and gain have in the past commanded the attention of agricultural chemists, and I think it may now be said that the sources of loss and the methods by which nitrogen is absorbed are pretty accurately understood; but the total quantities so lost and gained to agriculture are by no means clearly defined. The quantities lost in the drainage waters as nitric acid under certain definite conditions have been ascertained at Rothamsted and elsewhere, and the contained nitrogen in the rainfall is also known within very narrow limits; but the quantities absorbed by the soil from the atmosphere, and that fixed by the nodules of our leguminous crops in pasture land, and in an ordinary course of rotation, are beyond the ken of our present science. To attempt to make, therefore, anything like an accurate balance of our outgoings of agriculturally-available nitrogen is a task fraught with grave difficulties, if not impossibility, and rather than run the risk of prophesying on insufficient data, I will ask you to bear with me while I briefly attempt to solve the problem from another standpoint.

Except for land in permanent pasture, the almost universal custom of English farmers has been to adopt a course of rotation of crops, which, experience has shown, can be worked at less cost, and yields larger crops for a given outlay than any continuous system can do. To modify these well established customs by the more frequent introduction of leguminous crops, and so fix the atmospheric nitrogen for subsequent non-nitrogen-accumulating crops, is a change calculated to captivate the scientific imagination, and experiments in this direction are already being undertaken. What will be the result of these investigations it is too early yet to even hazard a guess; but unless it can be shown that the produce of these extra crops has directly for sale or indirectly for consumption a value equal to, or nearly equal to the crops which would be grown in the usual course of rotation, it is doubtful whether the suggested alteration will find favour among agriculturists, at all events with nitrogen compounds, at their present low price. On very few farms is it customary to dress any crop with nitrogen compounds at a greater rate than 2 cwt., or its equivalent, of nitrate of soda per acre, and as this amount can be purchased at present prices for about 16s., it follows that unless the interposed crop can assimilate nitrogen to that extent, or more, and yield money returns over a series of years equal, or nearly equal, to the present systems, there will be little inducement to farmers to alter their methods of cultivation. Whether this can be done the future alone can decide.

As the result of many field experiments conducted in all parts of the kingdom, it has been amply shown that on most farms, even when considerable sums are expended in the purchase of cake and other feeding stuffs, nitrogenous manures may be applied with advantage to nearly all crops, except perhaps the leguminosæ.

It is difficult to estimate to what extent this nitrogenous manuring can be profitably carried for the reasons previously given; but there seems little reason to doubt that a dressing of nitrogen equal to 1 cwt. of commercial nitrate of soda (= 17·5 lb. nitrogen per acre) over the whole area of the United Kingdom under arable cultivation, might be applied with advantage. The average annual loss in a four-course rotation is about 21 lb. of nitrogen per acre for the crops and stock sold off the farm. To this must be added the very considerable loss of nitrates in the drainage waters, and as a set-off there is the gain from the rain and the atmosphere. As the loss of nitrates by drainage is an unknown quantity over the whole area, the only method at my disposal of making anything like an estimate of the actual requirements, is by taking general experience, and the results of carefully-conducted experiments. From these the above estimate of 17·5 lb. of nitrogen per acre does not appear to me, under the present system of cultivation, to be an extravagant one, taking into consideration the limits of profitable manuring at the present prices of produce, and of nitrogenous materials.

Against this annual deficiency there were importations of nitrogen last year as follows:—

| | Tons of Nitrogen. |
|---|---|
| Oil cakes, 336,958 tons at 5 per cent. ....... | 16,646 |
| Cotton seed, 412,879 tons at 3·5 per cent. ... | 14,450 |
| Linseed, 349,350 tons at 3·5 per cent. ....... | 12,197 |
| Rape seed, 34,420 tons at 3·4 per cent....... | 1,170 |
| Maize, 2,689,269 tons at 1·7 per cent. ....... | 45,717 |
| Wheat offal ($\frac{3}{10}$ of imported wheat) at 2·75 per cent. ................................. | 25,824 |
| Barley, 947,936 tons at 1·65 per cent. ....... | 15,740 |
| Oats, 805,840 tons at 2·00 per cent. ......... | 16,116 |
| Beans, 142,002 tons at 4·00 per cent......... | 5,680 |
| | 153,540 |

The residual manurial value of which would be equal to 6·6 lb. of nitrogen, or 42 lb. of nitrate of soda per acre, over the whole of the cultivated area of the United Kingdom, or about 16 lb. of nitrogen over the area in arable cultivation.

Of the more directly manurial importations and supplies of nitrogen used for agricultural purposes, the following are the chief:—

| | Tons. |
|---|---|
| Sulphate of ammonia, 45,000 tons at 20 per cent. ....................................... | 9,000 |
| Nitrate of soda, 105,832 tons, of which say 70,000 tons for manure.................. | 10,640 |
| Guano, 16,164 tons at 4 per cent. ........... | 644 |
| Bones imported, 58,208 tons at 4 per cent. .. | 2,328 |
| „ from imported cattle, sheep, beef and mutton.................................... | 1,333 |
| „ from home grown cattle, 50,000 tons at 4 per cent. .......................... | 2,000 |
| Dried blood, shoddy, ground leather, hoofs and horns, &c ........................... | 1,400 |
| | 27,345 |

equal to nearly 1¼ lbs. of nitrogen, or about 8 lbs. of nitrate of soda per acre over the whole cultivated area, or about 3 lbs. of nitrogen over the area in arable cultivation.

From these figures it would appear that even if the whole of the nitrogen were utilised on the arable land, the limit of the profitable application of nitrogen in the form of nitrogenous manures, is only just reached by imported foods and manures, and by home consumed sulphate of ammonia. As, however, some of the imported foods would be utilised for purposes other than agricultural, and much of the nitrogen contained in the farm-consumed remainder would be lost, or applied directly or indirectly to pasture lands, it follows that, if my premises be correct, the importations of nitrogen in all forms, and the home consumption of sulphate of ammonia fall far short of what must be considered the profitable limit of nitrogenous manuring.

The total production of sulphate of ammonia in the United Kingdom for last year was :—

| | Tons. |
|---|---|
| From Gas works.......................... | 132,724 |
| „ Iron works.......................... | 17,779 |
| „ Shale works ........................ | 37,153 |
| „ Coke ovens and producers.......... | 10,624 |
| | 198,280 |

The total exports were 153,000 tons, partly from reserve stores.

The home consumption in terms of sulphate of ammonia, including liquor used in various processes of manufacture, is estimated by Messrs. Bradbury and Hirsch at 70,000 tons.

The nitrogen in the exported sulphate of ammonia is equal to about 206,000 tons of nitrate of soda, or about three times the nitrate at present used for agricultural purposes, and would give nitrogen equal to 23 lbs. of nitrate of soda over the whole area of land in arable cultivation.

It does not appear, therefore, that our present imports of nitrate of soda and other nitrogenous manures, or of feeding stuffs yielding a manurial residue of nitrogenous matters, need be very greatly augmented to raise the fertitity of the land to its paying limits provided our production of sulphate of ammonia be utilised at home. The production of sulphate of ammonia, notwithstanding a steadily declining selling value, has increased in a phenomenal manner, and there seems little reason to doubt that, with the improvement in processes and the keener competition on all sides, much of the nitrogen at present wasted will be pressed into service.

If I am right in this surmise it is evident that the pessimistic views of Sir William Crookes, expressed at the British Association meeting at Bristol, as to the exhaustion of our natural supplies of combined nitrogen to satisfy the rapidly increasing maw of the wheat-consuming human race, will have little terrors for the British agriculturalist, whatever they may have for the toiling millions of towns.

With free nitrogen around us in unlimited quantities in the atmosphere, the most scrupulous conscience need not suffer any pangs at any untoward drains upon our natural supplies of combined nitrogen, and, indeed, if Sir William Crookes' estimate of the cost of producing nitrate of soda by the combustion of the atmosphere at Niagara be correct, the question of nitrogenous plant food will be for ever solved, and the already depressed nitrate of soda industry will soon be a thing of the past!

From a purely-agricultural point of view it is of little moment whether the extra nitrogen required to raise the fertility of the land to its profitable maximum is obtained from nitrate of soda or sulphate of ammonia, but from the chemical manure manufacturers' point of view it is important. Nitrate of soda does not lend itself satisfactorily for mixing with superphosphate in the manufacture of a compound manure, whereas sulphate of ammonia not only mixes well, but tends to produce a friable mixture which can be sown easily by drilling, or otherwise. Apart from the better condition obtained by the use of sulphate of ammonia, there are other reasons why it should be used in preference to nitrate of soda in the manufacture of compound manures. If used as a top dressing, the balance in favour of the use of one or the other, the cost per unit of nitrogen being equal, is determined to a very large extent by the wetness or dryness of the season. Thus Dr. Voelcker, in his summary of the results of the Woburn experiments in the Journal of the Royal Agricultural Society for last year, cites the following comparative experiments in exemplification of this point:—

| Year. | Season. | Rainfall. | Ammonia Salts. | | | Nitrate of Soda. | | |
|---|---|---|---|---|---|---|---|---|
| | | | Corn (Wheat). | Straw. | | Corn (Wheat). | Straw. | |
| | | inches. | bushels. | cwt. | qrs. | bushels. | cwt. | qrs. |
| 1887 | dry | 15·04 | 26·1 | 22 | 1 | 35·0 | 29 | 0 |
| 1890 | „ | 16·80 | 24·7 | 19 | 2 | 31·2 | 26 | 3 |
| 1889 | wet | 23·94 | 26·3 | 26 | 1 | 18·9 | 23 | 0 |
| 1894 | „ | 26·34 | 45·5 | 40 | 1 | 36·2 | 36 | 3 |

On the average of 20 years the balance is slightly in favour of sulphate of ammonia when used alone at the rate of 50 lbs. ammonia per acre, but when used in conjunction with mixed mineral manures, the balance is slightly the other way. As however, the average annual rainfall at Woburn is less by some 10 inches or so than the average of the United Kingdom there can be scarcely any doubt that taking an average of the whole country for a series of years, the balance, unit for unit of nitrogen, would tend to be in favour of sulphate of ammonia as against nitrate of soda as a manure.

There is, however, a still more potent reason why sulphate of ammonia, or other more slowly acting nitrogenous compound, such as dried blood, should be used in the manufacture of artificial manures. These are usually sown earlier than nitrate, and consequently the risks of loss by drainage are less in the case of slowly nitrifying materials, which are either comparatively insoluble, or which, as in the case of sulphate of ammonia, form insoluble compounds with the soil. As the available quantities of sh oddy, dried blood, hoofs and horns, &c. are comparatively small and not capable of any immediate considerable expansion, it follows that if the manufacture of artificial manures is to extend the nitrogen necessary for their production, will have to be drawn from the supplies of sulphate of ammonia at present exported.

With regard to the only other essential constituent of artificial manures potash there need be no concern with regard to future supplies. On very few soils is potash of great value, and the supplies in Germany seem to be capable of meeting our needs for many years to come. Even were these to become exhausted it does not seem to be beyond the reach of chemical science to extract some of the 2 per cent. or more of potash contained in many of our clay soils to supply those on which potash is found to be necessary.

From what has been said it is evident that while a small increase in the home consumption of phosphates may be looked for, there is every reason to expect, should the present ratio between the prices of produce and manures remain the same, a considerable increase in the consumption of nitrogen compounds. In potash manures it is not likely that there will be any greatly increased trade.

Such, then, being the general outlook, so far as consumption is concerned, to what extent is it probable that the manure manufacturer, in contradistinction to the manure dealer, will profit? I fear the answer to this question will not be of so satisfactory a character as the inquiry into the prospects of consumption. During the past 10 years a great change has come over the scene. The spread of technical education, the wide extension of field experiments, the introduction of basic slag, the largely increased production and systematic advertising of nitrate of soda, and the straits through which agriculturalists have passed, have all tended to educate farmers in the use of artificial manures, and without doubt the general trend of this education has been to increase the consumption of the simple forms of manure, such as nitrate of soda, sulphate of ammonia, mineral superphosphate and basic slag, at the expense of compound manures. The Fertilizers and Feeding Stuffs Act, 1893, has also played an important part in this change. By the constant statement of percentages of the valuable fertilising ingredients, farmers have been familiarised with the market values of these substances from various sources, and the Act has had a tendency to reduce prices to the level of the cheapest forms, without due regard to any differences in assimilability that may exist. As the lowest prices are not usually those at which a high-class mixed manure can be produced, manufacturers have more and more felt the brunt of this unequal competition, and on present lines there does not seem to be much hope in the future.

Another cause tending to limit the production of artificial manures is the low price of wheat and other produce, which has induced farmers to reduce their expenditure by putting land into permanent pasture. During the past 10 years about one and a quarter millions of acres of land have been thus converted, and as a consequence less money has been spent in this for manures than would have been spent had it remained in arable cultivation. If money has been spent on imported fertility it has gone to the cake merchant for cattle foods rather than to the manure manufacturer.

The demand for basic slag is rapidly increasing, and I am informed that it has now outstripped the supply, and that there is a probability of the consumption for the year ending June next being 120,000 to 130,000 tons. Such consumption necessarily diminishes the output of superphosphate and compound manures, and educates farmers in the use of simple manures. It seems to me, therefore, that there will be an ever-increasing tendency for the manure manufacturers' operations to be limited to the production of superphosphate, and that the compound manures upon which he has hitherto relied for the bulk of his profits will have to give place to the simpler forms of manure, or the prices will have to be reduced to compete in equal terms with outside sources of nitrogen and phosphates.

In conclusion, I have to express my indebtedness to Mr. Ryland for the details of the early history of the industry, to Mr. Hermann Voss for particulars regarding our phosphatic supplies, to Messrs. Bradbury and Hirsch for many details respecting nitrogenous production, to Messrs. H. and E. Albert for information about basic slag; to Dr. Voelcker for details respecting the Woburn experiments; and I now take this opportunity to thank them for their courtesy and help.

## London Section.

*Meeting held on Monday, November 7th, 1898.*

MR. BOVERTON REDWOOD IN THE CHAIR.

Mr. BOVERTON REDWOOD, on assuming his position as Chairman, addressed the following remarks to the members assembled:—

"You will have seen, gentlemen, from the notice which has, I believe, reached you all, that we propose to pursue that course which is customary in this Section of the Society of plunging *in medias res* by proceeding to the reading of a number of communications, the titles and the names of the authors of which have already been placed before you, without anything in the way of an introductory address from the Chair. But I will ask you to bear with me a very short time while I make a few remarks. I wish in the first place to express to you my deep sense of the high honour which you have conferred upon me by my unanimous election to the chair of this Section. I am well aware that it is only my loyalty and devotion to the Society which can be held to constitute any real justification for your choice. My work for many years has been of a practical rather than a theoretical character, and it is probably in consequence of that that throughout a somewhat lengthy period of official connection with this Society, as member of the Council and of the Publication Committee, I have found myself growing more and more in sympathy with what I regard as the practical and useful work which this Society throughout its various Sections is doing. As a chemist I feel that I can lay no claim to the position to which you have been so good as to elect me. If I have some knowledge of a certain branch of technology it is due to the fact that chance in the first instance brought me intimately into connection with an industry which then was one of no great magnitude, and I have been fortunate thus to be afforded the opportunity of making mental growth commensurate with that of that industry. But if I cannot bring to bear upon our deliberations anything more than knowledge which is narrowly circumscribed by the limits I have mentioned, I can, at any rate, assure you that it will be my earnest desire to emulate the good example of those who have preceded me in this Chair. I scarcely dare hope that circumstances will admit of my equalling the unremitting attention of my immediate predecessor, who, I understand, never once missed presiding over a meeting of the Section. I am the servant of a great many people (I may be pardoned for going into a purely personal question at this moment), and all of them think themselves entitled to demand and receive my exclusive services whenever they choose to call for them. If I should thus be prevented at any time from discharging the duties of my office, it is a great satisfaction to me to feel that those duties will devolve on such a capable man as my friend the Vice-Chairman, Mr. Hehner. I may also say that in accepting office it was a source of support to me to feel that I should be associated with so efficient a Committee, and I may say, speaking in the spirit of prophecy, with so able a

Secretary as Mr. Ling. It will be my aim to preserve the high level to which the deliberations of this Society in general, and this Section in common with others, has attained in its discussions, and if I should not always be able to succeed in my aim I will ask you to be lenient, and to ascribe any shortcomings to the fact that in my predecessor we had one who was so unremitting in his attendances that he never afforded to his understudy an opportunity of preparing himself by appearing on the boards. I would refer to the very great loss which this Society has sustained in the death of the Hon. Treasurer. It is well known to all of us that Mr. Edward Rider Cook was one of the promoters of the movement which resulted in the establishment of this Society, and while the value of Mr. Cook's services is generally known to the members of the Society, it is only those who have been officially connected with him who really understand quite how much he did for the Society, and how much the Society owes to him. Mr. Cook, as we know, was, at the founding of the Society entrusted with the control of the Society's finances—it was in the year 1881—and he continued to occupy that important and responsible position until the time of his death. It is not too much to say that the interests of the Society were never absent from Mr. Cook's mind during the whole of that period, and the Society very largely owes its present prosperous position to the judicious counsels and heartfelt solicitude which he exhibited. In our sorrow at the loss we have sustained it is a consolation to feel that it has been practicable to provide in effect for the continuity of those services by the election of Mr. Samuel Hall, who, to the very great satisfaction of the Council, has consented to assume the duties in question. I would also refer to the loss which the profession of chemistry has met with in the removal by death of Mr. J. A. R. Newlands, whose modest and retiring character always appeared to me to stand in the way of any adequate recognition of his exceptional merits. Mr. Newlands, the discoverer of the law of periodicity of the atomic weights of the elements, was a deep and original thinker. Such men are rare, and we can ill afford to lose them.

He would interrupt the proceedings at this stage for the performance of a ceremonial act which could not take place earlier owing to the absence of a gentleman who was deeply interested in the proceedings. The ceremonial in question pertained to previous sessions of the Section, and for that reason he would call upon the late Chairman, Dr. Messel, to perform it.

Dr. R. Messel then presented to Mr. John Heron an illuminated testimonial signed by the four chairmen under whom he had acted as hon. secretary of the Section from November 1892 to June 1898, in recognition of his distinguished services to the Section during that period.

Mr. Heron briefly addressed the meeting in acknowledgment of the testimonial.

## ON A CENTRAL AMERICAN LIGNITE, MOSTLY SOLUBLE IN BENZENE.

BY WATSON SMITH.

A few pounds of this peculiar mineral were sent to me as a coal recently discovered in Central America, and I was requested to investigate it for composition and possible uses. The wording of the message showed me that it was regarded as *bona fide* coal, but no information was vouchsafed as to precise locality, and for some reason it was not considered desirable to make more known about it.

I observed that the mineral had all the external appearance of a good coal. It was fairly friable, and readily broke into bits, partaking generally of cubical form, as true coal or some advanced lignites are known to do; but on handling the mineral further I observed that its dust was brown, though in bulk the mineral was black and lustrous. Now this peculiarity is generally given as characteristic of the advanced lignites, and I next began to think of my old acquaintance—the Japanese Miike coal (lignite), and then to resolve to apply a test for soluble bituminous matter. On placing some fragments in a test-tube and warming with benzene, judge of my astonishment on seeing the benzene turn almost immediately dark from the quantity of soluble matter extracted, and on filtering and evaporating some of the filtrate in a small dish, on finding so considerable a residue of tarry or bituminous residue. However, I will now repeat this simple experiment side by side with one with the Miike lignite, that you may see the great difference, and you will remember the latter contains 9 to 10 per cent. of soluble bituminous matter.

You will observe that a fragment of this coal on contact with the flame readily takes fire, and burns like a candle. The Japanese Miike coal does this too, but it does not melt in the process as this Central American specimen does. I can cause this coal to melt like sealing-wax, and even to drop, a thing the Miike coal will not do, and quite impossible with any of our true Cannel coals. I have gone so far as to make an attempt to determine the melting point of the coal, though unsuccessfully. I found, however, that to determine the softening point or agglomeration point was more feasible. This I did by placing some of the powder of the coal in a test-tube, and placing the latter in a bath of paraffin wax. On heating to 300° C. I found that the coal dust became caked upon the sides of the test-tube, as I can now show you.

The specific gravity of this coal is 1·26.

The amount of moisture present was only 0·052 per cent. With regard to the sulphur present, I found in two experiments 9·00 and 9·21 per cent., mean = 9·10 per cent. of sulphur, a remarkably large amount for a coal. The Miike coal contained 1·06 per cent. of sulphur, which is a similar amount to that contained in our Cannels and caking coals on the average.

But whereas the amount of ash of the Miike coal is 9·45 per cent., the amount of ash in this Central American lignite is only 3·76 per cent.

Now if we were to assume that the 9·10 per cent. of sulphur in this latter coal were contained in it as pyrites ($FeS_2$), we should expect a residue of $Fe_2O_3$ of 11·38, and our percentage of ash would have to be in such case at least 11·38 per cent. Nevertheless, as we find that it is only 3·76 per cent., we must conclude that most of this sulphur is contained either as free sulphur, or in the form of organic sulphur compounds. The amount of $Fe_2O_3$ in the ash of this lignite is 13·6 per cent., the iron of which, $Fe_2O_3$, would combine with an amount of sulphur to form pyrites ($FeS_2$), representing 0·41 of the percentage of the sulphur in the coal, thus leaving 8·69 per cent. combined as organic sulphur, if indeed it does not exist as free sulphur.

A destructive distillation was now made from an iron ¾-in. tube, of 25 grms. of the coal, at about a gas-making temperature as nearly as could be judged. 6200 c.c. of gas were obtained, equal to 8899 cb. ft. per ton, 12·44 per cent. of tar, with an odour like that of ordinary coal tar with some Russian petroleum mixed with it, and 44·8 per cent. of coke left as a residue.

So small a yield of coke as this indicates something peculiar in the nature of a coal, for an ordinary gas coal would yield 65 per cent., and even more. But in a lignite so small a yield is more usual. The extraordinary nature of the coal was revealed on extracting 10 grms. with about 500 c.c. of pure benzene in a Soxhlet's apparatus for eight days. In two experiments of this kind the amounts of bituminous matter extracted were 80·2 and 80·0 per cent. respectively, mean = 80·1 per cent. Thus, then, no less than 80·1 per cent. of this remarkable coal or rather lignite, is soluble in benzene.

The benzene extract was now subjected to distillation to remove benzene, and the residue was subjected to further distillation. After removal of all the benzene the first drop of distillate fell into the receiver between 120° and 130° C. Below 350° C., 2·04 grms. distilled over = 20·4 per cent. on the lignite. Above 350° C., 2·01 grms. passed over = 20·1 per cent. on the lignite. The remainder was a viscid mass, which did not solidify on cooling. The oils distilling below and above 350° C. have an odour greatly resembling that of Russian crude petroleum oils, more especially the heated heavy oils.

It now occurred to me to endeavour to discover if this bituminous matter in this Central American lignite contained any nitrogen bases extractible by sulphuric acid, or any phenolic or phenoloid bodies extractible by caustic soda. My efforts as regards the nitrogen bases have met with decided success, though with considerably greater success as regards phenoloid constituents, for on acidifying the caustic soda extract, a film of phenoloid bodies separated with an odour closely resembling that of the phenols which, about 12 years ago, I extracted from Gartsherrie blast-furnace tars. (This Journal, 1883, 497—499.)

With regard to possible uses for such a lignite as this, I see no difficulty in using it as ordinary fuel, if it be judiciously mixed with coal of harder or more anthracitic character. It could also readily be used for asphalting footways and roads, &c., by treating like pitch, and melting up with a certain proportion of creosote oil, and mixing with cinders and gravel.

It might even be employed as pitch is, for the making of black varnishes for iron-work.

In reply to any suggestion that this substance is neither coal nor lignite, but a mere variety of mineral pitch or substance like Ozokerite, or Trinidad pitch, I would point to the fact of its decomposition on destructive distillation, with the fairly good yield of coal gas, and of tar to a considerable extent, of benzenoid character, taken in conjunction with the fact that on attempting to melt it with the idea of making a melting point determination, I found that fusion and decomposition with semi-incineration took place in stages following each other so closely, that uniform melting was impossible. Besides this there are for consideration its physical properties and appearance, &c.; and the fact that it conducts heat like a coal and unlike a bitumen. In short, the physical conditions of a bitumen or mineral pitch are almost as unlike those of such a lignite as this, as the physical conditions of an amorphous substance are unlike those of a crystalline substance. To pass from the physical condition of this mineral to that of bitumen, involves a change of state. Heat might bring this about; but no means that I know of could be found by which to effect a retracing of that step. I believe there would be no serious difficulty in the way of using this lignite to charge the ordinary gas-retort, though the coke produced would, I anticipate, be of a light and friable character. The coal tar obtained by a gas manufacturer from the Central American Coal would, I can certainly predict, be practically useless to a tar distiller, because of its containing along with aromatic hydrocarbons so much of the paraffins and other members of the aliphatic series.

With regard to the character of geological formation in which this remarkable lignite is found, I think some conclusions may be drawn from the character of its ash left on incineration. This ash, as you will here see, is reddish, though not much so. It contains even more alumina than iron. The chief constituent is, however, silica, and the siliceous grains are perfectly visible with the aid of a magnifying glass. I should judge, then, that the stratum in which this lignite is found is one of sandstone, and that any underlying clays will be very siliceous. This evidence will, it seems to me, show that the deposit is above the coal measures, and goes to confirm previous evidence that it is a lignite or tertiary coal. In his work on Fuel (1875), p. 317, Percy mentions a Trinidad lignite, which is black and bright, like good bituminous coal, but with dark brown powder. It scintillates in the flame, "and evolves on heating an odour of petroleum." No doubt this odour is that of the vapours of the bitumen contained in it, which will doubtless resemble that from heated Trinidad pitch. If Trinidad pitch be heated, and also this Central American lignite, and the Miike coal from Japan, the odours are remarkably alike, resembling the smell evolved in our streets when asphalting with Russian petroleum residues is being carried on. That this peculiar odour is characteristic of the bitumen, is, I think, shown by taking a specimen of true coal—a Cannel—containing but very little of this soluble matter, and heating it. It burns with a bright flame like the others, but on blowing out the flame, the odour is perceived to be but very faint, as well as different in character. Nevertheless, it is an odour characteristic of all the bituminous coals and Cannels of the coal measures when heated, so far as I have examined them.

In this singular mineral, if we chose to regard the soluble portion as a form of crude petroleum, we have, in every 100 tons of it, no less than 80 tons of petroleum. This 80 tons being agglomerated into a hard solid mass of coal-like structure, by 20 tons of other constituents, organic and inorganic, insoluble in napthas and hydrocarbon oils. Of what assistance the discovery of this product will prove with regard to the theories of formation of petroleum in the earth, I can hardly at present say, but its occurrence does not in the least disturb my own views, *viz.*, that there is enough petroleum in the world, and in sufficient variety, to give most, if not all, the current theories, opportunities for each to play some part. At present, I should not like to dogmatise as to the universal truth of any one theory in this direction. I have much pleasure in thanking my assistant, Mr. Albert Shonk, for his help in the experimental part of this paper.

## DISCUSSION.

The CHAIRMAN observed that before taking the discussion on this Paper it would be convenient for Mr. Watson Smith to add a few remarks on the exhibit of Miike coal, as the two subjects were very nearly related.

Mr WATSON SMITH thereupon observed that they had the advantage of the presence of Mr. Dan, the chief engineer of the Miike Mines, who would be able to tell the meeting much more about the character of the coal than he could. He might observe, however, that a special interest attached to this coal, inasmuch as it could properly be called an Imperial product, seeing that the first person to take an interest in the subject and to arouse sufficient interest in it to bring about what was now an important national industry, was no other than his Majesty the Emperor of Japan. (See this Journal, 1891, 976, col. 2, and 977.)

The CHAIRMAN said that before calling upon Mr. Dan he would like to observe, in reference to the interesting paper read by Mr. Smith, that he, personally, felt considerable doubt as to whether the subject of Mr. Smith's paper was correctly described as either a lignite or a coal. He had, in his own work on petroleum, referred to certain minerals found in North America under the names of gilsonite, uintahite, and other names, notably in Utah, near Fort Duchesne, and in another Indian reservation, near the Colorado State line, which possessed properties very similar to those attributed to the mineral which was the subject of Mr. Smith's paper. Moreover, in Dana's "Mineralogy," various forms of petroleum were described in detail, and some of those descriptions were not very dissimilar from the results brought before them that evening. For instance, there was a form of bitumen, wurtzilite (named after Dr. Henry Wurtz, of New York), which contained 90 per cent. of soluble bituminous matter, presented a general resemblance to jet and some of the cannel coals, melted in the flame of a candle, burned with a luminous flame, and with little smoke, fused in a glass tube with emission of a dense white and yellow smoke, and distilled over a thick brown tarry oil, leaving a small residue of fixed carbon. Then again, there was the well-known material albertite, which was said to differ from ordinary asphaltum in being only partially soluble in oil of turpentine. This body also appeared to behave very much in the manner described by Mr. Smith. Again, there was grahamite, which also was soluble mostly in oil of turpentine, and partially in ether, naphtha, and benzene, wholly so in chloroform and carbon bisulphide, but was insoluble in alcohol. Uintahite was described as a variety of asphalt, black and lustrous. It fused easily and burned with a brilliant flame, much like sealing wax, and would give a clean, sharp impression from a seal. Unless very hot it did not adhere to cold paper, was extremely plastic; on distillation gave a small quantity of clear white dense oil, and a little gas or vapour, dissolved freely in oil of turpentine and in ordinary alcohol, but not in cold ether. Moreover, in "The Mineral Industry, 1892," under the head of Utah, he found a reference to deposits of gilsonite confirmatory of the description which he had already given, and it was said that this body had found a ready market in St. Louis, where it was used as a varnish, bringing 50 dols. per ton, the shipment in 1892 amounting to 1,485 tons, valued at 74,250 dols. He ventured to trouble the meeting with these remarks because it appeared desirable to supplement the interesting particulars which the author had given them by any information which might lead to an exact knowledge of what the substance under notice really was. It was regrettable that the author was not in a position to state definitely where the mineral came from, except that it was Central America. Judging from its characteristics it seemed to him likely to be one or other of the substances to which he had referred.

Mr. T. Dan, in the first place, desired to thank the Society for the hospitality which had been extended to him in connection with the present visit, and which was the more valuable to him as he had now very few opportunities of coming into touch with scientific centres. He would like to say a few words with regard to the remarkable paper read before the Society some years ago by Mr. Watson Smith (this Journal, 1891, 975). He was particularly interested in that paper because of a prediction which Mr. Smith then gave with regard to the nature of the Miike coal-field, which he (the speaker) was then engaged in mining. He had several times tried to make firebrick from the clays lying next below the coal in the position usually occupied by the "fireclays," but he had never succeeded, on account of the presence of a large amount of lime. This was exactly the state of things which Mr. Smith had predicted from his analysis of the coal and its ash. Practical results had also confirmed Mr. Smith's prediction with regard to the use of the coal for the production of gas and coke. He (the speaker) had constructed several coke ovens of the beehive type, and was now making coke in them which could not be distinguished from any of the best cokes made in England. But of course the yield of the coke was only about 51 per cent., owing to the character of the ovens and the use of part of the carbon in the burning. He hoped soon to be using closed ovens, and expected them to realise quite 60 per cent. As to the gas coal, it was now used generally in the Chinese and Japanese markets for the manufacture of gas. He feared that any remarks of his with respect to the Japanese coal formation could have little interest for the meeting, as they would be made chiefly from a mining engineer's standpoint, not from a chemical point of view. But with regard to the coal formation in the south of Japan, there were perhaps some peculiarities that might be interesting. He had already mined about 4,000 acres of land, and in all that area had not found any large fault, certainly none to the extent of more than 5 feet. He thought that that was very remarkable, bearing in mind the volcanic nature of the country. Another peculiarity was that there was no bed of stone overlying the coal that was impervious to water. The result of this was that in mining the coal they had to encounter very great difficulties with the water soaking through, for it followed them everywhere, and had to be pumped up continuously. Another matter peculiar to the Japanese coal was the formation of natural coke by the action of volcanic lava. Large areas of coal had been converted entirely into coke by this action; and it was no uncommon thing to find three or four seams of very good coal, and in the middle, one seam which had been converted in that way. Possibly that might be due to the smaller resistance offered by that particular seam to the inflow of lava. At any rate, he did not know of any similar case except in Colorado, where he had recently been. There they had dykes, and for some distance from them the coal was converted into natural coke. But the case was different in Japan, for there this phenomenon did not only occur near the dykes, but a whole area would be affected. Moreover, there were two kinds of natural cokes, one of which suffered decrepitation when put on a fire, while the other burned like charcoal. This led to their being employed for different purposes, the first-mentioned kind being used for burning lime, and the other for domestic purposes. With regard to the Miike coal, he had made extensive tests of its evaporative power, but unfortunately with a boiler which was by no means new, so that the results were not the best obtainable. He found that 1 lb. of Miike coal, under those circumstances, would evaporate a little more than 8½ lb. of water at the boiling point. With regard to coal mining in Japan, it might perhaps be of interest to mention that most of the mining there was done by convict labour. That state of things had been introduced by the Government at the very beginning, and the result was that at the present time convict labour was more efficient in those fields than ordinary labour. The miners were made as happy as conditions would allow; they had good food, and were kept clean and healthy, and were in some respects in a better position than ordinary labourers.

Mr. F. W. Rudler said that the author had submitted to him for examination some of the fossils from the Miike coal-fields now exhibited, with a request that he would ascertain their geological horizon, as an assistance to settling the point whether the substance was to be regarded as a lignite or as a coal. Unfortunately, most of the samples had been so ill-preserved that it was impossible to speak with any certainty as to their character. There was, however, one specimen upon the table which was in fairly good condition. That specimen was pronounced by Mr. Newton, the palæontologist of the Geological Survey, to be undoubtedly a nautiloid form of cephalopod, probably of a genus having a geological range between the upper cretaceous and lower tertiary system. Consequently, its evidence, so far as it went, tended to support Mr. Watson Smith's contention that the substance in question should not be regarded as a true coal, but possibly a lignite. He had that day had a microscopic section of the specimen made, but it was not satisfactory. There was a deep brown homogeneous substance, through which were sparsely disseminated a few seed-like substances. That fact fitted in well with Mr. Smith's hypothesis. Probably, therefore, the bed of Miike coal, from which this specimen was taken, was lignite, but so altered as to have lost all traces of its vegetable tissue and to have become a highly bituminous lignite. Mr. Smith had also placed in his hands a few fragments of the mineral from Central America, but without any geological information. It was suggested that it might have some kinship, if not with the albertite, possibly with the gilsonite, which had been referred to by the Chairman as a body of industrial importance.

Mr. J. W. Thomas said that 20 years ago he had made experiments with regard to lignites, some of which he thought were especially interesting. The specimens dealt with were to be found at Bovey Tracey in Devonshire, but the mines were now flooded and consequently unworkable. In making his experiments he dealt with a body commonly known as Retin asphaltum, which had some resemblance to the mineral now before them, inasmuch as it was nearly wholly soluble in ether, but it contained some water. He had experimented with a view to obtain the occluded gases from that asphaltum, and also from the lignites associated with it. The body would burn in the same way as Mr. Smith's sample; and in attempting to get the occluded gas from a Bovey lignite by means of a Sprengel vacuum, he had obtained between 120° and 150°, a quantity of light oils, and shortly afterwards the mercury in the Sprengel was affected by the sulphur compounds which came over. He expected that Mr. Smith would find, on further investigation, that the sulphur existed largely in the free state, for in his experiments crystals of sulphur were eventually formed. In one instance (not from Bovey Tracey), a considerable proportion of a white substance came over, which ultimately proved to be paraffin. Notwithstanding the remarks of the Chairman, he believed it would be found that this compound was a lignite of the mineral resin type. Other characteristics were probably due to the fact that it was formed from decayed wood, in contradistinction to the undergrowth of the forest trees.

Mr. Watson Smith said, in reply, that he had inquired into the subject of *gilsonite, &c.*, and hesitated to accept the view of the Chairman because that body (*gilsonite*) did not agree in physical structure (coal-like structure), nor had it the conductivity for heat of the mineral, and he considered these points of variation extremely important. He had recently obtained a specimen of *gilsonite*, which might be accurately described as to appearance and behaviour as a mineral pitch. It had neither the structure nor appearance of the Central American product. He would point out also that Dr. Percy had described definitely as a lignite a mineral found in Trinidad (not the pitch-lake product), which seemed similar to this Central American product in physical properties and presence of bituminous matter. The fact, however, that coals, lignites, and mineral pitches or bitumens shaded the one into the other almost insensibly was, he thought, of very great interest, inasmuch as these lines of demarcation seemed to be marked by differences of content of soluble matters, which as regards chemical composition were of similar character to the higher boiling constituents of petroleum.

## A NEW METHOD FOR THE DETERMINATION OF THE MELTING POINTS OF FATS.

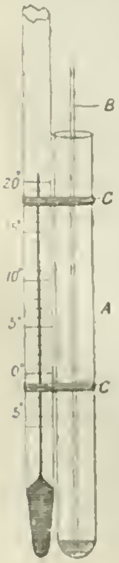
HENRY R. LE SUEUR, B.SC., AND ARTHUR W. CROSSLEY, M.SC., PH.D.

Many methods have at various times been proposed for the determination of the melting points of fats and waxes, but to most of them objections may be raised. One difficulty, which has led various observers to quote different melting points for one and the same substance, is the uncertainty as to which temperature should be considered the true melting point; is it that temperature at which a fat begins to show signs of liquefaction, or is it that temperature at which the fat becomes thoroughly transparent, or, in other words, is completely melted?

In chemical manipulation, the saving of time is a very important factor, and unfortunately all the methods which have so far been proposed require a considerable amount of care and attention, if anything like satisfactory results are to be obtained.

Perhaps the method most frequently employed is the one in general use in chemical analysis, and which may be described as the "capillary tube method." Here in the case of fats and waxes the difficulty, which presents itself, is the filling of the capillary. If the substance under examination be of a waxy nature, which does not admit of its being ground in an agate mortar and then introduced into the tube in the ordinary way, it becomes necessary to first melt the substance, suck it into the capillary, and allow it to cool for some considerable time before carrying out the determination. Besides the expense of time so incurred, it must be remembered, that the melting point of a fat is influenced by the length of time it is allowed to cool after once having been melted. A similar difficulty is met with in the case of a fat, such as butter, which possesses a low melting point; in such a case the warmth of the hand may often be sufficient to partially liquefy it during the filling of the capillary. It is not necessary to enumerate the objections to the several other methods which have from time to time been proposed.

We have recently been engaged in the examination of the physical and chemical constants of a number of Indian oils, many of which are solid at the ordinary temperature, and as we experienced some difficulty in determining the melting point of these solids by the ordinary method, we sought a new one, and we trust not in vain. In any case the new method is so simple and the results obtained so satisfactory that we think it is of sufficient importance to bring before the notice of this Society.



The principle of the method depends on the fact that liquids show the phenomenon of capillarity, whilst solids do not.

The determination is carried out in the following manner:—

Into a small thin walled tube A (a convenient size being 75 mm. long and 7 mm. wide) is placed a fine capillary B, open at both ends, the diameter of which should not be more than ¼ mm.; and then a small portion of the substance under examination is introduced so that the end of the capillary is well surrounded by it.

The whole is then attached to the stem of a thermometer by two rubber bands (C), and placed in a beaker of cold water, which is slowly heated and continually stirred.

That temperature at which liquid is seen to rise in the capillary tube is noted as being the melting point of the fat. Provided the substance has been slowly heated, and this is perhaps the one precaution which it is absolutely necessary to observe, there is no difficulty in recording this temperature, for the moment at which the liquid rises is perfectly apparent.

It was at first thought that the results obtained might depend to some extent on the position of the capillary in the tube, that is, whether it (the capillary) were placed right in the centre of the substance or along the wall of the tube. This was tested by placing three capillaries in one tube, the one in the centre and the others at the sides. On gently heating, the liquid rose in the three capillaries at practically the same moment, and at exactly the same temperature.

The accuracy of our method was further tested by observing the melting points of substances (varying from 45° to 219°) by the "capillary tube method" and by our method at the same time. The results are given below:—

| Substance. | Capillary-tube Method. | New Method. |
|---|---|---|
| *o*-Nitrophenol | 45·5—46 | 46 |
| Dinitrotoluene 1.2.4 | 70—71 | 71 |
| Phenylacetic acid | 76—76·5 | 76·5 |
| Naphthalene | 79—79·5 | 79·5 |
| Saligenin | 83·5—84 | 83·5 |
| Acetanilide | 113—113·5 | 113·5 |
| Salicylic acid | 154·5—155·5 | 155·5 |
| Mannitol | 164·5—165 | 165 |
| Camphoric acid | 184—184·5 | 184·5 |
| Camphoric anhydride | 218—219 | 219 |

It will be noticed that the melting points observed by the "capillary tube method" range over 0·5° to 1·0°, whilst by the new method an exact temperature is recorded; a result which naturally follows from the principle of the method.

The advantages of this method are briefly the following:—

1. The manipulation and apparatus is of the simplest order, and the time required for the determination a minimum.

2. The objection to the ordinary "capillary tube method" already mentioned, namely the filling of the tube, is entirely done away with, as it is easy to introduce a sufficient quantity of the substance into a tube of the dimensions stated.

3. The choice of temperature which should be recorded as the melting point and to which allusion has already been made, is not left to the discretion of the observer; there being only one possibility, namely that temperature, at which any of the substance has been converted into the true liquid state. This is of great importance, as it enables different observers to record the same temperature as the melting point of one and the same substance. Of this we have assured ourselves, by submitting three substances to different observers, who, unknown to one another, have determined the melting points with the following results:—

| Substance. | Observer A | Observer B | Observer C | Observer D |
|---|---|---|---|---|
| Butter | 33 | 32·8 | 33° | 33 |
| Spermaceti | 45 | 44·5 | 44·5 | 44·5 |
| Palmitic acid (Kahlbaum's). | 62·5 | 62·7 | 62·5 | 62·5 |

In the majority of cases we find that the melting point of fats, as determined by this method, is the temperature at which the substance becomes entirely liquefied, if maintained at this temperature for some time. In some cases, however, the fat only melts completely within a range of temperature and consequently the melting point recorded by our method is the lowest temperature at which any of the substance is converted into the true liquid state. Nevertheless we suggest that this temperature be recorded as the melting point of the substance.—H. D.

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### DISCUSSION.

The CHAIRMAN said he should like to know how the process answered in the case of mixtures, such as, for instance, the solid paraffins of commerce, which were mixtures of hydrocarbons of various melting points. It would be interesting to learn whether the authors had determined the applicability of their process in such a case. Further, would the authors take, in such a case, as a melting point the fusing temperature of the most fusible of the hydrocarbons under consideration. If so, he would point out that there might be present, even accidentally, a small proportion of a readily fusible substance. He thought they must all agree, however, that the authors' experiment had proved their method to give an extremely sharp definition of the melting point found.

Mr. E. GRANT HOOPER considered that there was a possibility of the method being extremely useful in connection with the substances to which the Chairman had referred. In specifications of petroleum jellies it was frequently laid down that the substance should not melt below a certain point. If such specifications further defined the melting point as that arrived at by such a process as the one brought before them to-night, concordant results would probably be arrived at, as, in all cases, a definite process would be employed.

Mr. OTTO HEHNER desired to point out that what was called the melting point of a fat was not, properly speaking, a melting point at all. There was no difficulty as a rule in determining the melting point of a pure substance, but natural fats consisted of a number of different substances, some fluid, some solid, and they had to determine the point at which liquefaction took place. But that was a different thing from the melting point; it was really the point where solution of the solid in the liquid fats were complete which was usually determined. There could be no doubt, however, about the ingenuity of the authors' process and the very excellent results which it gave. Referring further to the point raised by the Chairman, he would say that in attempting to arrive at the melting point of a mixture, say of cocoanut oil and stearine—if these substances were dissolved in each other at a suitable temperature, so as to form a homogeneous mixture, and then cooled, and an attempt made to determine the "melting point" by this method, at the temperature of the melting point slightly above that of cocoanut oil, the whole mass would become fluid without being clear, and the temperature would have to be raised by nearly 20° before the solution was complete.

Dr. A. W. CROSSLEY said, in reply, that with regard to the point raised by the Chairman, he could only say that he had had no experience with the substances named, but if one tried to determine the melting-point of any of those substances by this method one would get a number—a temperature—and it would be the lowest temperature at which any of the substances was converted into a true liquid state. The authors did not propose that the method should be used for determining the melting-points of substances other than fats and waxes, in dealing with which the filling of the ordinary capillary tube became, to say the least, difficult and tiresome. He quite agreed with Mr. Hehner that the melting-point of a mixture must be an arbitrary point. But was it not less so by this method than by the other methods now in use? And were the so-called melting-points hitherto obtained as concordant and satisfactory as those he had shown? If not, he thought he was justified in claiming that the method introduced to their notice was an improvement.

## THE CHEMICAL AND PHYSICAL CHARACTERS OF SOME INDIAN EDIBLE OILS.

BY ARTHUR W. CROSSLEY, M.SC., PH.D., AND HENRY R. LE SUEUR, B.SC.

THIS inquiry was undertaken for the purpose of comparing the chemical and physical properties of the more important Indian edible oils, with those of similar oils produced by other countries. The samples examined were collected under the supervision of the Reporter on Economic Products to the Government of India for the scientific and technical department of the Imperial Institute, and were handed to us for investigation by Prof. Dunstan, at whose suggestion the inquiry was undertaken. Many of the oils are well-known, but some of them (*e.g. Carthamus tinctorius* or safflower oil) have, so far as we have been able to ascertain, not been previously examined and therefore their properties are of special interest. As every sample is authenticated, the genuineness of which is therefore undoubted, we think the results of our inquiry are of sufficient interest and value to place on record. The expenses incurred in this inquiry have been defrayed by a grant from the Government Grant Committee of the Royal Society, for which we take this opportunity of recording our thanks.

The following information as regards the collection and preparation of the samples, and of the percentage yield of oil from the various Indian seeds, has been obtained from the Officiating Reporter on economic products to the Government of India.

### *Collection and Preparation of the Oils.*

As a general rule the country oil-mill or "Kohlu" was used for the extraction of the oil. This consists of two parts: (1) The mortar which is fixed, containing the seed; and, (2) A piece of wood like a large pestle, which is made to rotate inside the mortar and crush the contents. The mortar is constructed of stone or wood and the pestle is worked by one or two bullocks, according to the size of the mill. The seeds are decorticated and placed in the mortar-like vessel and are then ground to a paste and the oil as it separates is allowed to run off through an aperture below, and decanted from dregs, if there be any, but no process of filtering is resorted to.

In one or two cases a difficulty was experienced, where the oil had a thick consistency at ordinary temperatures, and could not readily be separated in the usual way. The Forest Ranger, Koderma, Bengal, for instance, heated the powdered kernels of *buchanania latifolia* in an earthen pot, by placing it over another pot in which water was kept boiling. The pasty mass was taken out, placed in a bag, and pressed firmly between two planks. This crude process gave a fairly satisfactory yield of oil.

As another instance, in Rajamundry, Madras, the kernels of *buchanania latifolia* were boiled with water, and the oil, which collected on the surface, was removed. In Baraich, North-West Provinces, the addition of hot water to buchanania seeds as they were being pressed in the mill was found to promote the separation of the oil.

The following table gives the percentage yield of oil from the various seeds:—

| No. of Oil. | Name of Oil. | Whence obtained. | Percentage Yield. |
|---|---|---|---|
| 59 | Bassia latifolia | North-West Provinces | 28·3 |
| 60 | " " | Central Provinces | 30·7 |
| 54 & 55 | " " | Bengal | 33·3 |
| 62 | " " | Bombay | 37·7 |
| 9 | Brassica alba | Bengal | 33·6 |
| 19 | " nigra | " | 28·5 |
| 18 | " napus | " | 32·5 |
| 17 | " juncea | " | 22·2 |
| 13 | " campestris | Punjab | 33·3 |
| 16 | " juncea | Bombay | 21·1 |
| 64 & 65 | Buchanania latifolia | North-West Provinces | 30—40 |
| 27 | Carthamus tinctorius | Hyderabad | 23·7 |
| 67 | Cocos nucifera | Bengal | 52·3 |
| 68 | " " | Bombay | 57·1 |
| 34 | Eruca sativa | " | 25·0 |
| 35 | " " | Punjab | 12·5 |
| 36 | " " | Cawnpur | 25·3 |
| 69 | Garcinia indica | Bombay | 33·3 |
| 37 | Guizotia abyssinica | " | 23·8 |
| 39 | " " | Bengal | 26·0 |
| 41 | Juglans regia | Punjab | 50·0 |
| 44 | Linum usitatissimum | Central Provinces | 28·2 |
| 48 | Papaver somniferum | Punjab | 47·0 |
| 57 | " " | Bengal | 33·3 |
| 49 & 50 | " " | North-West Provinces | 39·6 |

It may here be mentioned that nearly all of the oils examined could be got in quantity if desired; it is simply a question of demand.

The liquid oils, with but one or two exceptions, were perfectly clear, and were not therefore submitted to any process of purification before analysis. When this was not the case the sample was first freed from foreign matter by filtration. The solid oils were in all cases melted, filtered, and dried before analysis. All determinations have been carried out by ourselves, with the greatest care and regard to accuracy, the figures given being in many cases the mean of two or more closely agreeing numbers.

### *Analytical Methods employed.*

The methods employed for determining the constants of the oils are those in ordinary use, and, generally speaking, we have followed the details as given in "The Chemical Analysis of Oils, Fats, and Waxes," by Lewkowitsch. It is therefore unnecessary to go into any detail on this subject. In some few cases, however, slight alterations have been made, and these may be here mentioned.

*Specific Gravity.*—This constant was determined by means of a Sprengel tube, as advocated by Archbutt and Allen (this Journal, 1883, 54—55). We find that extremely accurate and concordant results may be obtained in this manner, and with a little practice the manipulation becomes extremely easy.

It is, we think, to be regretted that there is no one universally recognised temperature for the determination of specific gravity. One result of this is that the specific gravities of oils and fats, as determined by various investigators, are not strictly comparable, not only on account of the different temperatures employed, but also on account of the fact that in some cases the specific gravity of an oil has been determined at 15° C., and then compared to water at 0° as unity.

For liquid oils we have chosen the temperature of 15·5° C., and for solid oils the temperature of 100° C., in each case taking water at the same temperature as unity. This latter seems to be a particularly suitable temperature for solid oils, being easily obtained by submerging the Sprengel tube in boiling water, since an alteration of 27 mm. in the atmospheric pressure is necessary to alter the boiling point of water 1° C.

*Melting Point.*—The melting point of the solid oils was determined by a new method devised by us and described in another short communication to this Society (this Journal, 1898, 82). The method gives reliable results, and its extreme simplicity is a point greatly in its favour.

*Insoluble Fatty Acids.*—In carrying out this determination we encountered two difficulties:—First, the securing of a filter paper which would completely retain the insoluble acids, and second, the drying of the fatty acids. The filter paper which was found in the end to give the greatest satisfaction was Schleicher and Schul's frosted hard paper (No. 602). Regarding the second difficulty, we quote the following instructions as given in Lewkowitsch's book, page 160:—"The washing of the acids being completed, immerse the funnel with the filter in a vessel of cold water, so that the water outside and the acids inside are at the same level. As a rule, the fatty acids will solidify. Allow the water then to drain off, transfer the filter to a tared beaker and dry at 100° for 2 hours. Weigh accurately, dry for another 2½ hours, and weigh again. *The difference between the weights will, as a rule, be below 1 mgrm.*" At a very early stage in our investigation, we did away with the use of a beaker, as we found it extremely difficult to get accurate weighings, on account of the absorption of moisture by the filter paper. In place of a beaker we used an ordinary stoppered weighing bottle, sufficiently large to easily hold the filter paper employed. Again, the length of time given for drying the acids at 100° is not necessary and, in our opinion, may lead to erroneous results, for we invariably found, that the second weighing, *i.e.*, after in all 4½ hours drying, was much higher than the weight taken after 2 to 2½ hours drying, due conceivably to the oxidation of the fatty acids. As a matter of fact, we afterwards assured ourselves, that in some cases drying for 2½ hours at 100° is more than sufficient to drive off all the water. We finally adopted the following procedure:—The acids were dried in a weighing bottle at a temperature of 100° for 1½ hours, and after cooling and weighing, were again dried for periods of ½ hour at a time; weighings being taken at the end of each period. In most cases the acids were dry after the third weighing, that is, after 2½ hours drying, and in some cases after the second weighing or 2 hours drying, whilst in all cases the acids had gained in weight at the fourth weighing or after drying for 3 hours. In support of the above statements we quote the following weighings taken from some of our experiments:—

| | | | | |
|---|---|---|---|---|
| Weighing bottle, filter paper and acids:— | | | | |
| 1½ hours drying....... | 45·6408 | 31·2859 | 47·7408 | 45·5140 |
| " + ½ drying... | 45·3584 | 31·0950 | 47·6504 | 45·4200 |
| " " + ½ drying | 45·3200 | 31·0970 | 47·6388 | 45·4102 |
| " " " + ½ drying | 45·3240 | .. | 47·6452 | 45·4132 |

*Viscosity.*—This constant was determined by means of a Boverton Redwood viscometer, kindly placed at our disposal by Mr. Boverton Redwood, to whom we beg to tender our sincere thanks. In the case of liquid oils the determinations were made at a temperature of 70° F., and of solid oils, at 140° F. The results are given in two columns; the first expresses the number of seconds required for the flow of 50 c.c. of the oil at the given temperature; and the second, the ratio of this number to the number of seconds required for the flow of 50 c.c. of water at the same temperature (25·4 seconds at 70° F. and 22·9 at 140° F.).

A very interesting point comes out when one compares the viscosity numbers with the iodine values of the various oils, and there appears to be an undoubted connection between the two, thus, the higher the iodine value the lower the viscosity, that is the less the number of seconds required for the flow of 50 c.c. of the oil, as will be readily seen from the following numbers:—

| — | Iodine Value. | Viscosity. | |
|---|---|---|---|
| | | No. of Seconds. | Compared to Water. |
| Ground nut................ | 92·43 | 429·3 | 16·90 |
| " ................ | 98·42 | 347·0 | 13·67 |
| " ................ | 98·47 | 350·1 | 13·78 |
| " ................ | 104·82 | 306·9 | 12·10 |
| Mexican poppy........... | 119·91 | 272·0 | 10·71 |
| " " ........... | 122·53 | 268·9 | 10·59 |
| Rape seed................ | 94·10 | 464·6 | 18·29 |
| " ................ | 96·66 | 413·8 | 16·29 |
| " ................ | 96·75 | 402·0 | 15·82 |
| " ................ | 96·25 | 393·2 | 15·48 |
| " ................ | 101·82 | 379·3 | 14·93 |
| " ................ | 104·84 | 371·8 | 14·64 |

A possible explanation of this is to be sought in the fact that the higher the iodine value of an oil the greater the percentage of unsaturated acids and also of the degree of unsaturation of these acids. As, therefore, generally speaking, the higher unsaturated acids are liquids not readily solidifying when cooled, one would naturally expect the viscosity of an oil containing the glycerides of such acids to be less than that of an oil containing a large percentage of the glycerides of the saturated acids. No doubt other factors have to be taken into account when considering this subject, to which we hope to give further attention. We have been unable to find any previous mention of this apparent connection between viscosity and iodine value.

*Rotatory Power.*—Little work seems to have been done in this direction, but still we thought it might prove interesting to ascertain whether any of the oils produced a considerable rotation or not. The instrument used was a Laurent half-shadow polariscope, made by Schmidt and Haensch, and the determinations were carried out in a 20 cm. tube at the ordinary temperature. None of the oils examined show a marked rotation, but the majority have a slight effect on polarised light, due possibly to the presence of a trace of some optically active substance in the oils rather than to a true property of the oils themselves.

*Drying Properties.*—For this determination a thin film of the oil was spread on a ground glass plate, and heated over a water bath for six hours. According to the hardness of the film the oils were divided into three classes, drying, semi-drying, and non-drying. The results are given under the characteristics and properties of the oils.

### *Characteristics and Properties of the Oils.*

Under this heading we give the general properties, *i.e.*, appearance, taste, etc. of the oils, and, in those cases where we find that the oils are only slightly noticed or not mentioned at all in Lewkowitsch's book, we give all the information at our command. Much of this information, especially as regards the usage of the oils in India, is taken from Watt's "Dictionary of the Economic Products of India."

*Amoora Rohituka* (No. 1).—The seeds of this evergreen tree yield, an oil which is used by the natives for various economic purposes, such as illuminating and medicinally. The sample examined by us is clear and of a reddish-brown colour, without any particular taste, and a slight odour resembling that of linseed oil. It is a drying oil and its constants approximate to those of Niger seed.

*Anacardium occidentale* (No. 2). Cashew nut.—Two distinct oils are produced from this plant, the one, pressed from the kernels, is a light yellow bland oil, the other, known as Cardole or Cashew apple oil, is prepared from the pericarp or shell of the nut, and is black, acrid and powerfully vesicating. The sample examined apparently belongs to the latter class it is clear and of a very dark reddish-brown colour. Though it possesses the abnormal iodine value of 294·6 (mean of three determinations) practical experiment shows it to be a non-drying oil.

*Arachis hypogea* (Nos. 3—6). Earthnut or peanut—The four sample are all of a very pale yellow colour, with little taste or smell, and deposit solid matter on standing. It will be noticed that the specific gravity is higher than generally recorded (0·9200—0·9256, highest previously recorded, 0·9200), approaching rather closely to that of poppy-seed oil (0·925—0·926), with which arachis oil is said to be adulterated. The iodine value, however, is normal, *i.e.*, below 100, whereas the iodine value of poppy seed oil is over 130. It is hardly necessary to again point out that the genuineness of our samples is undoubted.

*Argemone mexicana* (Nos. 7 and 8). Mexican or prickly poppy.—The spiny herbaceous annual from the seeds of which this oil is extracted is very abundant in India, and practically an unlimited amount of the oil is available. Of the two samples, one, No. 7, is of a dark orange colour, and possesses a slight but distinct smell. The other, No. 8, is greenish-brown in colour, contains a very large percentage of free fatty acids and is completely soluble in 9—10 volumes of absolute alcohol at the ordinary temperature, whereas No. 7 is not soluble even at a temperature of 40° C. We mention this fact, because Charbonnier has stated, that this oil is soluble in 5—6 volumes of alcohol at 32·2° C., a statement which has been refuted by Flückiger. It is evidently quite possible for both statements to be correct. Both our samples come under the heading of drying oils.

*Bassia latifolia* (Nos. 54—62). Mahua butter.—Our samples of this oil, which is also known as Illipe butter, possess in the solid state a yellow colour, one sample, No. 58, having a decided green tinge; when liquefied they are clear and vary in colour from yellow to orange. This oil is not identical with, and should not be mistaken for, Mohwah butter or Mowrah seed oil, which is extracted from the seeds of *Bassia longifolia*, nor with Phulwara butter, which is obtained from *Bassia butyracea*, or Indian butter tree.

*Brassica* family (Nos. 9—20).—No. 9, *Brassica alba* or white mustard, is of a clear pale yellow colour and smells of mustard seed oil. Its constants are almost identical with those of the other members of this family.

*Brassica campestris* (Nos. 10—15) include specimens of all the varieties produced in India. They are clear oils, varying in colour from yellow to pale brown, and possess the characteristic mustard smell.

*Brassica juncea* (Nos. 16 and 17) is largely used in India as an article of food and also medicinally. They are clear yellow oils, possessing a distinct odour and taste.

*Brassica napus* (No. 18), Lutni mustard, is clear and light-brown in colour.

*Brassica nigra* (Nos. 19 and 20) are clear and dark orange coloured.

The whole (Brassica) family belong to the class of semi-drying oils.

*Buchanania latifolia* (Nos. 64 and 65).—This oil is rarely prepared, as the seeds are much prized by the natives of India as a sweetmeat. The constants of the specimens, which in the solid state are orange and in the liquid state clear orange and pale brown respectively, agree closely with those of Bassia latifolia.

*Carthamus tinctorius* (Nos. 21 to 32).—Safflower or Saffron oil is extracted from an herbaceous plant, which until recently was cultivated all over India for the preparation of the well-known dye. Two distinct varieties of plants are grown, the spiny or thorny variety and the spineless or thornless variety. The oil is obtained from two kinds of fruit, one the cultivated, a white and glossy form, the other smaller, and of a mottled brown grey and white appearance. Two methods are employed for the preparation of the oil, the ordinary cold dry expression method and the so-called hot extraction process, which latter is in reality a crude process of downward distillation. The first method yields an oil which is largely used in India for burning, whereas that obtained by the latter process is useless for this purpose, and is employed by the natives for the preservation of leathern vessels, ropes, &c., exposed to the action of the water. The samples (21—32) include all the different varieties prepared in India. They are, with the exception of No. 22, clear, and vary in colour from very pale yellow to orange, possess a faint odour and taste, neither of which is particularly characteristic. They rotate the plane of polarisation slightly to the right, and in general resemble the white poppy-seed and walnut oils.

Safflower is an exceedingly good drying oil, and its chemistry is being thoroughly investigated. No. 22, prepared by the hot extraction process, is quite black in appearance and has a decided pitchy smell. Its specific gravity is much higher and its iodine value lower than any of the other samples. It is a non-drying oil.

The variety *oxyacantha* (No. 33) differs but slightly from the *tinctorius*.

*Cocos nucifera* (Nos. 66 to 68), Cocoanut palm.—The constants of these Indian specimens agree with those on record for this oil, except the Reichert-Meissel value, which is markedly higher (6·65—6·79: previously recorded, 3·5—3·7.) In the solid state, Nos. 66 and 67 are white, with a faint yellow tinge, and in the liquid state clear and pale yellow in colour; No. 68 is pure white, and when liquefied clear and colourless.

*Eruca sativa* (Nos. 34 to 36).—This oil is obtained from an extensively cultivated herb, closely allied to the mustards. In India it is used for burning purposes and also to some extent as a food. The physical constants resemble those of the oil yielded by *Brassica campestris*. All three samples are clear and vary in colour from yellow to pale brown; two of them, Nos. 34 and 36, have a well marked odour and flavour of turnip, whereas No. 35 has an odour of mustard and a rather pleasant taste. Eruca sativa is a non-drying oil.

*Garcinia indica* (No. 69).—This oil was sent to us in egg-shaped masses of a dirty white colour containing a good deal of foreign matter. After purification it was of a pale brown colour when solid, and melted to a clear brown liquid.

*Guizotia abyssinica* (Nos. 37 to 40), Niger seed.—The samples of this drying oil are clear and vary in colour from pale yellow to orange.

*Juglans regia* (No. 41), Walnut tree.—The sample is clear and of an orange yellow colour, with a slight odour of linseed and a sickly nutty flavour. Its constants agree well with those previously recorded, and are almost identical with those of carthamus tinctorius. Practical experiment shows it to be a strong drying oil.

*Lepidium sativum* (Nos. 42 and 43), Garden cress.—The samples of this oil are of an orange colour and possess a faint smell. The difference in their iodine values is marked; they belong to the class of semi-drying oils.

*Liquid Oils.*

| No. | Scientific Name. | English Name. | Locality. | Remarks. | Specific Gravity at 15·5° C. Water at 15·5° C. = 1·0000. | Acid Value. | | Saponification Value, Mgrms. KOH. | Iodine Value, per Cent. | Reichert-Meissl Value, c.c.'s $\frac{1}{10}$ N KOH. | Insoluble Fatty Acids, per Cent. | Optical Activity in 20 cm. Tube. | Viscosity. | | Refraction. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Mgrms. KOH for 1 grm. of Oil. | Oleic Acid, per Cent. | | | | | | No. of Seconds for Flow of 50 c.c. at 70° F. | Compared to Water at 70° F. | Scale Division Readings. | Calculated Refractive Indices. |
| 1 | Amoora rohituka | Evergreen tree | Bengal | .. .. | 0·9386 | 17·03 | 4·28 | 189·70 | 134·86 | 1·64 | 93·23 | .. | 375·8 | 14·79 | 64·5 | 1·4687 |
| 2 | Anacardium occidentale | Cashew nut | Madras | .. .. | 0·9594 | .. | .. | 45·10 | 294·26 | 1·26 | .. | .. | .. | .. | too high for scale. | |
| 3 | Arachis hypogaea | Ground, earth, or pea nut. | „ | No. 1 | 0·9223 | 5·76 | 1·45 | 190·10 | 98·47 | 0·00 | 95·63 | − 7′ | 350·1 | 13·78 | .. | .. |
| 4 | „ „ | „ „ | „ | No. 2 | 0·9223 | 9·60 | 2·42 | 185·60 | 98·42 | 0·00 | .. | +24′ | 347·0 | 13·67 | 57·5 | 1·4642 |
| 5 | „ „ | „ „ | „ | No. 3 | 0·9256 | 32·91 | 8·28 | 194·80 | 92·43 | 0·00 | .. | Inactive | 429·3 | 16·90 | .. | .. |
| 6 | „ „ | „ „ | „ | .. .. | 0·9195 | 27·02 | 6·80 | 192·10 | 100·82 | 0·00 | .. | − 7′ | 306·9 | 12·10 | .. | .. |
| 7 | Argemone mexicana | Mexican or prickly poppy. | Bengal | .. .. | 0·9247 | 6·00 | 1·51 | 190·30 | 122·53 | 0·00 | 95·07 | .. | 268·9 | 10·59 | 62·5 | 1·4675 |
| 8 | „ „ | „ „ | „ | .. .. | 0·9259 | 83·90 | 21·13 | 187·80 | 119·91 | 0·00 | .. | .. | 272·0 | 10·71 | .. | .. |
| 9 | Brassica alba | White mustard | .. | .. .. | 0·9142 | 5·40 | 1·36 | 171·20 | 96·75 | 0·00 | 95·86 | − 9′ | 402·0 | 15·82 | 58·5 | 1·4649 |
| 10 | „ campestris | Rape seed | N.W. Provinces | Variety red | 0·9148 | 2·90 | 0·73 | 171·60 | 99·20 | 0·79 | 96·30 | − 7′ | 390·6 | 15·38 | .. | .. |
| 11 | „ „ | „ „ | „ „ | „ glauca | 0·9142 | 1·78 | 0·45 | 171·40 | 97·71 | 0·67 | 95·04 | −10′ | 402·6 | 15·85 | 59·2 | 1·4653 |
| 12 | „ „ | „ „ | „ „ | „ dichotomo | 0·9154 | 1·57 | 0·39 | 172·20 | 104·84 | 0·22 | 95·57 | .. | 371·8 | 14·64 | .. | .. |
| 13 | „ „ | „ „ | Punjab | .. .. | 0·9163 | 2·57 | 0·65 | 173·40 | 96·25 | 0·43 | 94·56 | .. | 393·2 | 15·48 | .. | .. |
| 14 | „ „ | „ „ | Bombay | Variety brown | 0·9171 | 4·00 | 1·00 | 172·80 | 94·10 | 0·00 | .. | .. | 464·6 | 18·29 | .. | .. |
| 15 | „ „ | „ „ | „ | „ yellow | 0·9141 | 1·42 | 0·36 | 169·40 | 96·66 | 0·00 | .. | − 5′ | 413·8 | 16·29 | .. | .. |
| 16 | „ juncea | Indian mustard | „ | „ mustard | 0·9206 | 3·72 | 0·94 | 180·10 | 108·29 | 0·89 | .. | −25′ | 382·8 | 15·08 | .. | .. |
| 17 | „ „ | „ „ | Bengal | „ rai | 0·9158 | 7·12 | 1·79 | 172·10 | 101·82 | 0·33 | 95·49 | 18′ | 379·3 | 14·93 | 60·0 | 1·4659 |
| 18 | „ napus | Lutni „ | „ | .. .. | 0·9146 | 3·77 | 0·95 | 167·70 | 97·70 | 0·00 | 95·55 | −15′ | 398·0 | 15·67 | 58·8 | 1·4650 |
| 19 | „ nigra | Black „ | „ | .. .. | 0·9155 | 7·35 | 1·85 | 173·30 | 98·84 | 0·03 | 96·05 | −17′ | 425·4 | 16·75 | 59·5 | 1·4655 |
| 20 | „ „ | „ „ | C. Provinces | .. .. | 0·9185 | 5·72 | 1·44 | 173·00 | 110·51 | 0·00 | .. | −30′ | 355·4 | 13·99 | .. | .. |
| 21 | Carthamus tinctorius | Safflower | „ | .. .. | 0·9267 | 10·41 | 2·62 | 192·50 | 140·05 | .. | 95·30 | + 8′ | 256·1 | 10·08 | 65·2 | 1·4692 |
| 22 | „ „ | „ | Bengal | Distilled | 0·9634 | .. | .. | 188·00 | 128·90 | .. | .. | .. | .. | .. | .. | .. |
| 23 | „ „ | „ | „ | Pressed oil | 0·9264 | 3·68 | 0·92 | 193·30 | 144·40 | .. | .. | + 7′ | 247·8 | 9·76 | .. | .. |
| 24 | „ „ | „ | Punjab | .. .. | 0·9275 | 8·28 | 2·08 | 186·60 | 138·64 | 0·00 | .. | .. | 274·5 | 10·81 | .. | .. |
| 25 | „ „ | „ | „ | Oil from cultivated form. | 0·9267 | 0·76 | 0·19 | 189·60 | 144·05 | .. | .. | +10′ | 249·1 | 9·81 | .. | .. |
| 26 | „ „ | „ | „ | Oil from wild form | 0·9253 | 2·90 | 0·74 | 187·40 | 129·80 | .. | .. | +12′ | .. | .. | .. | .. |
| 27 | „ „ | „ | Hyderabad | Cold dry expression | 0·9277 | 2·58 | 0·65 | 187·90 | 138·60 | .. | .. | +14′ | 274·7 | 10·79 | .. | .. |
| 28 | „ „ | „ | Bombay | Oil from dye plant | 0·9262 | 2·67 | 0·67 | 187·20 | 143·40 | .. | .. | + 8 | 250·3 | 9·85 | 65·2 | 1·4692 |
| 29 | „ „ | „ | „ | „ „ oil seed plant | 0·9269 | 6·20 | 1·56 | 191·30 | 149·93 | 0·00 | .. | + 4′ | 249·6 | 9·83 | .. | .. |
| 30 | „ „ | „ | Cawnpore | Variety thornless | 0·9271 | 5·92 | 1·49 | 189·40 | 141·42 | 0·00 | .. | + 7′ | 258·8 | 10·19 | .. | .. |
| 31 | „ „ | „ | „ | „ thorny | 0·9251 | 20·02 | 5·04 | 190·52 | 141·12 | .. | .. | .. | 243·0 | 9·57 | .. | .. |
| 32 | „ „ | „ | Madras | .. .. | 0·9280 | 4·12 | 1·04 | 192·42 | 142·85 | .. | .. | + 6′ | 268·8 | 10·58 | .. | .. |
| 33 | „ oxyacantha | „ | Punjab | .. .. | 0·9270 | 3·66 | 0·92 | 189·40 | 135·49 | .. | 95·44 | + 7′ | 294·0 | 11·57 | 68·2 | 1·4711 |
| 34 | Eruca sativa | .. .. | Bombay | Allied to mustard | 0·9177 | 2·10 | 0·53 | 170·40 | 99·72 | 0·66 | 95·49 | .. | 371·0 | 14·61 | .. | .. |
| 35 | „ „ | .. .. | Punjab | „ „ | 0·9152 | 3·70 | 0·93 | 169·00 | 97·41 | 0·11 | .. | −11′ | 405·8 | 15·98 | 59·2 | 1·4653 |
| 36 | „ „ | .. .. | Cawnpore | „ „ | 0·9165 | 2·51 | 0·63 | 174·10 | 99·10 | 0·77 | .. | −18′ | 369·4 | 14·54 | .. | .. |
| 37 | Guizotica abyssinica | Niger seed | Bombay | .. .. | 0·9263 | 9·75 | 2·45 | 191·80 | 126·60 | 0·23 | 94·11 | Inactive | 292·6 | 11·52 | .. | .. |
| 38 | „ „ | „ „ | Madras | .. .. | 0·9251 | 5·21 | 1·31 | 191·30 | 131·70 | 0·11 | .. | Inactive | 263·1 | 10·36 | .. | .. |
| 39 | „ „ | „ „ | Bengal | .. .. | 0·9248 | 11·69 | 2·94 | 188·90 | 128·60 | 0·11 | .. | Inactive | 283·7 | 11·17 | 63·0 | 1·4678 |
| 40 | „ „ | „ „ | C. Provinces | .. .. | 0·9263 | 7·05 | 1·77 | 192·20 | 133·80 | 0·63 | .. | +18′ | 273·9 | 10·78 | .. | .. |
| 41 | Juglans regia | Walnut tree | Punjab | .. .. | 0·9259 | 10·07 | 2·53 | 192·50 | 143·10 | 0·00 | 95·44 | +16′ | 231·8 | 9·13 | 64·8 | 1·4689 |
| 42 | Lepidium sativum | Garden cress | Bengal | .. .. | 0·9210 | 10·55 | 2·66 | 181·50 | 101·72 | 0·44 | .. | Inactive | .. | .. | 60·5 | 1·4622 |
| 43 | „ „ | „ „ | Punjab | .. .. | 0·9221 | 4·18 | 1·05 | 183·10 | 118·40 | 0·22 | 95·57 | .. | 321·6 | 12·66 | .. | .. |
| 44 | Linum usitatissimum | Linseed | C. Provinces | .. .. | 0·9337 | 4·84 | 1·22 | 191·50 | 165·40 | 0·00 | 94·81 | + 6′ | 211·7 | 8·33 | 72·5 | 1·4740 |
| 45 | Luffa egyptica | .. .. | Bengal | .. .. | 0·9251 | 28·85 | 7·26 | 187·80 | 108·51 | 1·43 | 94·80 | .. | .. | .. | 62·0 | 1·4680 |
| 46 | Nigella sativa | Smallfennel | „ | .. .. | 0·9248 | 97·41 | 24·46 | 196·40 | 116·20 | 5·40 | 88·83 | .. | .. | .. | 58·5 | 1·4519 |
| 47 | Olea europea | Olive | Punjab | .. .. | 0·9163 | 5·06 | 1·27 | 190·90 | 93·67 | 0·60 | 95·14 | .. | 312·3 | 12·29 | 56·4 | 1·4635 |
| 48 | Papaver somniferum | Opium or white poppy | „ | .. .. | 0·9255 | 10·78 | 2·71 | 196·80 | 137·10 | 0·00 | .. | Inactive | 254·8 | 10·03 | .. | .. |
| 49 | „ „ | „ „ | N.W. Provinces | .. .. | 0·9260 | 6·35 | 1·60 | 190·10 | 133·70 | 0·00 | .. | Inactive | 259·1 | 10·21 | 63·4 | 1·4680 |
| 50 | „ „ | „ „ | „ „ | .. .. | 0·9268 | 11·02 | 2·77 | 190·60 | 133·90 | 0·00 | 94·97 | Inactive | 258·7 | 10·19 | .. | .. |
| 51 | „ „ | „ „ | Bengal | .. .. | 0·9255 | 8·68 | 2·18 | 189·00 | 134·00 | 0·00 | .. | + 4′ | 253·9 | 10·0 | .. | .. |
| 52 | Prunus armeniaca | Apricot kernel | Punjab | .. .. | 0·9195 | 12·14 | 3·05 | 188·00 | 96·02 | 0·00 | 95·40 | +11′ | .. | .. | 56·3 | 1·4634 |
| 53 | Raphanus sativus | Radish | Assam | .. .. | 0·9163 | 14·47 | 3·64 | 173·80 | 92·85 | 0·33 | 95·94 | +17′ | 385·3 | 15·17 | 57·5 | 1·4642 |

*Solid Oils.*

| No. | Scientific Name. | English Name. | Locality. | Remarks. | Specific Gravity at 100° C. Water at 100° C. =1·0000. | Acid Value. Mgrms. KOH for 1 grm. of Oil. | Acid Value. Oleic Acid, per Cent. | Saponification Value, Mgrms. KOH. | Iodine Value, per Cent. | Reichert-Meissel Value, c.c.'s $\frac{1}{10}$N KOH. | Insoluble Fatty Acids, per Cent. | Melting Point. | Viscosity. No. of Seconds for Flow of 50 c.c. at 140° F. | Viscosity. Compared to Water at 140° F. | Refraction. Scale Division Readings. | Refraction. Calculated Refractive Indices. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 54 | Bassia latifolia .......... | Butter or mahua..... | Bengal ........ | .. .. | 0·8975 | 11·79 | 2·97 | 194·00 | 62·11 | 0·44 | 94·95 | 24·5 | 97·1 | 4·24 | .. | .. |
| 55 | „ „ .......... | „ „ ..... | „ ........ | .. .. | 0·8962 | 19·15 | 4·80 | 192·60 | 64·88 | .. | .. | 26·0 | 93·8 | 4·10 | 51·8 | 1·4605 |
| 56 | „ „ .......... | „ „ ..... | Madras ........ | .. .. | 0·8981 | 10·33 | 2·60 | 191·80 | 67·85 | .. | .. | 25·0 | 96·9 | 4·23 | .. | .. |
| 57 | „ „ .......... | „ „ ..... | „ ........ | .. .. | 0·8970 | 4·83 | 1·23 | 190·90 | 58·53 | .. | .. | 26·5 | 107·0 | 4·67 | .. | .. |
| 58 | „ „ .......... | „ „ ..... | „ ........ | .. .. | 0·8964 | 8·67 | 2·16 | 187·40 | 58·45 | .. | 94·69 | 29·0 | 100·6 | 4·39 | .. | .. |
| 59 | „ „ .......... | „ „ ..... | N.W. Provinces | .. .. | 0·8969 | 21·20 | 5·33 | 189·50 | 63·51 | .. | .. | 23·0 | 93·9 | 4·10 | .. | .. |
| 60 | „ „ .......... | „ „ ..... | C. Provinces ... | .. .. | 0·8971 | 17·05 | 4·29 | 188·80 | 63·01 | .. | .. | 24·0 | 96·7 | 4·22 | .. | .. |
| 61 | „ „ .......... | „ „ ..... | Calcutta ....... | .. .. | 0·8943 | 70·82 | 17·83 | 193·20 | 58·69 | 0·88 | .. | 25·5 | 90·4 | 3·95 | .. | .. |
| 62 | „ „ .......... | „ „ ..... | Bombay ....... | .. .. | 0·8980 | 6·83 | 1·72 | 190·50 | 53·43 | .. | .. | 24·0 | 96·9 | 4·23 | 52·4 | 1·4609 |
| 63 | „ butyracea ........ | Indian butter........ | N.W. Provinces | .. .. | 0·8970 | 16·44 | 4·14 | 190·80 | 42·12 | 0·44 | 94·86 | 39·0 | 110·4 | 4·82 | 48·2 | 1·4570 |
| 64 | Buchanania latifolia ..... | .. .. | „ „ | .. .. | 0·8941 | 15·03 | 3·78 | 195·40 | 54·73 | 0·33 | 95·80 | 32·0 | 89·3 | 3·90 | 48·8 | 1·4584 |
| 65 | „ „ .... | .. .. | „ „ | .. .. | 0·8943 | 40·11 | 10·09 | 191·80 | 59·92 | .. | 94·87 | 32·0 | 84·6 | 3·69 | .. | .. |
| 66 | Cocos nucifera........... | Cocoa palm ......... | Malabar ....... | .. .. | 0·9030 | 35·21 | 8·86 | 258·20 | 8·54 | 6·71 | .. | 23·5 | 63·9 | 2·79 | .. | .. |
| 67 | „ „ .......... | „ „ ......... | Bengal......... | .. .. | 0·9040 | 11·84 | 2·98 | 255·60 | 8·41 | 6·79 | 82·35 | 24·5 | 64·7 | 2·83 | .. | .. |
| 68 | „ „ .......... | „ „ ......... | Bombay ....... | .. .. | 0·9042 | 9·95 | 2·50 | 255·50 | 8·25 | 6·65 | .. | 25·0 | 64·5 | 2·82 | 34·2 | 1·4482 |
| 69 | Garcinia indica.......... | Kokam butter....... | „ ....... | .. .. | 0·8889 | 14·15 | 3·56 | 186·80 | 34·21 | 0·11 | 94·59 | 42·0 | 101·1 | 4·41 | 46·0 | 1·4565 |
| 70 | Vateria indica........... | White dammar....... | Madras ........ | Piney tallow ........ | 0·8907 | 5·18 | 1·30 | 189·29 | 39·63 | 0·44 | 95·21 | 37·5 | 104·0 | 4·54 | 47·5 | 1·4575 |
| 71 | „ „ ........... | „ „ ....... | „ ........ | „ „ ........ | 0·8900 | 15·34 | 3·86 | 188·70 | 37·82 | 0·22 | 95·14 | 37·0 | 101·5 | 4·43 | .. | .. |

The following table, giving the principal uses to which these oils are put in India, is included in our paper in the hope that it may be of some service in pointing out the possible uses of these oils, more especially of those which appear to be little known in this country :—

| —— | Edible. | Illuminant. | Soap Manufacture. | Medicinal Purposes. | —— | Edible. | Illuminant. | Soap Manufacture. | Medicinal Purposes. |
|---|---|---|---|---|---|---|---|---|---|
| Amoora rohituka ................................ | .. | I | .. | M | Cocos nucifera (cocoa palm)......................... | E | I | S | M |
| Anacardium occidentale (cashew nut) ............. | E | .. | .. | M | Eruca sativa............................................ | E | I | .. | .. |
| Arachis hypogaea (ground earth or pea nut)....... | .. | I | S | M | Guizotica abyssynica (Niger seed) ................. | E | I | .. | M |
| Argemone mexicana (Mexican or prickly poppy) .. | .. | I | .. | M | Garcinia indica (kokam butter)...................... | E | .. | S | M |
| Bassia latifolia (butter or mahua tree) ............ | E | I | S | M | Juglans regia (walnut tree) ......................... | E | .. | .. | .. |
| „ butyracea (Indian butter) ................. | E | I | S | M | Lepidium sativum (cress) ............................ | .. | .. | .. | .. |
| Brassica alba (white mustard) ..................... | .. | I | .. | .. | Linum usitatissimum (linseed)....................... | E | I | S | M |
| „ campestris (rape seed).................... | E | I | .. | .. | Luffa ægyptica.......................................... | .. | .. | .. | M |
| „ juncea (Indian mustard) ................. | E | .. | .. | M | Nigella sativa ........................................... | E | .. | .. | M |
| „ napus (lutni mustard)..................... | .. | .. | .. | .. | Olea europea (olive).................................. | E | .. | S | M |
| „ nigra (black „ ) ....................... | .. | .. | .. | M | Papaver somniferum (opium or white poppy) ...... | E | I | .. | M |
| Buchanania latifolia................................ | E | .. | .. | .. | Prunus armeniaca ...................................... | E | I | .. | .. |
| Carthamus tinctorius (safflower) ................... | E | I | .. | M | Raphanus sativus (radish) ........................... | E | I | .. | .. |
| „ oxyacantha ( „ ) ................. | E | I | .. | M | Vateria indica (white dammar or piney tallow) ..... | .. | I | S | .. |

Chemical Laboratory,
St. Thomas's Hospital.

*Linum usitatissimum* (No. 44), Linseed.—The sample is of a clear orange colour, and possesses an iodine value lower than is usually recorded (165·4 as against 170—180).

*Luffa Ægyptica* (No. 45).—The oil is extracted from the seeds of a genus of climbing plants belonging to the family Cucurbitaceæ. The sample is dark reddish brown in colour, possesses a slight smell, and is a semi-drying oil.

*Nigella sativa* (No. 46), Small fennel.—Two kinds of oil are expressed from the seeds of this plant; the one is described as dark, fragrant and volatile, and the other clear, nearly colourless, and of the consistency of castor oil. Our sample evidently belongs to the first class; it is of a dark reddish brown colour, with a strongly marked odour of eucalyptus, and is a semi-drying oil. On standing it deposits solid matter.

*Olea europea* (No. 47).—The sample is clear, of a greenish brown colour. Its iodine value is abnormally high (93·67 as against 77—88).

*Papaver somniferum* (Nos. 48 to 51), Opium or white poppy.—The oils vary in colour from pale yellow to orange, and possess a small but decided laevo-rotatory power. They are drying oils.

*Prunus armeniaca* (No. 52), Apricot kernel.—The oil is of a pale orange colour; it has a marked odour and flavour of bitter almonds, and is a semi-drying oil.

*Raphanus sativus* (No. 53), Radish seed.—The sample is clear, of a light brown colour, and has a distinct acrid flavour. It is a non drying oil.

*Vateria indica* (Nos. 70 and 71).—Both samples in the solid state are white with a greenish tinge, and when melted are clear and greenish yellow in colour. The constants resemble those of garcinia indica.

In the accompanying table, the oils are divided into three groups, according to their drying properties as found by actual experiment, and as indicated by their iodine values. (Drying = 120 and above; semi-drying = 95-120; non-drying below 95).

*Group I.—Drying.*

| Name of Oil. | From actual Experiment. | From Iodine Value. |
|---|---|---|
| Amoora rohituka................ | Drying | Drying |
| Argemona mexicana (mexican or prickly poppy). | " | " |
| Carthamus tinctorius (safflower). | " | " |
| Guizotica abyssynica (Niger seed). | " | " |
| Juglans regia (walnut) .......... | " | " |
| Linum usitatissimum (linseed) .. | " | " |
| Papaver somniferum (white poppy). | " | " |

*Group II.—Semi-drying.*

| | | |
|---|---|---|
| Brassica family (rape seed) ...... | Semi-drying | Semi-drying |
| Lepidium sativum (cress)........ | " | " |
| Luffa aegyptica ........ ......... | " | " |
| Prunus armeniaca (apricot kernel) | " | " |

*Group III.—Non-drying.*

| | | |
|---|---|---|
| Arachis hypogaea (ground nut) .. | Non-drying | Semi-drying |
| Eruca sativa ...................... | " | " |
| Nigella sativa...................... | " | " |
| Olea europea (olive) ............. | " | Non-drying |
| Raphanus sativus (radish) ....... | " | " |

### ADDENDUM.

*Refractive Index.**—Through the kindness of Mr. Otto Hehner, who placed his Zeiss butyro-refractometer at our disposal, we have been enabled to carry out this determination, and we wish to express here our most cordial thanks to Mr. Hehner.

The results are given in two columns added to the general tables: the one expresses the number of scale divisions for each oil, at a temperature of 40° C., for ordinary light. The determinations were also done using the sodium flame, without, however, any appreciable differences. The second column gives the actual refractive indices, calculated from the scale division readings, using the table given for this purpose, in Lewkowitsch's book, 2nd edition, p. 627.

On comparing the refractive indices and iodine values, it will be seen that the former afford, as a rule, a direct confirmation of the latter, as has been pointed out by Hehner and other observers. In our opinion, therefore, the refractive index cannot be looked upon as a factor which throws any new light on the nature of the oil; but as a confirmation of the accuracy of the iodine value, it may be considered of some importance.

### DISCUSSION.

Mr. E. **GRANT HOOPER** inquired if the authors had determined the refractions of the oils, particulars of which he did not see in the tabular statement before them. He would strongly recommend the determination of refractive power in any investigations connected with oils and fats, for such constants were both characteristic and readily ascertained.

Mr. **CROSSLEY** replied that he had not as yet done so.*

## NEW FILLING MATERIAL FOR REACTION TOWERS.

BY OSCAR GUTTMANN, ASSOC. M.I.C.E., F.I.C., F.C.S.

I HAVE pleasure in submitting to the notice of the Society some samples of a new filling material of my invention.

As you know, absorbing towers are intended to absorb gases by means of a liquid, and the usual means adopted is to cause water to trickle down such a tower within which the gases are ascending.

Fig. 1.

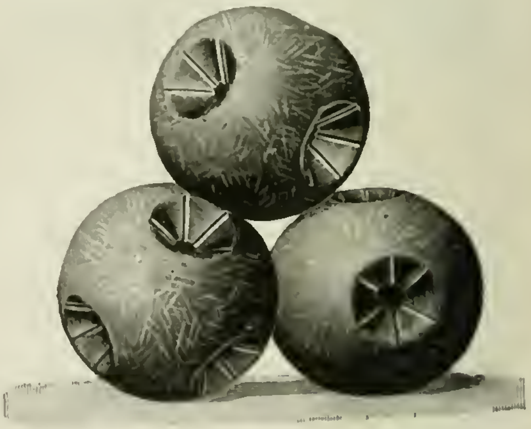
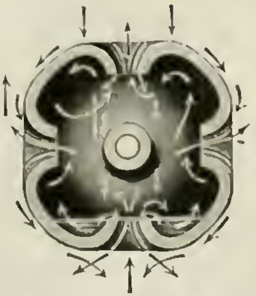


Fig. 2



The following conditions are therefore necessary:

1. Thorough sub-division of the liquid.
2. Diffusion of the gases within the tower, and
3. A large surface for absorption.

I believe you will find that the balls exhibited unite all three conditions in a very good way. The liquid running down does not merely run over the surface of the ball, but very soon finds itself forced to enter one of the side holes, thus not only the outer but also the inner surface of the ball is kept moist. The liquid collects in the ball, until it reaches a certain level, when it overflows through one of the openings, and the drop issuing therefrom falls on to the next ball.

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* When this paper was communicated to the Society, it was suggested that the refractive indices of the various oils would be valuable information, which we are now able to give.

* See Addendum to paper.

The gases go either round the ball or enter it. If they go round, they are forced to surround the ball, because there is no such thing possible as a direct channel, like with the coke tower. If the gases enter the ball, they expand from a small orifice into a larger space, and thereby go along the wall of the ball before they can get out again. By having both the outer and the inner surface of the ball contributing to the surface, and by having a constant stock of liquid in each ball, you will readily understand how much greater the efficiency of a tower of this kind must be. As a matter of fact, the absorbing surface per cubic foot of tower space is 2¼ times as large as that of a coke tower and over 2½ times that of a plate tower.

It is further apparent that such a tower does not want elaborate precautions for fixing, because all that is required is to fill the balls into the body of the tower. In whichever way a ball lies, it will always retain a stock of liquid, and it will always be effective.

This filling material has already been used in several factories, and is giving satisfaction.

## THE CAUSES OF "STENCH" IN BEER.

BY G. HARRIS MORRIS, PH.D., F.I.C.

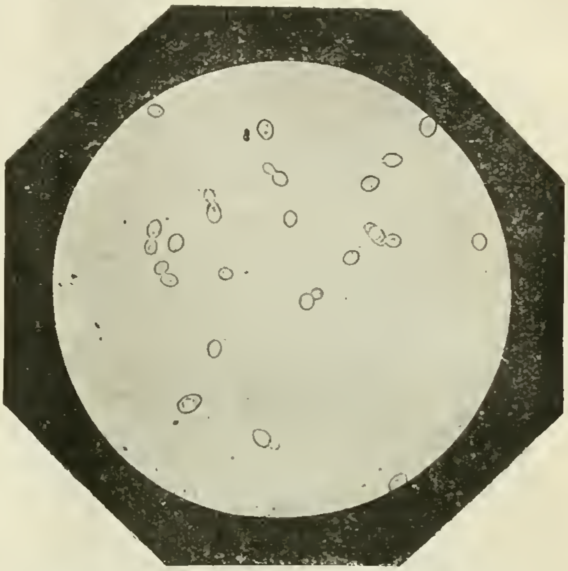
In the June issue of this Journal there appears a preliminary note by Dr. W. Frew under the above title. In this note the author speaks of the "stench" sometimes shown by beer, either in the fermenting square or in cask and bottle, and he says: "The 'stench' of the primary "fermentation, and that showing itself during storage, have, "as far as I have been able to discover, been regarded as "identical by all previous writers on this subject." He then goes on to state that the two forms of "stench" are entirely distinct in their nature, and are due to quite different agencies. He also gives a preliminary description of a yeast which produces a "stench" in cask, and promises a fuller account later in the year.

Although Dr. Frew may be correct with regard to the confusion by previous writers on the subject of the two forms of "stench," yet I am able to say that it has long been recognised in the brewery laboratories of Burton-on-Trent (the beers of which are more prone to show "stench," or "sickness" as it is more generally termed in Burton, than are those of any other district) that the smell of sulphuretted hydrogen which sometimes accompanies the primary fermentation is entirely distinct from the "stench" or "sickness" shown by beer during storage in cask or bottle, and which is generally accompanied, or preceded, by a want of condition. It has also been thoroughly recognised that the latter form of "stench" is produced by the growth of a special form of "wild-yeast," and I believe that others, in common with myself, have separated and cultivated this particular form of disease yeast.

I find from my note-book that, in 1891, I produced "sickness" in perfectly sound beer by inoculating it with some (one quart to the barrel) diseased beer. Decided symptoms of "sickness" were present in seven days from the date of inoculation, and the special form of yeast associated with the disease could be readily detected under the microscope. I also succeeded in separating, by Hansen's method, the yeast in a state of pure culture, and with this pure-cultivated yeast I produced "sickness" in beers by inoculation shortly after racking. In all cases marked signs of the disease were present in the inoculated beers at the end of a few days, whilst control casks of the same gyle placed under the same conditions, but uninoculated, presented a perfectly healthy and normal appearance.

The pure culture of the yeast producing "stench" or "sickness" was photographed by me at the time mentioned above. This photograph is shown in the accompanying figure.

I may add, that I fully agree with Dr. Frew that the "stench" given off by beer during storage is not due to sulphuretted hydrogen or any other sulphur compound, as at various times during the past 15 years I have endeavoured, without success, to obtain indications of the presence of any volatile compound of sulphur in diseased beers. On the other hand, sulphuretted hydrogen can readily be detected in beers showing "stench" during the primary fermentation.



Circumstances prevented me from following up the examination of the yeast I separated eight years ago, but I have no doubt that the note-books of my late colleagues of Burton-on-Trent will show the results of much fuller and more extended experiments on the yeast or yeasts producing this troublesome and unpleasant disease of beer than are outlined in the preceding remarks.

## THE MANUFACTURE OF PIG IRON IN INDIA.

BY HAROLD HARRIS, F.C.S.

During the years 1894–96 the author held the position of chemist to the Bengal Iron and Steel Company, whose works are situated at Barakar, Bengal, about 150 miles from Calcutta on the East Indian Railway, and 12 miles from Asansol, the junction of the Bengal, Nagpur, and East Indian railways. These being the only works of the sort in India, it seemed likely that a short description of them and of the materials used might be of interest to other chemists and metallurgists.

*Iron ores used.*—The principal iron ore used is a brown hydrated hæmatite, which is found as a surface deposit quite close to the works in laminated beds of from a foot to 18 inches thick. There are no regular mines, but the ore is worked in a desultory manner by petty native contractors who bring it to the works in bullock carts, carrying from 10 to 15 cwt. each, at a contract price delivered. The following is an average analysis of ore put into the furnace in 1896, although of course it varies considerably at times.

| | Per Cent. |
|---|---|
| Ferric oxide, $Fe_2O_3$ | 66·00 |
| Silica, $SiO_2$ | 12·50 |
| Alumina, $Al_2O_3$ | 5·00 |
| Phosphoric anhydride, $P_2O_5$ | 2·09 |
| Manganese protoxide, MnO | 1·73 |
| Lime, CaO | 1·48 |
| Magnesia, MgO | 1·00 |
| Loss on ignition | 10·50 |
| | 100·30 |

As will be seen from the above analysis the phosphorus is high, and consequently the pig produced is phosphoric, and suitable for the manufacture of steel by the Bessemer basic process. As, however, a pig for foundry purposes was required at the works, the following ores have been smelted in conjunction with the above to reduce the phosphorus in the pig produced, *Magnetic ore from Ramkanali, very hard to smelt.*

| | Per Cent. |
|---|---|
| Ferric oxide, $Fe_2O_3$ | 74·67 |
| Ferrous oxide, $FeO$ | 15·93 |
| Silica, $SiO_2$ | 9·30 |
| Phosphoric anhydride, $P_2O_5$ | 0·07 |
| Loss on ignition ($H_2O$) | 0·06 |
| | 100·03 |

*Non-magnetic ore from Manharpur, easily smelted.*

| | Per Cent. |
|---|---|
| Ferric oxide, $Fe_2O_3$ | 85·50 |
| Silica, $SiO_2$ | 11·00 |
| Phosphoric anhydride, $P_2O_5$ | 0·10 |
| Loss on ignition | 3·50 |
| | 100·10 |

With special mixtures of these three ores a pig was produced, containing a diminution in the phosphorus of 0·50 per cent. and upwards.

*Limestone used.*—Several limestones were used at the works, the two principal ones, however, were Sutna limestone and Pachete limestone. The Sutna has to be brought a distance of nearly 600 miles by rail, but even at this it shows an advantage both in price, composition, and results obtained in the blast furnace over the Pachete, which may be obtained within 20 miles of the works. The following are the analyses of the two different limestones used:—

*Sutna Limestone.*

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 7·00 |
| Ferric oxide, $Fe_2O_3$ | 0·60 |
| Alumina, $Al_2O_3$ | |
| Lime, $CaO$ | 50·30 |
| Magnesia, $MgO$ | 1·10 |
| Loss on ignition ($CO_2$) | 41·00 |
| | 100·00 |

*Pachete Limestone.*

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 10·00 |
| Ferric oxide, $Fe_2O_3$ | 7·00 |
| Alumina, $Al_2O_3$ | |
| Lime, $CaO$ | 37·80 |
| Magnesia, $MgO$ | 7·50 |
| Loss on ignition ($CO_2$) | 37·70 |
| | 100·00 |

*Fuel used.*—The Company have their own coal mines and coke ovens within half a mile of the works. The coal raised is very inferior in quality, and the coke manufactured is very soft, and contains a large percentage of ash, which has to be taken into serious consideration in making up the burden for the furnace.

The following is an analysis of the coke, which contains from 18 to 22 per cent. ash:—

| | Per Cent. |
|---|---|
| Fixed carbon | 79·10 |
| Volatile matter | 1·50 |
| Silica, $SiO_2$ | 10·94 |
| Ferric oxide, $Fe_2O_3$ | 3·40 |
| Alumina, $Al_2O_3$ | 4·20 |
| Lime, $CaO$ | 0·60 |
| Magnesia, $MgO$ | 0·26 |
| | 100·00 |

*Slag produced.* The slags produced are usually very heavy in proportion to the ton of pig iron produced, running from 25 to 30 cwt., according to the nature of the minerals used. The following is an analysis of a slag made when using Sutna limestone and making No. 1 pig:—

| | Per Cent. |
|---|---|
| Silica, $SiO_2$ | 36·00 |
| Alumina, $Al_2O_3$ | 16·50 |
| Ferrous oxide, $FeO$ | 1·60 |
| Manganese protoxide, $MnO$ | 1·20 |
| Lime, $CaO$ | 43·00 |
| Magnesia, $MgO$ | 1·70 |
| | 100·00 |

*Pig iron produced.*—As before stated, the pig is high in phosphorus, and also contains manganese in varying quantities. The following are the analyses of different pigs produced:—

| — | No. 1 Pig made from Local Ore. | No. 1 Pig made from Local and Ramkanali Ore. | No. 1 Pig made from Local and Manharpur Ore. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Silicon, Si | 3·50 | 4·50 | 4·00 |
| Phosphorus, P | 2·00 | 1·25 | 1·40 |
| Manganese, Mn | 2·50 | .. | .. |

*Furnace.*—The furnace used was of a modern type of about 12,000 cb. ft. capacity driven by hot blast (1,300° F.), and was capable of turning out from 350 to 450 tons of pig iron per week. The output was much less on account of the soft nature of the coke and the variation of materials, together with shortness of fuel, which latter has, I understand, now been overcome by the erection of more coke ovens.

*Labour.*—The labour is all native, with the exception of three European furnace foremen, who take eight-hour shifts in superintending the natives. There is also a European blast furnace manager. Native labour can be had in abundance at very cheap rates.

# Manchester Section.

*Meeting held on Friday, November 4th, 1898.*

DR. DAVID WATSON IN THE CHAIR.

## CHAIRMAN'S ADDRESS.

The City of Manchester is the capital of a district characterised by its practical connection with modern metallurgy, and nowhere is more attention paid to every aspect of the subject. In the counties of Lancashire and Cheshire the treatment of metals, whether we take the smelting of metals from their ores or their working up into useful products, employs a vast capital and a large army of workers, and it has been so for many years. To the great port of Liverpool have naturally gravitated much of the ores and crude products of copper, silver and gold, lead, tin, antimony and zinc exported from the producing countries of the world, and these are smelted at important works in various parts of the district, while the great coalfield about Wigan has been the natural home of an immense iron industry.

We have close at hand the original home of sodium and magnesium manufacture, and the now universal process of producing aluminium by the electrolysis of the chloride was first put into effective operation in this country at Patricroft.

At Northwich an important manufacture of zinc by an electrolytic method has recently been established and is in successful operation.

Living as we do in this great metallurgical district, I am not far wrong if I assume that your interest in the subject of metals is sufficient to warrant me in asking your attention while, for a short time, I speak on the present position of the metallurgical industry.

I have here a table showing in round figures what have been estimated as the production in the whole world of the chief industrial metals in the year 1871, and 25 years after in 1896.

The first point to be noticed is that the production of all the metals enumerated is very much greater at the end of the 25 years than it was at the beginning. During that short period new uses have been found for almost all metals. For instance, the electrician is responsible for a new and enormous demand for copper. It is only recently that the steel trade has called for nickel and aluminium. The increase in the demand for tin is to a great extent due to the extension of the trade in canned provisions.

| —— | 1871. | 1896. |
|---|---|---|
| Cast iron | 13,315,000 | 31,000,000 |
| Copper | 128,000 | 381,000 |
| Lead | 300,000 | 720,000 |
| Zinc | 133,000 | 403,700 |
| Tin | 26,500 | 70,400 |
| Silver | 1,442 | 5,696 |
| Gold | 169 | 313 |
| Nickel | 600 | 4,130 |
| Aluminium | .. | 1,000 |

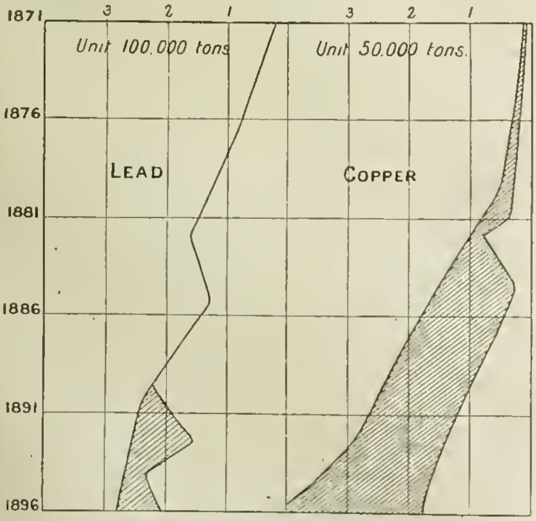
At the commencement of the period aluminium was a curiosity; now it is a necessity. Then it could not be produced at less than 40*s.* per lb.; now it can be had for less than 1*s.* 6*d.*

Something of the same sort can be said of nickel, although in 1871 nickel had a limited use for making German silver alloys for coinage and other purposes, but its price was then almost as many shillings a lb. as it now is pence.

There is every prospect that the annual requirements of the world for the principal metals will continue to grow, and the question naturally arises as to what probability there is that the supply will keep pace with the demand. I will not attempt to answer that question, which, indeed, would be too general. Probably the demand for some metals will be more easily met than for others. One thing I may say without venturing into the region of prophecy, and that is that in the two years since the end of 1896 supply has kept well up with the demand, and there is no sign that a famine is imminent with regard to any metal.

Before leaving this part of my subject I will draw your attention to the very important part that the United States of America has borne in this increase in the metallurgical industry, both as a producer and a consumer.

The production by the United States of America of copper and lead as well as zinc was, in 1896, nearly ten times what it was in 1871. The diagrams show the progress of this increase in the case of copper and lead. The shaded areas show the amount of the production which was exported.



At the present time the United States exports more copper than any other country, but a reference to the diagram shows that the export has only attained large dimensions within 15 or 16 years, and that that period has been characterized by an increase in domestic consumption of a very striking character.

A few years ago the United States of America began to export lead to a large extent, but the supply from that source has not been kept up, and to-day the United States of America consumes as much as it produces, the export quantity not being more than that imported into the country from Mexico and Canada.

The diagram shows how enormously the domestic demand for this metal has grown within the period.

What are the uses for which the enormous increase of metal is wanted?

The electrician has seen the addition to his department within a comparatively few years, and as a consequence of the invention of the modern dynamo, of electric light and electric transmission of power, entirely new industries demanding as the great essential plenty of copper, and the telephone and telegraph systems absorb every year more and more copper.

The now enormous trade in canned goods has grown rapidly from small dimensions; tin is the essential metal, and the bulk of the increased production of tin is no doubt spread upon iron and employed by this trade.

The larger part of the production of zinc is employed in coating iron sheets and articles of iron of various kinds for buildings and for domestic uses, or is rolled into sheets which are employed chiefly by the building trades.

Zinc and tin are also largely used for alloys with copper, and except pewter, soft solder, type metal, and anti-friction metals no alloys of lead are in extensive use, and the bulk of the lead production is rolled into sheets or made into tubes required for the builder or the sanitary engineer, or converted into white and red lead for the painter.

As a less important but new and increasing peaceful use for copper may be named the manufacture of sulphate of copper, a trade which, within the last 25 years, has been multiplied many times, and the product of which is almost entirely utilized by another great industry, agriculture.

The great increase in the use of lead is a consequence of the increase of the standard of domestic comfort in all the more civilized countries. The diagram showing the production and consumption of lead in the United States of America illustrates not only the statistics of lead but also the great progress that country has made in the convenience and comfort of its houses.

In the aggregate I estimate that of the four metals mentioned there have been raised from the earth and reduced to the metallic form in the last 25 years 5,000,000 tons of copper, 9,000,000 tons of lead, 5,000,000 tons of zinc, and 800,000 tons of tin, large totals.

Where is all that metal now, and where is all the iron and steel that has been produced in the same period?

How much is gone for ever, and how much remains as an addition to the substantial wealth of nations?

Some is certainly lost, for instance, who could recover for us the metal in the sulphate of copper that has been used for steeping grain or for salting the ground.

The tin plate which contained all the canned provisions that have been consumed in that period is it not almost entirely lost. For the most part it lies at the bottom of the sea and hidden in rubbish heaps in every quarter of the globe, and the tin is a total loss. No hope can be entertained that the lead and zinc made into paint will ever reappear for remanufacture, but the metals used in the construction of machinery, railways, ships, in buildings, in conduits for water and gas, for domestic appliances, for electrical conductors are, in the main, permanent and indestructible; much has no doubt been lost by corrosion and abrasion, but the quatity lost can hold but a small proportion to that which remains.

There is, therefore, always available in case supply should be temporarily unable to meet the demand, an accumulation of metals which can be drawn upon, and which is not recognised in any estimate of stocks. High prices for iron tends to bring out quantities of metal which the owners find it more advantageous to convert into money than to leave unproductive.

One effect of the high prices caused by the great syndicate corner in copper in 1889–1890–1891 was to bring into circulation relatively, very large quantities of scrap copper, old gun-metal, copper coins, and other old articles of copper and copper alloys.

To such an extent indeed were these and other hidden stocks of copper put into the market that the final collapse of the operation and the failure of the syndicate was attributed by some well qualified persons to a want of proper appreciation by them of this probability.

The metallurgist is primarily the servant of the miner. What the miner produces is his raw material.

The miner assists him in some degree by separating the metalliferous parts of what he raises from the earth, as tin, stone, or galena are concentrated and the gangue removed, but in the main he has to be ready to take material of all kinds of purity and variety. Workable copper ores vary from 1 per cent. to 70 per cent. A very large proportion of the world's supply is from ore of 5 per cent. or less. Silver and gold may be found in the ore of almost every metal. Lead sulphide may be associated with zinc sulphide and silver sulphide. He may have to extract its gold from ores containing not half an ounce to the ton, one part in 73,680, or to treat ores of copper containing arsenic, bismuth, antimony with gold and silver, or the miner may provide him with native copper almost absolutely pure and with hardly any admixture of foreign mineral.

He may have to make steel from iron peroxide almost perfectly pure or from a mineral charged with phosphorus and sulphur and with not more than 40 per cent. of iron.

At first, after the establishment of chemistry as a science, scientific metallurgists were chiefly engaged in the critical examination of the metallurgical processes which were already existing, and by the middle of the century the work was nearly completed. A great majority of the metallurgical processes now in use are identical with or very evidently founded upon these old methods. Thirty years ago, when I was a student in Dr. Percy's class at the school of Mines, the lectures were chiefly devoted to the description of the various metallurgical processes in use. Except the Bessemer process, scarcely any method of smelting was described that was not then already an old one, although the open-hearth methods of steel production were in the bud.

From that time no important addition has been made to the main productive processes of the iron trade except the extension of the powers of the Bessemer and open-hearth processes of ingot iron and steel production by the basic process of Thomas and Gilchrist, now so largely in use in this country and on the Continent, whereby phosphorus free steel is produced from materials which contain large quantities of that objectionable element.

Tin and zinc are extracted from their ores by the same processes that were in use many years ago, except that some little zinc is now produced by electro deposition.

The processes employed in silver smelting are numerous. The old processes of treating rich silver ores in which reduction and amalgamation are effected in one operation, an amalgam being obtained from which the mercury is separated by retorting, are still in use, as are also the Pattinson process and the Parkes process for desilverizing lead only—25 years ago the Pattinson process was almost universally employed, but to-day the reverse is the case, and by far the larger proportion of lead desilverized is treated by the Parkes zinc process. The Claudet process introduced in 1870, for separating the silver from cupreous pyrites treated by the Henderson process, is still the only process in use for that purpose.

The interesting old process of liquating copper purposely alloyed with lead is not now, I think, in use anywhere.

The extraction of silver from copper ores is effected for the most part either by the Ziervogel process, or by electro refining the metallic copper into which it is made to pass. By the last named means the silver is extracted from nearly 100,000 tons of copper annually. We will refer to this process again in speaking of the processes in use in copper production. Some silver and gold is separated from copper by the treatment of the latter by sulphuric acid. The sulphate of copper which results is an important article of commerce.

In the extraction of gold, the old amalgamation processes have been supplemented by and to some extent displaced by chlorination and cyanide processes in which the gold is rendered soluble as chloride or cyanide, and afterwards precipitated from the solutions. The cyanide process is new, but some work had been done by chlorination process before 1871. These two processes are extremely valuable to the metallurgist, because they enable him to extract gold from ores that previously could not be profitably worked. In copper metallurgy there have been great advances and I will shortly refer to these.

Bessemer pig is a name which is familiar to all who are interested in iron. The term is applied to a kind of pig iron prepared specially for conversion into steel by the pneumatic process of Bessemer. The same term is now equally familiar in the copper trade.

In this case it signifies crude copper produced by the treatment of copper sulphide by a pneumatic process similar to Bessemer's.

The first who demonstrated successfully that sulphuretted ores might be melted by the combustion of the sulphur contained in them was Mr. John Hollway of London, whose patents are dated 1878. His ideas were put into operation, but the processes did not make headway at the time.

M. Manhès introduced the process at his own works in France, and took out various patents, the earliest being in August 1880, but the process did not find a proper field until after trial; the mining companies of Montana adopted it as specially applicable to the treatment of the rich mattes produced in smelting their ores, and now the process is in operation on a great scale in Montana, in Japan, and in other places where the conditions are favourable.

It has not been found profitable to use this process in England, and as far as I know the use of the Bessemer process in copper smelting is everywhere confined to this one operation of the reduction of rich mattes.

Twenty-five years ago the production of copper in Arizona was undertaken under very difficult conditions, coke cost 25 dollars a ton, firebrick was worth a dollar, transit facilities were of the very worst. A form of furnace was there adopted and brought to perfection which needed little fuel and no firebricks, the so-called water-jacket furnace. The principle was already known to metallurgists. Blast furnaces have been used for copper smelting from time immemorial, and water-jacket furnaces similar to those employed in lead smelting had already been used for the reduction of slags. Since that time the Arizona water-jacket furnace has been put into use in suitable localities all over the world.

Attempts have been made generally without commercial success to put into regular practice one of John Hollway's ideas and to melt pyrites in blast furnaces by the heat evolved by the combustion of some of the sulphur and iron. Working on a specially suitable ore at Tilt cove in Newfoundland the operation has been quite successfully effected. The term applied to this operation by Mr. Austin and others who have worked at it and written about it is "pyritic smelting." It is an unfortunate term to use as it does not describe the process, and has been a recognised term for another operation altogether, which is conducted in a blast furnace, the smelting of poor silver and gold ores in admixture with pyrites as a first step to the separation of the gold and silver.

The original installation for the electro refining of copper was put into operation in this country by Mr. J. B. Elkington, who patented the process in 1865, and applied it to the recovery of silver and gold from copper containing those metals. His process is fully described in his second Patent Specification of 1870. Its principle is the same as that of the most important installations now at work. Up to the year 1878 only 5 or 6 tons per week were refined in this manner. In 1885 the use of the process had extended, and at several places on the Continent installations differing in details from that of Elkington, but the same in principle were refining a few tons weekly, perhaps 20 tons. When the United States commenced to export copper in large quantities, first ores and then mattes, a great deal of the copper was associated with silver, and the English smelters met this by installing important electro refining works capable of dealing with large quantities of argentiferous material in extension of the few already existing. In 1881 the first electro refining works was established in the United States, by 1892 the production in the United States had grown to 25,000 tons per annum, to-day it is probably three times as much; and at the present time the policy of all large copper mining companies is not to allow any of their argentiferous ores or mattes to come to this country, but refine all electrolytically at home, and ship the copper freed from its silver either in the condition in which it leaves the vats, or after melting and casting into saleable forms.

The process of transference by an electro current either with the object of procuring a pure cathode or for obtaining a valuable constituent which is separated from the anode can be employed in other cases besides copper. Lead can be separated from the silver and gold it contains. A process for effecting this has been thoroughly worked out and abandoned because it did not compete successfully with other processes for effecting the same purpose. Gold containing platinum as also the zinc crusts proceeding from the desilverization of lead by the Parkes process have been refined in this way, and the constituents separated.

At the time when practical electricians first began to apply the powers of electricity in metallurgy, it was expected that the extraction of metals directly from their ores would be largely effected by the electric current. But those anticipations have not been realized at present, although many metals are refined by electricity, and zinc, sodium, and aluminium are commercially separated from their chemical compounds by it; none are extracted direct from the ore.

Within the last few years the reduction of metallic oxides by carbon at very high temperatures has been effected by the use of the electric furnace, but I do not think that the powers of this furnace are at the present time employed commercially on the production of metals. The Cowles process introduced in 1886 was much talked of at the time, and some alloys produced by it were sold. The great objection to these productions was a want of uniformity and purity which could not be avoided, and the process was abandoned. Quite lately similar temperatures and similar reductions have been attained by the oxidation of aluminum by metallic oxides. The reducing agent as well as the fuel is aluminium, which, in a finely divided state, is mixed with the oxide to be reduced, the reaction is started by a fuse imbedded on the mixture, which is ignited by external means or the oxides are thrown into molten aluminium. Alloys containing chromium and molybdenum and other metals are offered to the trade that have probably been produced by one or other of the last-mentioned processes.

So far I have spoken only of metallurgy as dealing with the extraction of metals from their ores into the metallic form.

It is further the business of the metallurgist to refine and combine these metals so as to meet the requirements of the constructor.

Up to a certain point the constructor was content to take metals as they were produced, and arrange his plans to utilise the properties of these, so to speak, natural products, but that is not so to-day. Twenty-five years ago, the engineer who was careful, required in his specifications Low Moor Iron, Coro Coro Copper, Virgin Spelter, Cold Blast Iron, and so forth, to-day such specifications no longer require the metal to be derived from certain brands or qualities, or to be made by certain specified processes, but explicitly state the properties that the metal must possess.

Steel plates or copper rods must have certain specified tensile strengths and ductility, they must stand successfully certain mechanical tests, steel or copper wire for electrical conductors must have also a specified conductivity, aluminium must be of specified purity and so on. This is as it should be. A direct stimulus is thus given to metallurgists to compete with one another in finding for the engineer something better than he has been able to obtain before.

Of course, there are requirements that are not likely to be met.

If an engineer could command a metal as strong as the strongest steel, as light as aluminium, and incorrodible as gold, and moderately cheap, what dreams could he not realize. But in striving to reach the pole, the explorer at least reaches high latitudes, and real progress results by striving to attain the ideal.

What has been done in this direction has been, perhaps, chiefly by means of happy-go-lucky synthetic experiments, often very scientifically conducted, and very intelligently worked out; but there are not wanting indications that this period is passing. It cannot entirely pass away until a great void in chemical science is filled. We must first have a new chapter of chemistry, dealing with the combinations of the metals amongst themselves and with other elements, and their re-actions with one-another.

The materials are accumulating for this chapter, but progress is slow because the workers are few and their resources are too small, and the subject of extreme difficulty.

One question has, more than any other, been asked by practical metallurgists, and the answer is still incomplete.

It is shortly "What is steel?"

A portion of the answer has long been given by the analyst. It is iron containing carbon in certain quantities, and the steel maker by his guidance can produce steel of various grades with more certainty than before. But the remainder of the answer involving the explanation of hardening and tempering is still wanting. In the course of researches to solve this problem, new and unforeseen phenomena have been observed, and new means of research have been invented.

It has been established chiefly by microscopic investigation that compounds of carbon and iron are readily formed at higher temperature that are disassociated or pass into other compounds as the metal cools, but that time is an element in these changes, and that by very rapid cooling the changes are arrested or modified. The temperature at which these changes take place have been established by the use of the thermo-electric thermometer, which show that they are accompanied like other chemical changes by heat, absorption, or liberation. The whole forms a most interesting study, but still leaves the matter unsettled. At present, the scientific workers who have taken up the subject so far are not agreed on the interpretation of these results.

The same methods of research which have taught so much in regard to steel are applicable to the investigation of the chamistry of metals in general, and I think it will be of interest to you if I briefly describe how these methods are applied.

First, the thermo-electric thermometer, which we owe to its present form to M. Chatelier, and which Professor Roberts Austen has much improved as an instrument of research by making it record its indications automatically.

It consists essentially of two parts, the couple and the galvanometer recording the E.M.F. produced in the circuit when the couple is heated.

The couple is composed of wires, one of an alloy of rhodium and platinum and the other of platinum. These are twisted together a turn or two, and the portion thus twisted together is the couple by the heating of which an E.M.F. is produced causing a current of a quantity which is proportional to the temperature of the couple to pass from the wires of the couple through the conductors and the indicating galvanometer.

The relative infusibility of the metals of which these wires are composed enables very high temperature to be indicated.

The galvanometer is a reflecting galvanometer, and its movements are recorded in the usual way by the movement of a reflected ray of light on a divided scale.

The instrument is calibrated by comparing the indication given when the couple is immersed consecutively in a series of molten substances, the melting points of which are known and which are allowed to cool. The point of the scale at which the spot of light becomes stationary, corresponding with the known freezing point of the test substances. The spot of light remains stationary until freezing is completed and the temperature begins to fall, then it commences again to travel to zero.

Another pyrometer which is perhaps preferable where it can be applied is also electric. In this case there is a complete metallic circuit in one part of which is an indicating galvanometric arrangement, and in another a standard galvanic cell, one portion of the circuit consists of a small coil of platinum wire, the coils of which are carefully insulated, enclosed in a platinum envelope. If the platinum envelope is heated, thus heating the platinum coil, the resistance of the coil is increased, so that if the galvanometric arrangement indicates zero at the ordinary temperature, a relatively higher temperature will be indicated by a change of position of the indicator. This is the principle of the pyrometer employed by Messrs. Haycock and Neville in their researches on the properties of alloys.

The ordinary glass and mercury thermometer has been improved so that it is now possible to get one graduated to indicate temperatures up to 1,000° F.

The other instrument of research to which I referred is the microscope.

The polished surface of a piece of metal is never uniform The want of uniformity is often to some degree perceptible by the eye unaided by the microscope, for instance, if the surface of a piece of cast zinc be polished and examined by oblique light which is incident at the same angle on all parts, the eye will detect that some parts reflect a greater amount of light than others. If the surface is then examined, a microscope magnifying 200 or 300 diameters it will be found the light reflected to the eye proceeds from a number of reflecting surfaces of great minuteness, and within definite areas of irregular polygonal shape the amount of reflection is uniform, but is more or less in the adjacent areas.

This phenomenon is a common one among metals, and it has been proposed to call the areas cells, a convenient term.

The reflecting points are due to the fundamental crystalline structure of the metal, which requires objectives of very high power for its resolution.

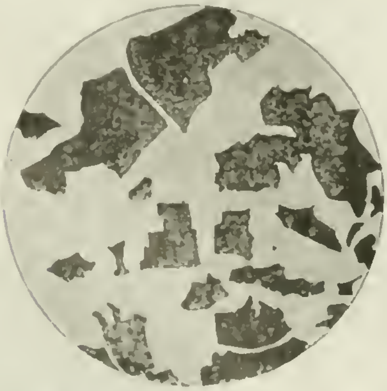
A pure metal is composed of minute crystals, but as we have seen the arrangement of these crystals varies in the different cells.

If the surface of a metal showing this structure is covered by a liquid which attacks and dissolves it, it becomes apparent that there is electric antagonism between the different surfaces of these minute crystals on which account the surfaces are roughened, and in addition, there is the same antagonism between the cells; and the surfaces of these are not equally dissolved, besides which the dividing lines between the cells are the parts most energetically attacked.

The metallurgist does not always deal with pure metals, and a metal which contains other metals usually presents distinct indications on its polished and etched surface.

For instance, the drawing No. 1 represents with accuracy the appearance presented by the polished surface of a piece of zinc, which contains 5 per cent. of iron when examined by a low power objective. The surface has not been etched in any way. In this case the surface of the metal was observed by oblique light; the bright part of the picture is where the surface was roughened by the polishing. These parts are pure zinc. The black parts are harder and have not been so much abraded in the process of polishing. These parts consist of an alloy of iron and zinc. Pure zinc treated in this way shows none of these reflecting spots, but even so small a quantity of iron as $\frac{1}{1000}$ part can be detected by isolated black spots.

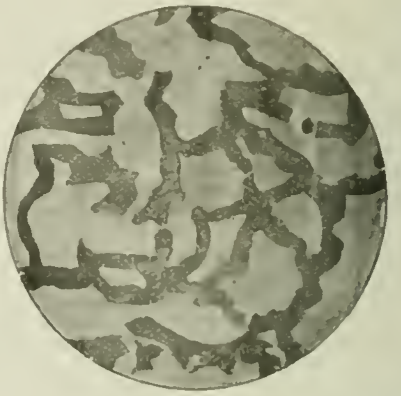
Fig. 1.



It is these iron zinc crystals that cause the peculiar roughening of the facets that is a sure sign to the practised eye that zinc contains iron.

The drawing No. 2 shows the appearance presented by the etched surface of copper containing ·35 per cent. bismuth. The copper has crystallized out forming the cells; the bismuth with some copper surrounds these cells.

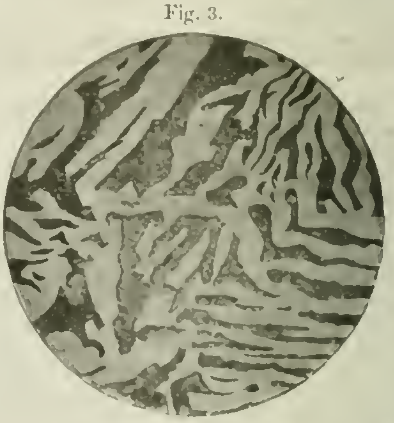
Fig. 2.



Copper and zinc are often said to alloy together in all proportions, which is only true to the extent that when molten mixtures of copper and zinc in all proportions can exist without separation into portions of different specific gravity. When such a molten mixture solidifies the resultant solid is a mechanical mixture of chemical compounds of zinc and copper with it may be copper or zinc.

As an illustration, I draw your attention to the drawing No. 3, which shows the appearance of a surface of a section of a casting containing 3 parts of copper and 2 parts of zinc.

Fig. 3.



After polishing and etching by nitric acid, two constituents are plainly shown under the microscope, each of which is a compound of copper and zinc.

I hope what I have said will enable those of you who are not at present familiar with these matters, to follow me while I say a few words on recent investigations into the structure and chemistry of metals and alloys.

It has long been recognised that metals appear to unite with one another in all proportions, and I quote from the "Elements of Chemistry," by Turner, the seventh edition, by Liebig and Gregory, published in 1842, in what follows:—

"It is certain, however, that metals have a tendency to combine in definite proportion, for several atomic compounds of this class occur native. The crystallised amalgam of silver, for example, is composed, according to the analysis of Klaproth, of 64 parts of mercury and 36 of silver, numbers which are so nearly in the ratio of 202 to 108 that the amalgam may be inferred to contain one eq. of each of its elements. It is indeed possible that the variety of proportion in alloys is rather apparent than real, arising from the mixture of a few definite compounds with each other or with uncombined metal, an opinion not only suggested by the mode in which alloys are prepared, but in some measure supported by observation. Thus, on adding successive small quantities of silver to mercury, a great variety of fluid amalgams are apparently produced, but in reality the chief, if not the sole, compound is a solid amalgam, which is merely diffused through the fluid mass and may be separated by pressing the liquid mercury through a piece of thick leather. This view is strengthened by some late experiments by Rudberg. He finds that variable mixtures of metals in cooling after fusion have generally two periods when the thermometer is stationary. In alloys of lead and tin one of these points is uniformly 368° for all mixtures, while the other point varies according as one or the other metal is predominant, and is near the fusing point of the predominating metal. From this it is inferred that the latter point is caused by the congelation of the predominating metal and the constant point is the congealing temperature of an alloy of uniform composition present in all the mixtures. This alloy is composed of 3 lbs. of tin and one equivalent of lead, its congealing point being 368°."

The writer of the text book thus left the question in suspense in 1842, and little was done to take the matter further till within the last few years. I ought here to mention with the greatest appreciation the debt which metallurgists owe to the Institution of Mechanical Engineers in regard to this subject. In 1890 the Society appointed an Alloys Research Committee, which, with Prof. Roberts Austen as its metallurgist and reporter, has been engaged on the investigation of alloys in a systematic manner, and has compelled the attention of those interested in such subjects by the suggestiveness of the facts ascertained and of the conclusions arrived at. Unfortunately the Committee has not, up to the present, published any observations made by the microscope, but has drawn its conclusions without reference to the revelations of that powerful instrument of research.

Comparing the examination of alloys with that of mixtures of ordinary organic or inorganic substances, the difficulties become at once apparent. Such substances are separated by crystallisation from solution, by distillation, and in other ways, and when separated they can be further purified for the exact determination of their physical properties and chemical reactions.

All these operations are extremely difficult in relation to some alloys and impossible with regard to others.

Often, or perhaps usually, what corresponds to a solvent is the least fusible, and during solidification the dissolved substance finds its place in the spaces between the crystals or cells of the solvent. It has been attempted to treat a partially cooled alloy, and after solidification of the solvent to remove, by squeezing out in a press, the liquid remainder, like oil is squeezed out of cotton seed. There is promise in this mode of research, but he who takes it up will find he has a difficult piece of work to carry out.

Sometimes the crystallised solvent is so much lighter than that it holds in solution that as it separates it rises from it. This is the case with lead containing antimony.

Mr. Stead, who investigated the matter, found that if, for instance, he took a molten mixture of lead and antimony, say 70 parts lead and 30 parts antimony and cooled it slowly, that first pure antimony crystallised out and rose to the top of the cooling mass, leaving a remainder which contained 12·7 per cent. of antimony, and that whether the lead or the antimony was increased or diminished, the composition of the alloy remained the same, less or more antimony separating.

Somewhat exceptional cases, of which this is one, enable one by analogy to more readily understand cases of more difficulty.

For comparison I will take a parallel case, but one in which the physical conditions made it hopeless to separate the solvent from the dissolved substance mechanically.

A mixture of copper and silver in which the silver is in excess—for instance, 10 copper 90 silver—is found to consist when cold of a mechanical mixture of silver and a compound of silver and copper. The freeing point of the silver holding the alloy in solution is about 875° C. Pure silver separated until the temperature falls to 700°, the remainder which has the composition 72 silver, 28 copper crystallises at that temperature, but there is no separation in mass on account of difference of specific gravity, the densities of silver and of the silver copper alloy being so nearly the same.

Silver is crystallised as the temperature falls until, it reaches the point at which the remainder also solidifies. It would be hopeless, therefore, to attempt to separate the silver from the remainder by the compression method.

M. Charpy has shown that Mr. Stead's lead antimony residue is a mechanical mixture of the two metals, and M. Chatelier proves that the residue of 72 silver and 28 copper is also a mechanical mixture. All such residual mechanical mixtures or alloys—for they are not always mixtures—whatever may be their composition, are called eutectic alloys, a term which it is difficult accurately to define but which it is convenient to use.

It is not my purpose to give you an exposition of a theory of alloys but to submit to you some examples to illustrate the course research is taking and the nature of the conclusions arrived at.

I will refer again to my drawing showing the microscopic structure of a casting containing 3 parts copper and 2 parts zinc. The two constituents shown are respectively solvent and eutectic. In each case we cannot doubt that they consist wholly of copper and zinc, as there are no other elements present; the yellow, the portion least attached by the acid, being that which separates first (the solvent) and the other the eutectic.

When the homogeneous mixture cools from a state of fusion, freezing commences at about 885° and is completed at about 870°. The temperature falls gradually between those two points and much more slowly than during either the period of cooling down to 885° or the subsequent cooling after solidification, owing to the evolution of the latent heat of fusion. At 885° the solvent compound of copper and zinc commences to freeze out, and at a certain lower temperature the eutectic solidifies, losing its latent heat.

As a result there is a solid mass in which the two alloys are alternated with one another in laminæ.

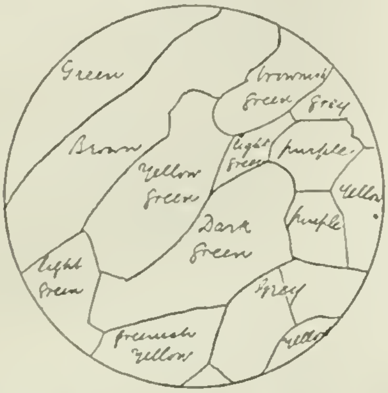
The drawing No. 3 is of a portion of a section at right angles to the cooling direction; the diameter of the circle drawn is $\frac{1}{100}$ in.; the laminæ are about 50 in that distance each being more or less $\frac{1}{5000}$ in. in width. Roughly speaking the surfaces occupied by the two constituents are equal. On the left of the drawing the width of the laminæ is greater. This is because there the laminæ lie more nearly parallel to the plane of the section and are cut obliquely, on the right side they are cut at right angles. It is to be noticed that the yellow is continuous, whilst the darker is not.

When the etched surface of the three-alloy is examined without magnification some slight irregularities of the surface can be detected; but not the development of cells in the remarkable way shown by the next alloy, also of copper and zinc.

The illustration, No. 4, shows the appearance by oblique illumination of a polished and etched surface of a casting containing 52 per cent. copper and 48 per cent. zinc. The diameter of the circle enclosing the portion drawn is $\frac{1}{4}$ in. The development of cells is very marked and is rendered specially distinct by the various colours of the different cells. Some are yellow, some of various shades of green, while others are red, purple and grey. A further microscopic examination shows the cause of this brilliant colouring, none of the colours being the real colour of the alloy—a brown grey.

In the further microscopic examination an arrangement is employed which is sufficiently uncommon to be worth a word or two of description. In the previous case the microscopic examination was made with objectives whose working distance allowed of the use of oblique illumination, but such objectives are not sufficient in any degree to resolve the structure of the surface of this metal, and the second drawing shows the structure of one of the green areas as seen by a $\frac{1}{12}$ in. oil immersion objective of N.A. 1·25.

Fig. 4.

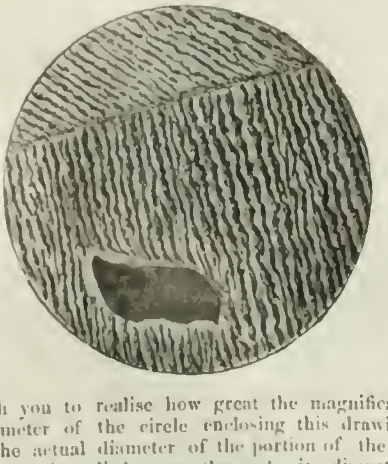


The reflector is one of great simplicity, but cannot be employed in ordinary work with covered objects and with non-immersion lenses, because of the reflection from the cover glass. The metallic surfaces I speak of are examined uncovered and the vertical reflector is most efficient.

It consists of a short tube inserted between the objective and the tube of the microscope. On one side is a hole through which a beam of light is thrown on to the reflector, a circular disc of transparent glass inclined at an angle of 45° to the axis of the tube of the microscope. A portion of the beam is reflected down to the objective and condensed on the surface under examination by the objective itself. The beam of light after modification by the surface is returned through the objective and visualised by the eyepiece as usual.

When one of the cells of the alloy shown on Fig. 4 is examined by the microscope, using vertical illumination and a $\frac{1}{12}$ in. oil immersion lens the surface presents the appearance shown in drawing No. 5. It becomes apparent that the colour which is to be seen no longer, is produced by light interference caused by the surface being striated. The striæ being the edge of laminæ which are developed by the etching. The resolving power of the objective used is very great but it is not sufficient to show any further structure.

Fig. 5.



I wish you to realise how great the magnification is. The diameter of the circle enclosing this drawing is 3 ins. The actual diameter of the portion of the surface depicted is only a little more than $\frac{1}{1000}$ in. diameter. No structure beyond that shown was distinctly visible.

We may judge what is probably the kind of structure that would become visible by more complete resolution by taking for comparison another alloy of copper and zinc which also shows a cellular and minutely striated structure on a polished and etched surface. The three drawings, Nos. 6, 7, and 8, represent the appearance when examined in the same way as No 5 of the polished and etched surface of an alloy of 80 parts of copper and 20 parts of zinc. Here, the structure being less minute, is more completely resolved and it is apparent that the striæ of the surface are the edges of laminæ with a tabular crystallisation.

Fig. 6.

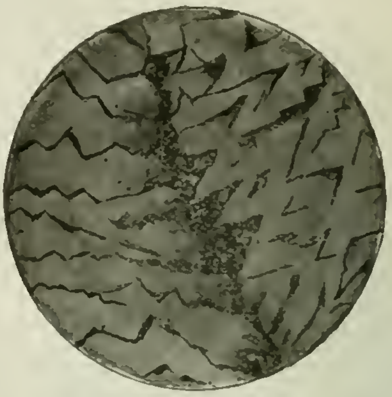
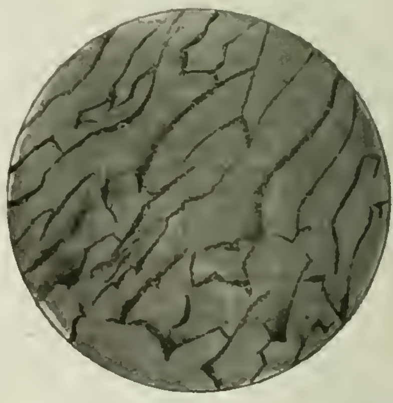
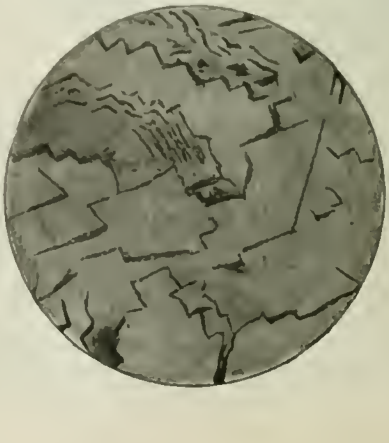


Fig. 7.



Facts like these leave no room to doubt that the true view to take of a solid metal is not, as has sometimes been imagined, that of a homogeneous mass holding distributed about it in atomic solution what other metal or metals it

Fig. 8.



may contain, but rather of a mass composed of metals or definite metallic compounds mechanically mixed.

The microscope and pyrometer are making this truth apparent, and a whole regiment of known facts are being explained by their means.

A few years ago it was customary among writers on the subject of metals and alloys to hold in reserve as a means of explaining the unexplainable a convenient theory of allotropy, but little is now heard of this.

I do not know any well proved case of allotropy among metals.

I have endeavoured in the short time at my disposal to touch upon many parts of our interesting subject, and I want to leave with you the knowledge that metallurgy is active and progressive, both as an art and as a science. It has adapted to its requirements all the highest products of mechanical skill and in return is striving to give to the mechanic improved materials for his constructions. It has been a reproach in the mouths of some chemists that metallurgists did little to advance that part of the science of chemistry that lay in their province. However justified that may have been in the past it is not so at present, and chemists may with confidence expect that the first few years of the 20th century will bring us the elucidation of the chemistry of metals.

## New York Section.

*Meeting held at the College of Pharmacy, on Friday, October 21st, 1898.*

MR. THOS. J. PARKER IN THE CHAIR.

### NOTES UPON ASPHALTS AND ASPHALTUM.

BY S. F. PECKHAM.

When I prepared the paper, read before this Society on the evening of Friday, April 22nd, 1898, I had not seen the paper that was read November 19th, 1897, (this Journal 1898, 13—32) by Mr. Clifford Richardson. There was little in his paper that would have led me to modify my views as expressed in my paper, but the careful perusal which I have since given it would have led me to add to my paper several statements which the subject-matter, as discussed by Mr. Richardson, has suggested. I shall therefore embody them in the review of his latest treatment of the subject which I herein propose to give.

Mr. Richardson has practically invited a review of his work upon Trinidad pitch since 1890, as he refers to and includes his report of 1892, and incidentally his work leading up to it. His last paper he so systematically arranged and divided under appropriate sections that the task of the reviewer is made easy by reference to the headings of these sections.

The sections entitled "Introduction" and the "Trinidad Asphalt Deposits," simply restate the subject. The "Form of the Lake Deposits," introduces new matter, relating to the extent of the deposit, of great interest. The last paragraph of this section, however, is not at all clear. He says: "The surface of the lake, it is very evident even from a very casual examination, is lower to-day than some years ago, and the deposit now seems to occupy a shallow depression." What does he mean by shallow? He has just shown that it is over 135 ft. deep and really of unknown depth. Continuing: "This fall is due to the removal of the crude pitch for shipment, which has reached over a million tons. From the difference in level of the surface of the lake between any two years, and from the amount of pitch removed, it is easy to calculate how many tons of it correspond to a fall of an inch or a foot. From the latter figures, it appears that the lake should have fallen many more feet than it has in the last 30 ; years and from a comparison of the results of the first and second calculation extending over three periods and three sets of levels, there is no doubt that there must be an influx of fresh pitch at the "soft spot," which in the periods between 1893 and 1896 amounted to an average of from 20,000 to 18,000 tons per year."

The lake, for the most part, is surrounded by an escarpment, except where this rim or escarpment has been broken down on the north-east side, for a distance of perhaps 100 ft., by the outflow of pitch to the sea. The surface of this outflow extends from the centre of the lake to the sea in an unbroken incline. Walking up this incline, which outside the lake is quite sharp, as the edge of the lake is approached it suddenly changes to almost a level; but the fact that the centre of the lake is higher than its edge is perfectly apparent to the eye and was the first feature to attract my attention as I walked on to the lake. I next noticed with surprise that there was no evidence that the pitch had recently receded from the escarpment at its edge. The pitch appeared to have fallen some 2 or 3 ft. when the outflow took place, but there was no evidence that the outflow had been a recent occurence.

One million tons represents an enormous volume. When extended over 30 years it amounts to $33,333\frac{1}{3}$ tons a year. The specific gravity of the pitch is about 1·45. Roughly, this may be estimated at a cubic yard to the ton, and 1,000,000 tons over 114 acres at 6 ft. in depth. There is no evidence of any such removal; in fact, if the present level of the lake were raised 6 ft. the pitch would overflow the rim for nearly its entire circumference. It is not necessary to assume that there is an inflow equal, or nearly equal, to the amount removed. The lake is not filled with a fluid or a substance of a uniform consistence. There seems to be no room to doubt that the material at the centre of the lake has gradually become more dense during the last 30–50 years. This fact can be explained without predicating the addition of a pound of bitumen. The enormous mass of 1,000,000 tons has all been removed from nearly the same portion of the lake surface, around an annular space enclosed by the loop of the tramway. If the level of the pitch did not practically adjust itself almost as rapidly as the pitch is removed, there would be an annular depression on both sides of the tramway, with the centre, the tramway and the external ring next the circumference of the lake at higher levels. Such is not the case. The surface slopes up from the circumference to the centre at a small angle, and as the pitch at the centre becomes harder it offers a greater resistance to the rising gas and the proportion of gas increases; consequently the mass of the pitch does not appear to be changed in volume by removals, as the excavated portions soon fill from below. This movement can only be explained by supposing a movement of pitch from the annular space outside the tramway toward the soft spot in the centre, and that the rising gas expands under decreased pressure sufficiently to create an equilibrium, in which the soft portion near the centre is gradually mingled with the harder pitch flowing into it. If an appreciable change of level had occurred in the lake, the surface would descend toward the centre from the level of the outflow, and the line across the outflow, instead of being nearly level, as at present, would inevitably represent some sort of a curve. The conclusion is, therefore, that the whole mass of pitch, from the centre of the lake to the sea, is in a state of unstable equilibrium that is constantly adjusting itself to conditions that are in a state of perpetual change, in consequence of two causes o movement—the outflow to the sea and the removals.

The successive adjustment of equilibrium to conditions that change with every removal of pitch must go on until the gas pressure becomes stable, and no lateral movement takes place from the circumference towards the centre. When this condition is reached the centre of the lake will cease to exhibit a soft spot, and the surface of the lake will fall and become, in all probability, depressed at the centre.

Under "Movement of the lake surface" some very interesting facts are stated that show the remarkable instability of the lake surface.

Under the section "Proximate composition of Trinidad asphalt" the work done by Miss Linton, in which she showed that volatile oils as well as water are expelled from crude Trinidad pitch at a temperature of 100° C., is fully confirmed.

It is proper to say here that Mr. Richardson's paper constantly changes the point of view upon which the discussion proceeds; that is to say, the commercial, technical, and scientific aspects of the subject are mingled together in a constantly shifting, kaleidoscopic movement, that renders a clear comprehension of the subject somewhat difficult.

Mr. Richardson states that Trinidad pitch, as it exists in the lake, is an emulsion, which is to say, that the bitumen being insoluble in water is mechanically mingled with it to form a mass resembling cheese in consistence. If a piece of this *cheese pitch* be laid in the sun the bitumen will melt in a film and form a sort of sack, in which the imperfectly dried pitch will be held in a more or less wet condition. If the piece of cheese pitch is dried in the shade, more of the water will be expelled, but there will still remain more or less water that is entangled in the gas bubbles and other cavities of the partially melted pitch, that under ordinary conditions will stay there for years. Of course, when the pitch is completely broken up and the water all expelled, a perfectly dry powder remains. There is no osmosis because the mixture is originally mechanical, as all emulsions must be, and not organic or structural; hence, what Mr. Richardson means by "cell wall of bitumen" is not clear, neither is the element of novelty acknowledged by the Patent Office. The novelty exists in the thing itself, which is perfectly unique; that is an emulsion of bitumen, water, and gas, containing a large percentage of mineral salts and humus. When it is dried out after being pulverised, like a thousand other things if exposed to damp air the hygroscopic salts and humus absorb water. The pulverised and dry pitch can be again emulsified with water if the temperature is kept so low that the bitumen is not melted.

The exact meaning of the last paragraph of this section, which relates to the samples collected by Mr. Henry, is not clear. Is it intended, besides being a statement of the manner in which Mr. Henry selected his specimens, to discredit all other specimens not collected in the same manner; those collected by myself for instance? The fact of the case is, a person can collect anything he pleases from the lake, from nearly pure humus with very little pitch, to nearly pure pitch with very little humus. I went there to secure specimens of commercial lake pitch, and the five specimens of that material secured by me were masses that were dug up to be thrown into the tram cars, and were taken from under the feet of the negroes who were digging the pitch. The whole mass of the pitch in that part of the lake from which the commercial pitch is taken, is in a constant state of slow ebullition from the escape of gas. There is no doubt this movement proceeds from the very bottom at an unknown depth. Pitch that is at the top to-day is in time at the bottom, as an effect of this incessant movement; hence, like a well-kneaded batch of dough, there is general uniformity in the entire mass, for, without doubt, the kneading has been going on for centuries.

### *The method of Analysis.*

The drying *in vacuo* over sulphuric acid is entirely unnecessary, as Mr. Richardson's experiments show that in dry air 100 per cent. of the water is lost in 24 hours. I have found no appreciable difference in the solvent properties of different samples of naphtha of 74° and upwards, if they are dried by washing with oil of vitriol and distilling with steam over lime. The naphtha should be kept in well-stoppered bottles. Wet naphtha dissolves the soluble salts in the pitch as well as the bitumen. I never use bisulphide of carbon. It is very volatile; it is very unwholesome to inhale; it is very difficult to obtain pure. It requires several distillations from an alembic to free it from dissolved sulphur, and if an attempt is made to free it from sulphur by agitation with mercury, as has been recommended, the method will be found troublesome, expensive, and unsatisfactory. Besides, as Mr. Richardson admitted in his testimony at the Peoria trial, bisulphide of carbon is not a complete solvent for the bitumen of Trinidad pitch. According to my experience, it is much less difficult to obtain dry turpentine and chloroform than it is to obtain *pure* bisulphide of carbon. Commercial articles cannot be depended on for any of them. From my experience they are all sold with all the water they will hold.

Having provided *dry* naphtha, spirits of turpentine, and chloroform, I prefer to use them rather than the naphtha and bisulphide of carbon for the added reason, that the use of the three solvents shows differences that are not revealed by two, that is to say, that as a general method of analysis applied to all solid bitumens, the use of three solvents furnishes a more complete analytical process than the use of two.

### *Bitumen soluble in Naphtha.*

Mr. Richardson says, "In my report of 1892 I showed that between the refined products of that pitch from the Trinidad Lake and that from deposits outside, known as land asphalt, a decided difference could be detected in the relative amount of their total bitumen soluble in 88° naphtha." Referring to this report of 1892, it is found that in the refined specimens of lake asphalt is included soft refined; and five out of fourteen of the samples ranging at 40 per cent. and upwards were doubtless oiled in the kettle. Of the land refined, soil, chocolate pitch, iron pitch, the cargo of the Teneriffe, all of which are not commercial articles, are included in a list of which nearly all are unusually low. It is difficult to understand why the rubbish of the deposit should be included in one list and not in the other, or perhaps, why it should be mentioned at all. In this report he says, further, "It is apparent, therefore, that in a commercial supply of land pitch which has been carefully selected, and from which alteration products and soil have been excluded, there is no essential difference in the relative proportions of mineral matter, bitumen, and organic matter not bitumen, from those in the lake." This statement is absolutely true, yet, it is equally true, that as a general rule, the farther a specimen is taken in the deposit from the centre of the lake, the larger is the percentage of the mineral matter, and, as a consequence, the smaller is the proportion of the other ingredients. Mr. Richardson, throughout the discussion, maintains a greater difference than fairly exists by using clean specimens in the one list and clean specimens mixed with rubbish, which has no commercial value, in the other; manifestly an unfair method of procedure.

In the paper under review Mr. Richardson returns to this discussion and speaks of the land pitch as occurring in deposits. He says, "Manross describes an overflow from the lake, and to this is undoubtedly due the presence of pitch on the lands adjoining it, and for some distance towards the sea to the east; but a large amount of the pitch must have been ejected independently of the lake source of supply, but so long ago that it has now been buried with soil." The condition at the lake exactly resembles a kettle of stiff molasses candy, from the side of which a piece has been broken allowing a part of the contents to flow out. This outflow has spread like a fan over many acres, from the lake down to the sea, extending in an unbroken glacier or lava flow, to both of which it has been likened.

Mr. Richardson refers to the analyses made by Miss Linton in 1895, and says, "I have arranged these analyses and classified them according to their lake or land origin, and leaving out one analysis of lake pitch, No. 21, which is plainly an error." This specimen to which he objects was taken from the cut where the tramway grade was carried through the escarpment, and was a sample of clean cheese pitch, full of water, and taken several feet below the surface and within 100 feet of the edge of the lake. I knew nothing of its peculiar character when I collected it, and Miss Linton did not know where it came from when she analysed it. As she is an analyst who never makes mistakes, and there is no possible mistake as to where it came from, Mr. Richardson had no right whatever to exclude it from the list, and thus make a greater contrast between lake and land pitch than really exists. Mr. Richardson also fails to observe another fact brought out by Miss Linton's analyses, *viz.*, that No. 17, taken from under the negroes' feet on the west side of the tramway, and No. 2, which came from the village lot nearest the sea, that is to say, the two specimens representing the two extremes in position between the centre of the lake and the sea, are almost exactly alike, the difference in the percentages of the total amount of bitumen being only $\frac{235}{1000}$ of 1 per cent. The apparent difference in solubilities of lake and land pitch in petroleum ether is, in many cases, due to the larger amount of mineral matter present, as is fully shown in my paper to which Mr. Richardson refers, and from all mention of which fact I regret that he carefully refrains, especially, as in this instance, the difference in petroleum ether soluble is almost exactly balanced by the difference in the mineral matter.

### *Water in the Pitch and in the Lake.*

Mr. Richardson goes into the discussion of this section very fully, and shows that the pitch is saturated with a strong mineral water containing ferrous sulphate, which floats on the pitch in the kettle when it is melted. He says further, "There is also a remarkably large percentage of ammonia, the presence of which is of great interest." This ammonia comes from amido-compounds in the pitch.

### *Mineral Spring in the Lake.*

This water was obtained where it oozed up into a basin in the pitch, and was thought to be remarkable because it contained carbonates instead of sulphates. These carbonates are to be expected as a result of the reaction between sulphates and the bitumen of the pitch, the result of which is the de-oxidation of the sulphur with the formation of hydrogen sulphide, and, through the union of the carbon, the oxygen, and the bases, the formation of carbonates of the bases present.

### *Gas evolved by the Pitch.*

Under this section Mr. Richardson makes statements that do not correspond with my own experience. He says that at low temperatures carbonic acid is evolved from the pitch, while at "comparatively high temperatures," hydrogen sulphide appears. I have found that both lake and land pitch melt and evolve hydrogen sulphide below 100° C., and continue to do so until they are reduced to coke. This gas without any doubt is due to the reaction between the sulphur dissolved and precipitated and the pitch itself.

### *Mineral Matter in the Pitch.*

Referring to my copy of Mr. Richardson's paper, I find in the first column of page 7 a reference to the paper read by me before the American Philosophical Society, February 5th, 1897. No allusion is made in that paper to an alteration product of asphalt. In the matter referred to the expulsion of carbon is said to be complete in metamorphosed pyrobituminous shales, and not in any form of asphalt.

The silica in Trinidad pitch cannot be properly observed after combustion of the organic matter. The method that I have found most satisfactory is to observe the particles of mineral matter which are left after the bitumen has been dissolved in chloroform, upon a microscope slide. The portions of silica are exceedingly minute and suggestive of gelatinous silica. I have never observed any clay or alumina associated with the silica under these conditions, and I do not believe there is any of these two substances existing in crude Trinidad pitch that are not in combination with organic matter and dissolved by the chloroform. The mineral matter that Mr. Richardson describes as so impalpably fine that "it will not separate from a solution of melted or dried Trinidad pitch in any of the usual solvents even after days of standing," &c., is in solution as a compound of alumina with organic matter, and is not in any proper sense mineral matter. There appear to be two of these compounds, one of alumina soluble in turpentine and cold bisulphide of carbon, the other of iron and soluble in chloroform and hot bisulphide of carbon. Mr. Richardson's method of analysis does not distinguish them, except when he uses his centrifugal machine, which is a curious adjunct to a defective chemical process. I have a considerable amount of experience to support these views, but not yet so completely elaborated as to be wholly conclusive.

Mr. Richardson is wholly wrong in saying, "It has been thought by Peckham and others, on this account (because it will pass through the finest filters) to be chemically combined with the organic compounds of the asphalt." I think it is so combined because such compounds were long ago shown by Way and Voelcker and others to be present in soils of which humus forms any considerable portion, and certainly no one can study Trinidad pitch *as it is*, not its ashes, but the pitch itself, without discovering that it is closely related to a peaty soil, a fact further demonstrated by the large amount of vegetation rooted within the pitch itself, and not in soil lying upon it.

For, the organic matter not soluble consists of what? Certainly not leaves and grass blown onto the lake in any considerable amount, but the products of the partial decay of trunks, branches, and stumps of trees, that are mouldering in the pitch, entombed in a manner exactly similar to the slow mouldering of the vegetation of swamps that are buried in peat. Mr. Richardson says, "This cannot be separated from the mineral matter in Trinidad asphalt after the removal of the bitumen with solvents, but its ultimate composition can be obtained by a combustion of the mineral and organic matter and the calculation of the results to an inorganic free basis." There is no difficulty in removing the peat acids by solution in alkaline hydrates or carbonates. Of course, the usual precautions required in the treatment of peat soils are to be observed in this instance. With proper care the cellulose that is present will remain undissolved, and can be distinguished with the microscope. The iron sulphide, which is always present, is decomposed by combustion, the iron becoming ferric oxide, and the sulphur being burned, escapes as sulphurous oxide and is included in the "organic matter not soluble." Really this name *organic matter not soluble* has been a great misnomer. It is true that there may or may not be insoluble products of the oxidation of the pitch present properly included under this heading, but we really know little or nothing about them.

### *Bitumen of Land Asphalt.*

Mr. Richardson gives a table by which he attempts to show that the bitumen of land pitch contains more carbon than the bitumen of lake pitch. His figures do not warrant his conclusion. His average of land pitch contains C, 83·68, H, 10·84; total 94·52, of hydrocarbon; of which 88·426 per cent. is carbon, and 11·574 per cent. is hydrogen.

His lake pitch contains C, 82·33, H, 10·69; total 93·02, of which, 88·507 per cent. is carbon, and 11·493 per cent. is hydrogen.

The difference is 0·081 per cent., which is wholly without significance, as scarcely two samples of pitch can be found in either the lake or on the land that are any nearer alike.

### *Nature of the Sulphur Derivatives.*

In this paragraph he mixes up the native sulphur bitumens soluble in naphtha with the substitution and decomposition of sulphur compounds found in sludge acid. These latter compounds certainly have no relation to the composition of crude Trinidad pitch.

In all of the succeeding sections, which form a considerable portion of this remarkable paper, while the details describe an immense amount of labour in fractionation and analysis, the work described was carried out in such a manner that but little satisfaction can be had from the discussion. It is to be regretted that, after the triumphs achieved by Mabery through distillation *in vacuo*, so much labour should be expended on ordinary and destructive distillations with other methods that the experience of years has proved to furnish results of doubtful value.

I regard these last sections as largely subject to the same criticism as that bestowed elsewhere upon the work of Dr. Endemann; that is to say, that this work is to be taken for just what it is in itself, largely a research on decomposition products, and as such is valuable, but it must not be mistaken for a research upon natural bitumens, which it is not to any considerable extent.

## ON ASPHALT.

BY H. ENDEMANN, PH.D.

I HAVE shown in my last paper on Asphalt (J. Soc. Chem. Ind., Feb. 27th, 1897) that asphaltogen, when heated in air, passes finally into asphaltic oxid by loss of hydrogen and absorption of oxygen. This part of the reaction was verified by analysis. Inasmuch as an intermediate body is formed, which was insoluble in ether, but soluble in chloroform, I had accepted it for granted, that this body of intermediate formation must be asphaltene.

In repeating the experiments it was observed that the material always lost at first considerably, and only acquired weight again during the last stage of the process, when the increase almost equalled the whole weight of the oxygen found in the final product. Often repeated experiments invariably gave the same results and I, therefore, finally concluded that the formation of asphaltene might be problematical and endeavoured to obtain the body thus intermediately produced.

A considerable quantity of unpurified asphaltogen as it was obtained as residue from the distillation of a Texan asphalt was heated in an air bath in an open dish to 200° C. for several days. The asphaltogen, which had been perfectly soluble in ether, gave now a considerable insoluble residue, which, however, was easily soluble in chloroform. After evaporating the chloroform, a black brittle body was left behind, which was then analysed. On combustion it was ascertained that the body thus produced contained carbon and hydrogen in even atoms, *i.e.*, for $C_{26}H_{26}$. Inasmuch as asphaltene has the proportion between carbon and hydrogen as $C_{26}H_{36}$, it is evident that this body is not asphaltene and that, therefore, the oxidation has taken a course different from the one expected. The body thus produced is hard and brittle and possesses a very high melting point; in fact it seems that it cannot be melted without decomposition. It is soluble in chloroform and can be softened by the addition of petrolenes. It was found, however, that the quantity of petrolene required for such purpose must be far greater than would be required for asphaltene in order to obtain a mixture of equal pliability.

From the above it is seen that a real and hard asphalt cannot be produced by oxidation of liquid asphalts, as has been surmised by me and variously stated by others. I have met such artificial so-called asphalts in commerce and use. The analysis of these requires special elaboration.

As regards petrolene, or perhaps more properly speaking the petrolene hydrocarbons, I have nothing to report beyond what I have said upon several former occasions, namely, that they are a mixture of hydrocarbons of different series, wherein, however, the paraffins are present in much smaller proportion than in petroleum. The highest proportion found by me was 25 per cent. of the petrolene or the volatile hydrocarbons obtained from asphalt. It is not my intention to continue at present the examination of the petrolenes. The subject is one which requires more time and attention than I shall be able to give it in the near future.

It becomes now my duty to give some consideration to a few of the latest publications on asphalt and especially those of Messrs. Peckham and Richardson, which have appeared in the Journal of our Society.

While both declared that what I published was entirely against their experience in every respect, they failed to produce any facts supporting their assertions. They never gave my method a trial. They never defended or justified their own method, they simply reiterated it; and as to my statements, they let all go unchallenged except the one that solid asphalt contained oxygen.

Mr. Peckham's papers make no addition to our knowledge they merely complain that notions entertained and upheld by him since 1865 should be criticised in the light of new facts and research. As to the time required for my work, this is a matter which scientific men generally subordinate to the results obtained; and my effort has been to replace the methods which have been proved fallacious with something better. In his papers I found not one single fact complete in itself in the form of a proof, as chemists are in the habit of producing in support of their theories. He seems not to differentiate between facts and suppositions, so for instance he puts the proven facts that asphaltogen oxidises to asphaltic acid upon the same level with my guess that the latter may be the cause of the solubility of inorganic matter in asphalt solutions.

Mr. Peckham says that I had stated that petrolene was a chemical individual, while in all my publications I have invariably stated that what Bousingault had called petrolene consisted of a series of hydrocarbons. He also makes the statement that I found asphaltulmic acid in asphalt, while such a statement cannot be found anywhere in my papers.

Then again, where I speak of the analysis of refined asphalts, and prescribe the heating of the bitumen in a carbon dioxide current, I state the result of this treatment as follows: Loss, petrolene; residue, asphaltene, and ash. (This, Journal 1896, 874, col. 2, line 19 from bottom.)

Compare with this Peckham (this Journal, 1897, 425, col. 2, 2nd paragraph from end of page), where he claims that I gave the loss as *water* and petrolenes, and then devotes a paragraph of 15 lines to discussing the absurdity of my expecting water in refined asphalt.

Mr. Peckham has really made no effort to disprove my findings, nor does he defend his own methods. As to the latter, he simply recounts them, and though now two years have elapsed since my first publication, he still does not explain how the results of his analysis can be utilised.

If a lime stone to be used for cement manufacture is analysed, we learn from such analysis what to add to make a hydraulic cement of given composition. With the results of the Peckham-Richardson analysis we remain, as before, in the dark. However, Mr. Peckham, evidently feeling the need of complying with such a reasonable demand, has promised the solution of this difficult problem—for a later period, however, since, perhaps, two years time has not been sufficient for him to solve it. In place of a real argument in favour of the extraction method introduced by them, he, as well as Mr. Richardson, calls attention to the great accuracy of the method shown by the very concordant results obtained on its application. (See this Journal, 1897, 426, end of paper; 1898, 21, article headed "Naphtha Soluble Bitumen.")

I fully admit that the method used by them gives very uniform results. It is not on this point that I have condemned it, but on account of the fact that it is not an analysis. A fundamentally wrong method evenly applied is just as apt to give concordant results as the best analytical method. The results obtained by the Peckham-Richardson method are well adapted to create prejudice, and thus may be used to encourage the use of a certain asphalt in preference to others almost identically constituted.

Trinidad land asphalt evidently is derived from the same source as the lake asphalt. It is, however, a more ancient deposit, and by reason of exposure in a hot climate has lost a couple per cent. of the more volatile petrolenes. This is so according to the showings of my method of analysis. There is no other difference as far as the bituminous portion of the asphalt is concerned, at least none that is discernible at the present state of our knowledge. Unbiased one would say that the land asphalt was the better for having lost a treacherous constituent, which, when left, would cause undue hardening by subsequent evaporation. Not so the parties cited.

They teach that an asphalt, if it were to be used for paving, must contain at the outset more petrolene than asphaltene. (Richardson.)

So a Trinidad lake asphalt containing—

| | |
|---|---|
| Petrolene (Richardson) | 31·86 |
| Asphaltene (Richardson) | 28·12 |
| Non-bituminous | 40·02 |

is a good material, for the petrolene exceeds the asphaltene, while a Trinidad land asphalte containing—

| | |
|---|---|
| Petrolene (Richardson) | 21·97 |
| Asphaltene (Richardson) | 33·67 |
| Non-bituminous | 44·36 |

is, according to Richardson, a dangerous material to use; it will make too brittle a pavement, the valuable petrolene has been burned out by the sulphur, and the polymerisation into asphaltene has gone so far as to make the asphalt unfit for proper work. While polymerisation sounds well I must oppose the application of the term to this subject.

From my analysis I have found that the only difference consists in a lack of somewhat less than 2 per cent. of volatile hydrocarbons in the land asphalt; and by adding the missing quantity I should be able to produce a lake asphalt from land asphalt at short notice, if my views were correct, while such could not take place with the unalterably spoiled land asphalt as Richardson views it.

By taking the identical land asphalt with analysis as given above and adding thereto 2½ per cent. of petrolene or volatile hydrocarbon oils I obtained an asphalt, which analyzes by the Richardson scheme as follows:—

| | |
|---|---|
| Petrolene (Richardson) | 40·15 |
| Asphaltene | 19·50 |
| Non-bituminous | 40·35 |

and which surpasses in petrolene even the lake asphalt, and hence should be a marvel of superiority, according to these views.

This experiment proves also that the petrolene Richardson is a solution of asphaltene and asphaltogen in the volatile hydrocarbons or real petrolenes. These real petrolenes are soluble in petroleum ether, and unless they aided the solubility of the other constituents, the results of my analysis should have been as follows:—

| | |
|---|---|
| Petrolene (Richardson) | 26·80 |
| Asphaltene (Richardson) | 32·85 |
| Non-bituminous | 40·35 |

It will be noticed, however, that I obtained only 19·50 per cent. asphaltene; 13·35 per cent. asphaltene have, therefore, been induced to go into the petroleum ether solution by reason of the presence of 2½ per cent. extra volatile hydrocarbons added to the asphalt. This experiment is only an experimental modification of the facts stated and illustrated in my first paper.

Though both authors have nothing to say about my criticism of their method of analysis, they at least now seem to be of the opinion that their use of the names petrolene and asphaltene was wrong; and they acknowledge in so far the correctness of my criticism, as in their latest publications to drop these names almost altogether for the extracts by petroleum ether and the subsequent chloroform or bisulphide of carbon extract. They have likewise nothing to say about my asphaltogen and its quantitative relation to asphaltic oxide.

My preliminary remarks about petrolene have in part been verified by Mr. Richardson. He, however, shuns the name of petrolene, I suppose on account of the confusion which has been caused by confounding the volatile hydrocarbons with the petroleum ether soluble part.

So as far as the main part of my first paper goes, they would seem to agree with me completely, that they do not oppose them with any arguments or analytical data. There is one point, however, to which they object.

Both parties have so much to say about sulphur, and, incidentally, one of them also so much about its polymerising influence, that I consider it time to call attention to the absolute want of proof in support of these claims. Polymerisation by means of sulphur has not been demonstrated by Mr. Richardson, nor has polymerisation by any other means. All he has done is to expose the distilled and apparently partially cracked petrolenes on a roof during the hot summer months. He found the refractive index "perceptibly" (whatever that may mean) raised. He has neither shown whether the sample consisted still only of hydrocarbons, nor has he given us any certainty but that the refractive index was "perceptibly" raised, perhaps, solely by the evaporation of the lower boiling fractions, which also are those possessing the lower refractive index. Mr. Richardson has also applied F. Heusler's method of polymerisation upon the oils obtained by distilling asphalt, and found that the refractive index of the product was increased. Nevertheless, while not denying the possibility of polymerisation, we cannot be certain whether this raising of the refractive index was not due to the loss of 15 per cent. of the lowest boiling fraction of the oil, which, according to his showing, was also the one possessing the lowest refractive index.

At this stage of our knowledge of asphalt it is certainly not necessary to indulge in fantastic theories. Polymerisation may wait until it is found necessary, and especially until it has been actually demonstrated. That this cannot be done by such crude means as have been chosen every chemist will concede. Polymerisation consists in the laying together or combination of smaller molecules, which then form larger molecules of a more saturated character than the molecules from which they have been formed. According to Heusler, Engler, and Schumann, polymerisation is measured by the percentage of paraffins in an oil.

If an oil contains much sulphur, which, according to Richardson, is the polymerising agent, we must expect large quantities of paraffins. Yet the opposite is the case. The best results by means of the sulphuric acid treatment, which I was able to realise in the asphalt distillates was 25 per cent. of such paraffins, which figure is really higher than the truth, for the reason that sulphuric acid does not only remove the non-paraffins, but really is known to produce paraffins by polymerisation. We find, therefore, in reality just the opposite from what we should expect from Richardson's reasoning. Instead of finding in asphalt more stable compounds, we find the unstable to predominate. I found that of the more volatile hydrocarbons, 75 per cent. are destroyed as such by fuming sulphuric acid, while the less volatile asphaltogen shows its instability by a rapid loss of hydrogen and accumulation of 15 per cent. of oxygen when heated in air. According to this showing, there would have been very little polymerisation, inasmuch as in an asphalt containing, say, 33 per cent. of petrolenes, no more than 8 per cent. of paraffins could be obtained.

It is doubtful if Mr. Richardson would have applied to asphalt the theory evolved by the authors mentioned if he had understood their reasoning and their conclusions. But Mr. Richardson seems not to be clear in his mind how sulphur does act, for in other places in his paper he seems to incline towards my view of the presence of sulphur in asphalt, namely, as the result of the action of sulphur oxides upon the organic part of the asphalt. The more sulphur oxides have acted upon the asphalt the harder it will be. Under these circumstance it is the oxygen which has wrought the change, and the sulphur is merely an incident. This is my view based simply upon our general experience of the presence of sulphur and sulphides in carboniferous minerals. If sulphur were to act by removing hydrogen and thus polymerise molecules, I should think that the hardest asphalt should contain least of it. Actual experiment shows that sulphur up to temperatures of 150° C. does not only not perceptibly harden asphalt but rather seems to soften it.

Mr. Richardson has given in his paper (this Journal, January 1898, page 29), a long list of so-called analyses of asphalte from various sources and other similar materials. From these analyses it would undoubtedly appear that my contention that hard asphalts contain oxygen is wrong. He does not say in the paragraph entitled "Sulphur in Asphalt and its Influence," that his bitumen contains anything but the substances there mentioned, which figure up pretty well to 100 in every case, leaving nothing for anything else.

It is a rather curious incident that Mr. Richardson should have failed to notify us here that all these extracted (?) bitumens contain considerable quantities of mineral matter. He does mention it, however, in an extremely remote part of his paper, where he states incidentally on page 19, first column, that he has invariably been unable to remove the mineral matter and that the best he has been able to accomplish had been once, when a reduction to about 2 per cent. was obtained, while in the majority of cases it was much higher. The totals given or the imaginary 100 on page 29, in the last column, can therefore not be the actual totals of the material taken for analysis, but they must represent a calculated remainder obtained by the deduction of the ashes containing all oxygen compounds from the quantity of bitumen taken for analysis. The sulphur, which may have been in combination with the metals or inorganic elements, is all given to the organic matter, though I have shown already how it may be removed mechanically from bitumen solutions if not completely to at least a considerable extent.

How such analysis will mislead I will here illustrate. Supposing that we have a mixture of 75 per cent. rosin and 25 per cent. iron pyrites. Such a mixture is composed as follows:—

| | | |
|---|---|---|
| 75 per cent. rosin contains C .......... | 59·63 | |
| H .......... | 7·43 | |
| O .......... | .. | 7·95 |
| 25 per cent. pyrites contains S .......... | 13·35 | |
| | 16·67 | |
| and produce ashes of $F_2O_3$ .............. | 97·07 | |
| leaves for O .............................. | 2·93 | 2·93 |
| Oxygen lost by calculation.............. | | 5·02 |

According to the composition of rosin, we should have found 7·95 per cent. oxygen, yet according to the notion of Mr. Richardson for calculating an analysis, we find but 2·93 per cent. and 5·02 per cent. of oxygen disappear. In following out his scheme of calculation be then would say the substance contained 16·67 per cent. of ashes, there could have been, therefore, only 83·33 per cent. organic matter, which in 100 would contain: C 71·53, H 8·92, S 16·02.

If the oxide of iron by any chance should not have been heated sufficiently to drive out all the sulphuric acid, the error would then become still greater, inasmuch as the ashes would weigh still more.

7·95 per cent. of sulphur present as mono-sulphide of iron would produce an apparent deficiency of 6·82 per cent. oxygen.

2 per cent. of calcium present as sulphide would produce an apparent deficiency of 4·8 per cent. of oxygen, for the reason that not only would oxygen accumulate in the ashes but the sulphur also be retained.

From the figures given in the tabulated statement referred to, it is also evident to me that the bituminous extracts have been made with a minimum of solvent, *i.e.*, that no efforts have been made to facilitate the separation of sulphides by means of dilution as I have described (this Journal, 1897, 426). If for instance Trinidad asphalt is extracted by little chloroform and then the chloroform evaporated, a residue is invariably obtained containing 6 per cent. of sulphur and more. Addition of chloroform to this solution produces a precipitate containing the bulk of the sulphur, while the bitumen remaining in solution contains far less, *i.e.*, between 1½ to 2½ per cent. of sulphur. Mr. Richardson has observed the formation of a precipitate, but attributes it wrongly to the fine silt passing through the filter, and has also made no effort to learn the respective percentages of sulphur. It is possible that the substance which has been used by him for the extraction of the bitumen, namely, the bisulphide of carbon, may less readily allow of the separation of sulphides, which according to my own experiments are more easily soluble in bisulphide of carbon solutions of bitumen.

A crude asphaltogen was extracted by carbon bisulphide and gave a bitumen containing 0·33 per cent. of sulphur, when no effort was made to reduce the sulphur. There was then finely ground pyrites added and extraction performed by chloroform; the quantity of sulphur was found 0·536. Then using bisulphide of carbon upon the same mixture an extract was obtained containing S 0·678, Fe 0·591, or in proportion of constituents as they occur in pyrites. The whole of these extracts were made in the cold, and no effort was made to remove the sulphides from the solution by any of the means prescribed by me.

From the results of these experiments, I should say that bisulphide of carbon was less fit for the extraction of the bitumen if we desire to exclude such impurities as sulphur, though it does possess, as Mr. Richardson correctly states, the advantage of cheapness. Another difficulty with bisulphide of carbon is due to its easy decomposition, whereby sulphur is separated and may defile the bitumen. This requires that we should use only recently distilled material.

The sulphur, which is capable of passing into the bitumen solution, is generally only a fraction of the whole amount of sulphur found in the bitumen. Thus Texas asphalt, which I mention repeatedly, contains when analysed as a whole 0·95 per cent. sulphur, while the properly extracted bitumen contains 0·1 per cent. The reason for this is that most sulphur is present as sulphuric acid. Now compare this almost solid asphalt with 0·1 per cent. sulphur adhering to the bitumen with the petroleum oils of Lima, Ohio, which contain 0·75 per cent. sulphur, and then draw your own conclusions.

### DISCUSSION.

Prof. PECKHAM said in reference to Dr. Endemann's paper, that he had endeavoured in his criticisms of Dr. Endemann's work to be absolutely fair. He thought the difference of opinion between them arose from the fact that he himself had been endeavouring to formulate a process for separating natural asphalts into proximate constituents, while Dr. Endemann had worked on a decomposition product or products of natural asphalts. He had no criticism to make on Dr. Endemann's asphaltogen or any of the other compounds which he had isolated by his peculiar method. The author admitted that he had been at work for 30 years on California hydrocarbons, and he did not know what petrolene or what asphaltene were. He (Prof. Peckham) did not believe that there was any such thing as petrolene or asphaltene, or that any chemical formula could be assigned to petrolene. The author said that petrolene was that portion of an asphalt which was soluble in petroleum ether, and what it was depended upon what the asphalt was. If California asphalt were dissolved, or such a portion of it as would dissolve, in the most volatile portion of Pennsylvania petroleum, commonly called petroleum ether, a certain percentage of extract would be obtained. If one used the most volatile portion of California petroleum in the same way, they would get another percentage of extract. If one analysed in any rational manner California petroleum, they would find not a particle of paraffin in it. It would contain some sulphur compounds and a comparatively very large proportion of nitrogen compounds. Now, if one used Richardson's method, as Dr. Endemann had called it, for the analysis of California asphalt, one could divide it into so much so-called petrolene and something else, which one called asphaltene; and if one used Miss Linton's method, although the asphaltene was divided through the use of hot spirits of turpentine and chloroform, they would find no chloroform soluble part in California asphalt. But if on the contrary one took Trinidad pitch, there was no means of distinguishing the difference in the so-called asphaltene unless turpentine and chloroform were used. If one treated California bitumens with a solution of caustic soda, with water, or with strong acids, there was not an appreciable amount dissolved. Dilute acids dissolved the nitrogenous basic oils. Petroleum ether dissolved nearly all of the bitumen and boiling spirits of turpentine the remainder. With Trinidad pitch water in the first place extracted a portion, solution of carbonate of soda extracted another, solution of caustic soda a still further quantity. There were no basic oils soluble in dilute acid in any considerable amount. One could extract another portion with petroleum ether, another portion with spirits of turpentine, and the residue would only dissolve in chloroform. The author asked what was the use of comparing the results of those two methods and calling a solution of nobody knew what (because no one had analysed Trinidad pitch with a view to determining the hydrocarbons present) the petroleum ether soluble portion, petrolene? One could dissolve a portion of California bitumen getting nothing but benzols and naphthas and call that petrolene. The author said it was a mere manner of applying words, and that when any chemist isolated a substance, proved its composition, and gave it the name of petrolene and a formula, one should have petrolene; but as long as they were talking of something that might be a mixture of one series of hydrocarbons or another there was no significance in it.

Dr. ENDEMANN said in reply to Professor Peckham, that he had never said petrolene was a single substance. He had always said petrolene was a mixture of substances. He had shown already in his papers that petrolene was a mixture, and he took the name from Boussingault, who was the first to make real investigations in that respect. Petrolene, if not a chemical compound, consisted of volatile hydrocarbons like petroleum, but was distinguished from this by origin and composition, inasmuch as it contained but little of the saturated hydrocarbons or paraffins.

Dr. **Schweitzer** asked the author (Dr. Endemann) in what way he obtained the petrolene which he added to the land asphalt and made that peculiar lake asphalt.

In reply to Dr. Schweitzer, the author stated that he distilled it *in vacuo* from the liquid asphalt and then took about 2½ of it. It commences to boil at something like 260° and thence upward. He referred to the paper just read.

## Nottingham Section.

*Meeting held on Wednesday, October 26th, 1898.*

MR. J. O'SULLIVAN IN THE CHAIR.

## ON ARACHIDIC ACID IN RAPE AND MUSTARD OILS.

BY L. ARCHBUTT, F.I.C.

According to G. Ponzio (*J. für pr. Chem.*, Vol. 48 (1893), 487) rape oil contains about 0·4 per cent. of arachidic acid. I have at various times examined by Renard's process a large number (about 51) samples of commercial rape oil and have obtained from about two-thirds of the samples a crude fatty acid, or mixture of acids, melting at 70°–72° C., in even greater proportion than Ponzio states. The following results are taken at random from my note books :—

| Commercial Rape Oil. | Crude Arachidic Acid (not recrystallised). | Melting Point. |
|---|---|---|
| | Per Cent. | ° C. |
| Sample No. 1 ....... | 1·18 | 71·5 |
| " 2 ....... | 0·71 | 72·0 |
| " 3 ....... | 0·94 | 72·0 |
| " 4 ....... | 0·92 | 70·5 |
| " 5 ....... | 1·25 | 70·8 |
| " 6 ....... | 0·69 | 70·5 |
| " 7 ....... | 0·58 | 71·4 |

In order to ascertain whether undoubtedly genuine rape oil contains this acid, the following samples obtained from thoroughly reliable sources were examined with the results stated :—

| Description of Rape Oil. | Crude Arachidic Acid. | Melting Point. |
|---|---|---|
| | Per Cent. | ° C. |
| Crude rape oil, several years old, pressed from Rubsen seed. | 0·59 | 69·2 |
| Crude rape oil, several years old, pressed from Rapps seed | 0·36 | 69·0 |
| Kustendje (Black Sea) rape oil. | 0·84 | 70·4 |
| Refined Stettin rape oil ...... | 1·14 | 69·0 |
| | After purifying by one recrystallisation from 90 per cent. alcohol | |
| | 0·91 | 72·2 |

Finally, some Guzerat rape seed, chiefly yellow, with some brown seed mixed with it, was crushed and extracted with ether in the laboratory. The ethereal solution was washed with caustic soda solution and then with water previous to evaporation. 44·8 per cent. of oil was thus obtained, which gave the following results on analysis :—

| | | |
|---|---|---|
| Specific gravity at 60° F. ................... | 0·9133 | |
| | Per Cent. | |
| Saponification value......................... | 17·5 | |
| Iodine value.................................. | 97·8 | |
| Arachidic acid— | | M. Pt. |
| First estimation, on 10 grms.......... | 1·50 | 70·0° C. |
| Second " on 20 grms.......... | 1·61 | 71·1° C. |
| " after purification by twice recrystallising from 50 c.c. of 90 per cent. alcohol.................. | 1·43 | 73·1° C. |

The above results conclusively prove that genuine rape oil may contain as much as 1·43 per cent. of so-called arachidic acid, really a mixture of arachidic and lignoceric acids, though the proportion contained in different samples seems to vary considerably, and in some samples is either not present at all or contained only in traces.

I have also found this same acid, or mixture of acids, in mustard oil, one sample, the only one tested, having yielded 1·18 per cent. of crude arachidic acid melting at 69° C.

It appears from these results that the fact of finding arachidic acid in olive oil is not conclusive proof of the presence of arachis (earth-nut) oil, unless rape and mustard oils are proved to be absent.

### Discussion.

Dr. **Stanley Kipping** suggested that perhaps it was a little misleading to assign an exact "melting point" to a fraction of a degree to the mixture of fatty acids obtained from arachis (earth-nut) oil, when probably the "melting point" ranged over several degrees; it was well known that the separation by fractional crystallisation of two or more fatty acids of approximately the same molecular weight was almost impossible, and consequently the temperature of fusion of a preparation obtained from such a mixture would not be sharp, and would depend on the number of crystallisations to which the substance had been submitted.

## THE CONSTANTS OF CURCAS OIL.

BY L. ARCHBUTT, F.I.C.

I recently received for examination an undescribed sample of oil which at first I was unable to identify, owing to a want of agreement between the analytical numbers obtained and the published constants of any other oil. Subsequent information that the oil came from Lisbon and was possessed of purgative properties pointed to its being Curcas oil, though some of my results, especially the Hehner and saponification values, were quite at variance with the numbers given by Horn (this Journal, 1888, 442), and also by De Negri and Fabris (*ibid.* 1893, 454) (see Lewkowitsch, "Oils, Fats, and Waxes," p. 417). Physiological tests, however, confirmed the purgative property. I have now, by the kindness of Dr. Lewkowitsch, been furnished with a sample of undoubted Curcas oil, and the resemblance between it and my sample, especially the close correspondence of the analytical data, leaves no doubt that my sample was Curcas oil, and that the hitherto published data concerning that oil are in need of revision. The numbers for the crude and refined oils obtained in my laboratory by Mr. T. H. Adams, and those of previous observers, are given in the first table on next page.

The crude oil was yellowish brown in colour; the refined oil was pale yellow. The odour of both was faint, and not pleasant.

All three samples saponified readily, in contrast with the experience of De Negri and Fabris, who found prolonged boiling necessary.

The oil, as stated by Horn and confirmed by De Negri and Fabris, is only slightly soluble in alcohol, but readily soluble in petroleum spirit, which, together with the much lower viscosity, easily distinguishes it from castor oil. According to my experiments, 100 volumes of "absolute alcohol" (sp. gr. 0·7966 = 99·1 per cent. of alcohol by weight) dissolved at 15·5° C. about 2·17 volumes of the refined oil No. 1. Sample No. 2 required about the same proportion of alcohol, but left a very small quantity of insoluble fat or oil, which did not dissolve even on warming.

Arachidic acid was carefully searched for in the refined oil No. 1, but none was found by Renard's process in 10 grms. of oil.

When slowly cooled to 40° F. (4·4° C.) the refined oil No. 1 became turbid, and on exposure for four hours to a temperature of 37°—38° F. (2·8°—2·9° C.) it solidified. Exposed to the air in a thin film at 50° C. (0·1 grm. of oil on a glass plate 3 ins. square) the refined oil No. 1 dried in about 24 hours; cotton-seed oil simultaneously tested dried in about 18—20 hours, and refined rape oil (a good sample)

| Analytical Data. | (Undescribed.) Crude Oil. | (Undescribed.) Refined Oil. No. 1. | Refined Curcas Oil from Dr. Lewkowitsch No. 2. | Constants of Curcas Oil as stated by Girard. | Constants of Curcas Oil as stated by Horn. | Constants of Curcas Oil as stated by De Negri and Fabris. |
|---|---|---|---|---|---|---|
| Specific gravity at 60° F. | 0·9205 | 0·9205 | 0·9205 | 0·911† | 0·9192† | 0·920† |
| *Absolute viscosity at 60° F. | 0·858 | 0·878 | 0·864 | .. | .. | .. |
| Maumené value (rise of temperature in ° C. with 97 per cent. sulphuric acid. | 67·5 | 66·6 | 65·0 | .. | .. | .. |
| Saponification value, per cent. | 19·28 | 19·25 | 19·26 | .. | 23·05 | 21·02 |
| Iodine value | 98·0 | 99·5 | 98·8 | .. | 127·0 | 100·9 |
| Hehner value | .. | 95·2 | .. | .. | 87·9 | .. |
| Acetyl value (Lewkowitsch) | 14·03 | 9·82 | 8·36 | .. | .. | .. |
| Reichert-Meissl value | .. | 0·28 | 0·48 | .. | 0·65‡ | .. |
| Acidity (as oleic acid), per cent. | 11·8 | 0·36 | 4·46 | .. | .. | .. |
| Unsaponifiable matter, per cent. | .. | 0·56 | .. | .. | .. | .. |
| Melting point of fatty acids, ° C. | .. | 27·5 | .. | .. | .. | 24—26 |
| Colour reactions— | | | | | | |
| (*a*) With nitric acid (1·375) | .. | Pale brown, changing to orange on standing. | .. | .. | .. | .. |
| (*b*) With silver nitrate (Milliau) | .. | Negative | .. | .. | .. | .. |
| (*c*) With amyl alcohol, $CS_2$ and S (Halphen) | Negative | " | .. | .. | .. | .. |
| (*d*) With HCl and sugar | .. | " | .. | .. | .. | .. |

* The absolute viscosity of refined rape oil at 60° F. ranges from about 1·09 to about 1·16, and that of refined cotton-seed oil from about 0·82 to about 0·91.

† At 15° C.

‡ Reichert value.

dried in 48 hours. Dr. Lewkowitsch's sample of curcas oil dried rather less readily than mine, taking about 30 hours. These results show that in drying properties curcas oil more nearly resembles cotton-seed oil than rape oil.

## NOTES ON THE CONSTITUTION AND MODE OF ACTION OF THE DUNG BATE IN LEATHER MANUFACTURE.

BY JOSEPH T. WOOD.

In a paper read before the Society of Chemical Industry (this Journal, 1894, 219), the author gave an outline of the bateing process as far as he had then investigated it. Further researches have shown that, although the views then put forward must be somewhat modified, in the main they are correct.

In 1895–96 a series of experiments was undertaken with a view of ascertaining how much of the reducing action of the bate was due to, (1) the chemical constituents; (2) the bacteria; (3) the digestive ferments and enzymes.

The puer used was dog dung obtained freshly every week from hunting kennels. It contained on an average, water, 85 per cent.; organic matter, 10 per cent.; mineral matter, 5 per cent.; about 3 per cent. of the organic is soluble and about 1 per cent. of the mineral matter.

According to Gamgee (Phys. Chem. 2), a dog fed on flesh diet excretes from 27 to 40 grms. of fæces in 24 hours, of which 12·9 grms. are solids. On a bread diet the amount of fæces is much more.

An ultimate analysis gavo the following results:—

| —— | Bread Diet. | Meat Diet. |
|---|---|---|
| C | 47·39 | 43·44 |
| H | 6·59 | 6·47 |
| N | 2·92 | 6·50 |
| O | 36·08 | 13·58 |
| Mineral matter | 7·02 | 30·01 |

These figures alone are sufficient to show what a variable quantity is the puer employed in a tannery.

The mineral matter is composed as follows:—

| | | | |
|---|---|---|---|
| Soluble in water ... | NaCl and $Na_2SO_4$ | 1·37 | 4·02 |
| | Sodium phosphate | 2·65 | |
| Insoluble in water. | Earthy phosphates | 80·37 | 91·93 |
| | Ferric phosphate | 2·09 | |
| | $CaSO_4$ | 1·52 | |
| | Silicic acid | 7·94 | |

The ammonia compounds combine with the lime remaining in the skin, but the constitution of these compounds was unknown.

The puer was examined for ammonia by Schlösing's method, 50 c.c. being mixed with 50 c.c. milk of lime placed under an air-tight bell-glass, together with an open vessel containing a measured quantity of standard acid, for 36 hours. No $NH_3$ was given off.*

The mixture of dung and $Ca(OH)_2$ was filtered and a perfectly clear filtrate of a golden yellow colour obtained. This contained free amines, calcium salts of volatile and non-volatile acids.

It was distilled until the distillate was no longer alkaline, and made up to 500 c.c. 50 c.c. required for neutralisation 5·7 c.c. of $\frac{N}{100}$ $H_2SO_4$, equivalent to 0·1938 grm. of $NH_3$ per litre of original puer, being the amines set free by $Ca(OH)_2$ in cold solution.

A further 50 c.c. was distilled with NaOH, the amines given off neutralised 8 c.c. of $\frac{N}{1}$ HCl, equivalent to 2·72 grms. of $NH_3$ per litre. The neutralised liquid was evaporated to dryness; the residue, consisting of amine hydrochlorates, amounted 0·27 grm. A qualitative test showed both primary and secondary amines to be present.

The action of these amine hydrochlorides was tried upon skin, pieces of limed sheep grain previously washed in water being used. The solution was maintained at 35° C. In the first experiment 0·27 grm. was dissolved in 100 c.c. of water. In two hours the skin was considerably reduced, but had not the touch of a "pured" skin. It tanned well and was a good colour, showing that the lime had been removed, but the leather was not sufficiently soft.

With a strength of 1 grm. per 100 c.c. the action was hastened, the same result being obtained in 1¼ hours, but the coriin of the skin was not dissolved. A comparison piece of the same skin in dung was bated too much.

With a view of comparing the action of analogous bodies, skin was treated in a solution of aniline hydrochloride, 1 grm. per 100 c.c., for 1¼ hours at 35° C. The solution was acid, lime was removed, and the skin felt very similar to that in the previous experiment. Coriin was not dissolved, and the skin did not "come down."

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* This is not invariably the case: other samples of puer examined have given off $NH_3$ when similarly treated, but only in small quantity. A similar instance has been observed by Rideal and Orchard in the liquefaction and decomposition of gelatin by *B. Fluorescens liquefaciens* (The Analyst, Oct. 1897), where the quantity of ammonia produced was insignificant, amounting even after 16 days' incubation to only 0·168 grm. of N per 100 c.c., corresponding to 0·204 grm. of ammonia.

Of the mineral constituents mentioned above, only the chlorides* have a reducing action on skin, the other compounds being inert, *e.g.*, silicates, or merely supply food for the bacteria.

For a general outline of the decompositions taking place in dung, see Dr. Herfeld's paper (this Journal, May 1895). We need only consider the action of the chief organic compounds, as the amount of chlorine is too small (in one case 0·053 per cent. on the dry dung) to be of importance. The principal compounds are :—

*Organic Acids.*—Formic, acetic, butyric, valerianic, lactic, malic, tartaric, citric, and glyceric.

*Amido Compounds.*—Leucin, tyrosin, glutamic acid, glutamine, asparagin, glycocol.

*Bases*, consisting of amines, skatol, and indol, and varying amounts of ammonia according to the age of the dung.

The organic acids exist partly as salts of the alkaline earths, partly combined with amines. A distillation of "puer," after acidifying with $H_2SO_4$, gave 2·2 grms. per litre of volatile acids reckoned as acetic. No HCl came over. On decomposing the sodium salts of the acids with $H_2SO_4$ the smell of butyric acid predominated. I have also shown the presence of lactic acid in the dung, but have been unable to determine the amount.

*Action of Amido Compounds.*—A mixture of glycocoll and leucin† was prepared by boiling 8 grms. of gelatin in 400 c.c. of water, acidified with 1 c.c. of HCl for two hours with inverted condenser. The acid was then neutralised with ammonia. The solution had a very considerable reducing action on skin at 35° C. in 90 minutes, but not sufficient for practical purposes. It appears to dissolve a little coriin in a similar way to dilute acids. It was found on further investigation that the action was largely due to the $NH_4Cl$ present.

*Action of Dilute Acids.*—Although the bate in practice is almost always alkaline, yet a fresh bate is *acid*, and it may be as well, before proceeding, to consider briefly the action of weak and dilute acids on skin.

The fibres of the skin have only a limited capacity for holding acids, and soon begin to swell abnormally and partially dissolve. Reimer (H. R. Procter, "Text Book of Tanning," 1885. p. 18) has found that the material may be reprecipitated by lime water. It forms a fibrous mass, which has not the sticky feel of gelatin, but is at once converted into that body by boiling.

The author has examined the action of dilute $H_2SO_4$ on sheep skins, and this may be considered typical of the action of acids on skin generally.

When pured and drenched sheep skin is treated for 20 minutes with a very dilute solution of $H_2SO_4$ ($\frac{1}{280}$). It swells up considerably and becomes soft and semi-transparent. The fibres of the skin which under normal conditions have a diameter of 4$\mu$—6$\mu$ become 20$\mu$ or more; on staining with picro-carmine there are seen to be certain fibres which are unaffected or only slightly affected by the acid. These are the elastic fibres (see Procter, "Text Book of Tanning," pp. 8 and 21) and the capillary blood vessels.

On filtering a solution of the above strength in which skin in excess has been treated for 20 minutes, and evaporating 100 c.c. to dryness in a platinum dish, a residue of 0·4992 grm. was obtained. A second experiment gave 0·496 grm. of which 61 per cent. was organic matter.

By Kjeldahling another portion of the residue the N in 0·12 grm. amounted to 0·0122 grm. equivalent to 0·073 grm., of skin substance; *i.e.*, 60 per cent. of the soluble matter is skin. It is evident that the matter in solution is a compound of skin substance and $H_2SO_4$ together with a little soluble mineral matter contained in the skin. The amount of substance dissolved depends upon the condition and previous treatment of the skin.

In the presence of NaCl, which prevents the swelling of the fibres, pured sheep grains take up 0·7 c.c. of $\frac{N}{1}$ $H_2SO_4$ from a soluble containing 1·7 c.c. of $\frac{N}{1}$ $H_2SO_4$ and 7·8 grms. of NaCl per 100 c.c. (0·833 grm. $H_2SO_4$ per litre). About 300 grms. wet skin per litre were used. The skin substance dissolved by the acid in the time named does not appear to be more than that dissolved by the weak acids of a drench, although an equivalent weight of $H_2SO_4$ has a much more powerful swelling action on skin than these acids.

A piece of pured and drenched sheep skin was carefully washed in distilled water and then dried *in vacuo* until it ceased to lose weight, the absolute dry was 4·2670 grms. The skin was soaked down in distilled water and again drenched for 30 hours in a clean bran drench, developing approximately 0·8 grm. lactic acid, and 0·5 grm. acetic acid per litre (see this Journal, May 1893). It was again washed and dried *in vacuo*. It weighed 4·12 grms., a loss of 0·14 grm. = 3·44 per cent.

The action, therefore, of the acids is a reducing one, inasmuch as skin is dissolved, but as the fibres hold a certain amount of acid the skin appears to be plumped. Where the acid is a mineral one the skin tans plump, but with a brittle and inelastic fibre, weak organic acids make a plump, soft leather with a moderately elastic fibre, giving to the skin a somewhat india-rubber feel. We thus see that weak acids alone do not produce a similar result to a dung bate, which causes the skin to fall and produces a flat soft leather with a stretch in it, which will not spring back.

I have also tried the action of the following Na and $NH_4$ salts upon skin :—

*Sodium lactate*, $C_3H_5O_3Na$, prepared by neutralising 2 grms. lactic acid with sodium carbonate in 1000 c.c. water. The solution was used under the same conditions as the other bateing experiments, *viz.*, at 37° C. the reducing action was nil, but on prolonging the digestion, the medium was found to be very suitable for the development of putrefactive organisms, and, as a consequence, the skin was attacked and partly peptonised.

*Amonium lactate*, prepared in a similar way, had an almost identical action on the skin, but was more effective in removing the last traces of lime.

*Ammonium butyrate*, prepared as above, removed lime but instead of a reducing action it appeared to plump the skin slightly.

### *Action of Bile.*

Amongst other compounds in the dung which might have some action on skin were the bile salts and bile colouring matters. (For a full description of these, see Gamgee, Phys. Chem. 2.)

Experiments were made with ox bile, 25 c.c. being diluted with 250 c.c. of water. Pieces of sheep grain previously washed in water were digested in this solution at 37° C. for 1—4 hours. The bile had no reducing action on the skin and in fact hardened it at the same time staining it a dirty yellowish brown, the colour differing from that of the colouring matter of dung (hydro-bili-rubin). Even after continued standing there was little or no development of bacteria, the bile appearing to have an antiseptic action.

### *Action of Bacteria.*

Having ascertained the action of the chief chemical constituents of the dung, that of the bacteria was next examined. In the first experiment, eliminating all but bacterial action, a tube of nutrient gelatin was inoculated from an active bate. In two days the gelatin had liquefied along the needle track.

The culture was now transferred to the following solution :—

| | |
|---|---|
| Gelatin | 4 grms. |
| Dextrose | 4 „ |
| $K_2HPO_4$ | 1·0 „ |
| $MgSO_4$ | 0·2 „ |
| NaCl | 0·4 „ |
| Water | 2,000 c.c. |

The growth in this was fairly rapid and the smell very slight at the end of a week. A piece of washed sheep grain worked in this culture at 37° C. for four hours was considerably reduced, the grain was not attacked, the action differing in this respect from a prolonged puring. This mixed culture was grown in various other nutrient media, gelatin-

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* Procter, "Text Book of Tanning," p. 85.

† According to the text books; but for later researches see Paal and Schilling, Chem. Zeit. 1895, 1487; also this Journal, 1898, 589.

gelatin and mineral salts, broth made from sheep and calf fleshings, &c., with practically the same results. In no case did the bateing action equal that of dung, and was insufficient for practical purposes.

A cursory bacteriological examination of dung will show that the number of species of bacteria it contains is very large, but on making a plate cultivation from a sufficiently dilute infusion of fresh dog dung, most of the colonies developing appear to belong to four or five species. The following is a list of all the organisms which so far as my knowledge goes, have been isolated from different kinds of dung:—

*List of Known Bacteria in Dung.*

1. Bacillus fluorescens putridus
2. „ „ liquefaciens
3. Proteus vulgaris.
4. „ mirabilis.
These orgaisms have been formerly described as Bacterium germs by various authors.

5. Bacillus subtilis.
6. Micrococcus fulvus.
7. Bacterium ureæ. (Cohn.)
J. T. Wood, this Journal, 1890, 28.

8. Bacillus saprogenes, I., II., III. *Vide* Herfeld, this Journal, May 1895.
9. Bacillus butyricus.
10. B. putrificus coli.
11. B. pyocyaneus.
12. B. janthinus.
13. B. coprogenes fœtidus.
14. B. pyogenes fœtidus.
15. B. prodigiosus.
16. B. zenkeri.
17. B. magnus.
18. B. spinosus.
19. B. liquefaciens.
20. B. coli.
21. B. duclauxii = B. ureæ.
22. Micrococcus ureæ.
23. „ „ liquefaciens.
24. Proteus sulphureus.
25. Bacterium sulphureum.
26. Bacillus amylobacter.
27. Clostridium butyricum.
28. B. acidi paralactici.
29. Bacillus I.
30. „ II.
31. „ III.
Isolated from horse manure by S. A. Severin, Centr.-Bl. f. Bact. **2**, [1], 97.

32. Spirillum serpens.
33. „ tenue.
34. „ undula.
35. „ volutans.
36. Vibrio I.
37. „ II.
38. „ III.
Isolated by Kutscher, Zeit. f. Hyg. **20**, 45.

39. Spirillum from pig dung. Smith. Centr.-Bl. f. Bact. **16**, [1], 124.
40. Streptococcus liquefaciens coli. Gamgee, Phys. Chem. **2**.
41. Bacillus mycoides.*

There are besides many unnamed species of bacteria in dung, among which are the two following isolated by me from puer—

(a.) *Plate Cultures.*—Small yellowish colonies, slightly fluorescent, liquefy the gelatin slowly. Rods resembling *B. Subtilis* when at rest, but moving with a rapid undulatory motion. Cultures in nutrient gelatin have a considerable reducing action on skin.

(b.) *Plate Cultures.*—Bluish colonies liquefying gelatin rapidly, micro-bacteria in pairs. Not so marked an action on skin as (a). The organism resembles *Proteus vulgaris*, but swarming islets not observed.

Of the species mentioned above, a pure culture of *B. Subtilis* in meat broth, maintained for one week at 35° C., had *no* reducing action on skin. *B. fluorescens liquefaciens* had a moderate reducing action.

The species developing on the plates vary according to the age of the dung. In horse manure, according to Severin (Centr.-Bl. **2**, [1], 97), bacilli predominate in the early stages. At the end of two to three weeks micro-bacteria, cocci and diplococci make their appearance, while the bacillar form becomes scarce. In three months cocci predominate, forming zoogloæ; strepto- and staphylo-cocci and spirilli were seldom found. Yeasts and sarcina were never observed. From observations extending over several years it is evident that a similar cycle occurs in dog dung. When quite fresh it contains comparatively few bacteria, then certain groups of species take possession, causing decompositions in the dung; these in their turn give place to other species, which decompose the products formed by the first, so that no one species produces or can produce the complex chemical and physiological changes which take place, or the bodies necessary for the bateing of skin, as has been supposed by some observers.

None of the experiments, either with mixed or pure cultures of bacteria, had a perfect reducing action on skin, although the action was considerably greater than that of the chemical solutions. It was found, however, that by adding a small quantity of the amines above mentioned to the bacterial cultures that the reducing action became nearly as rapid and effective as with dung itself, and it was, therefore, clear that the principal bateing effect was due to a combination of two things, *viz.*, an active growth of bacteria in the presence of amine compounds.

It was now necessary to ascertain the mode of action of the bacteria on the skins.

I have previously shown (this Journal, 1894, March 31, 218—221) the action of digestive ferments or enzymes to be notable in the case of pancreatine. A careful study of the behaviour of the digestive enzymes in the animal body shows, however, pretty conclusively, that they are all destroyed before the fæcal matter is discharged (see Gamgee, Phys. Chem. **2**), and, therefore, the enzymes in the dung are not from this source.

An attempt was made to filter the dung diluted with water through a Berkefeld filter, and thus obtain a clear filtrate containing all the enzymes and free from bacteria; it was found impossible to filter the liquid in this way.

The method of Claudio Fermi (Ann. de Micrograph, **2**, [6], March 20, 1896), was also tried, but a clear filtrate could not be obtained.

The author has succeeded in obtaining the enzymes best in the following manner:—About 150 c.c. of the above-mentioned "puer" was well mixed with an equal quantity of glycerin and allowed to stand for seven days; it was then capable of being filtered on a filter pump, although very slowly, and yielded a clear filtrate of a deep golden brown colour; the filtrate was poured in a thin stream into a tall vessel containing about 1,500 c.c. of 98 per cent. alcohol. A copious flocculent precipitate of the albuminous matters and enzymes was thrown down, the alcohol was filtered off and the precipitate washed on the filter with absolute alcohol, and dried over sulphuric acid. The resulting amorphous body was of light brown colour and became darker when exposed to the air. The amount obtained from 150 c.c. of dung was 0·55 grm. (3·66 grms. per litre). The body consists of a mixture* of all the enzymes existing in the dung along with other albuminous bodies. It has a very slight diastatic action on starch. 0·5 grm. in 100 c.c. of water at 35° C. had a very considerable reducing action on skin. A further experiment with 0·5 grm. of amine hydrochlorides, 0·5 grm. of enzymes and 100 c.c. of water, at 35° C. brought down a piece of limed sheep grain in 30 minutes exactly like puer; the reaction of the solution at the commencement of the experiment was faintly alkaline; at the end of the experiment it was considerably alkaline.

It was noted that the fæcal odour of the glycerine solution disappeared on standing for two or three weeks, and the solution smelt strongly of ethyl butyrate, the enzymes being still present.

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* Since the above was written, Severin (Centr.-Bl. **3**, [2], 628) has described seven other species of bacteria isolated from horse dung: for the description and property of these the original memoir must be consulted. Popp and Becker, German Patent 86,335, 28, describe 14 species which they isolated from dog and pigeon dung, of which three have considerable bateing action on skins and which form the subject of the patent. Some of these are probably identical with organisms in the above list.

* I did not consider it necessary to purify the enzymes, the object being to examine the action of the bacterial products apart from the living organisms.

These enzymes were prepared from dung, and it now remained to prepare them in the laboratory by the action of bacteria alone. For this purpose 200 c.c. of a mixed culture of dung bacteria in the solution above described, seven days old, was mixed with 200 c.c. of dilute alcohol (alcohol 65, water 100) and well shaken. Gelatin and albuminoid bodies are by this means precipitated. The liquid was filtered and poured into eight times its volume of 98 per cent. alcohol. The precipitate which came down was washed with absolute alcohol and dried in the usual way. The enzymes thus obtained were redissolved in water and the former experiment with skin repeated with this solution. The same effect was produced, showing conclusively that it is the enzymes produced by the bacteria, acting in conjunction with the amines which bate the skin. It would seem that the special action of the enzymes is aided by the presence of amine compounds, in addition to the chemical action which these latter have upon the skin. The action is shown to be interdependent, *i.e.*, bacterial action alone is insufficient, and chemical action alone is insufficient, the true bateing action being a combination of the two.

In conclusion the author has pleasure in acknowledging the valuable assistance of Mr. H. S. Shrewsbury in carrying out some of the experiments.

### Discussion.

The Chairman stated that it was of much interest to note that the author and others to whom he had referred had found no chlorine, or next to none, in dog's dung. If it were looked for in a water extract of the incinerated dung, the chlorine would not be found, for, from his experience, a chlorine estimation in substances containing much organic matter cannot be made in the ash, and this applies as well when the chlorides of the alkaline earths are present as to the chlorides of the alkalis. A large proportion of lime must be added before incineration. There can be little doubt that the published estimations of chlorine in plants, wine, and milk, &c., made in the ashes of these do not give the amounts present.

In consequence of what appears to be the normal constitution of the fæcal matter towards the end of the alimentary canal in all animals, swarming as it does with bacteria, it would not be reasonable to expect the digestive fluids of the dog to pass unchanged in the dung, and he agreed with Mr. Wood that they cannot be considered to take any part in the process so carefully studied by him.

The papers read are highly suggestive, and show what useful, though difficult, ground the authors wish to cultivate.

Mr. J. T. Wood briefly thanked Mr. O'Sullivan for calling his attention to the point in question. He had estimated the chlorides in a watery extract of the ash of the dung, but he did not know how they had been estimated by the author he quoted. At the same time he was sure that the amount of chlorides originally existing in the dung was insufficient to have any appreciable effect on the skins.

## Scottish Section.

*Opening Meeting held at Glasgow, November 1st, 1898.*

PROF. G. G. HENDERSON IN THE CHAIR.

The Chairman, in the course of a few introductory remarks, expressed the congratulations of the Section to Mr. G. T. Beilby on the well-merited honour conferred upon him in his election to the highly important office of President of the Society, and thanked him in the warmest manner for his distinguished services to the Section while occupying the post of chairman. He also reminded the meeting that almost six years ago the Section had passed a resolution, calling for the elevation of the minimum flash point of burning oil to 100° F. (Abel test), and congratulated the members in general, and Mr. D. R. Steuart in particular, that the Petroleum Committee in their recent report had recommended the adoption of this most necessary change.

## SOME CHEMICAL PROPERTIES OF SCOTCH COAL.

BY W. CARRICK ANDERSON, M.A., B.SC., AND JAMES ROBERTS.

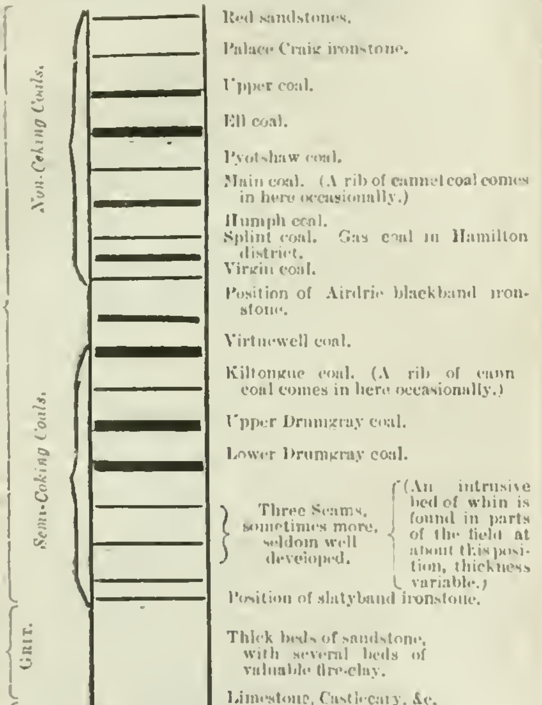
In bringing this subject before the Society of Chemical Industry in a paper, it would be unnecessary to advert at all to the importance that attaches to a thorough and complete knowledge of the substance of coal, were it not that, even in scientific quarters, the latent potentialities of the coal industry have sometimes been overlooked.

An output last year of over 200 millions of tons in the United Kingdom alone, and of 190 millions in the United States of America indicates with sufficient vividness the magnitude of the interests that are directly affected by any practical improvement in the application of the product from this fundamental industry. A polluted atmosphere, and a sun rendered invisible for many days each year to hundreds of thousands in every manufacturing city, offer a perpetual indictment of the present wasteful and unscientific methods of using fuel, and are a sufficient indication of how far increased knowledge would benefit at once the community at large. At the recent meeting of the British Association in Bristol, Sir Wm. Crookes in his presidential address drew attention to the probability in the not distant future of a scarcity in the staple article of food of a very large section of the human race, through the tendency of the white population to outgrow the limits imposed by nature on the supply of wheat-bearing land. The only possible means of averting the crisis was, he pointed out, to increase the fertility of the soil capable of carrying wheat by the administration of nitrogenous manures. But to do this adequately, 30 years hence, will, he believes, be impossible with the means which are now in use. At present $1\frac{1}{4}$ million tons of nitrate of soda are consumed each year; 30 years hence 12 million tons additional would be annually required. But even at the present rate of export, and without allowing for increased demand, the deposits of caliché will be exhausted in from 20 to 30 years. The existence of a source of combined nitrogen in the ammonia got from coal is admitted, but "all Europe does not yield more than 400,000 tons of sulphate annually . . . . . . . and in view of the unlimited nitrogen required to substantially increase the world's wheat crop, this slight amount of coal ammonia is not of much significance." In arriving at the conclusion that the imminent danger, to which he points, is only to be averted by the wholesale fixation, within the next generation, of atmospheric nitrogen to the equivalent of 13 millions of tons of nitrate of soda per annum, Sir Wm. Crookes ignored the possibility of a more scientific utilisation of the world's vast supplies of mineral fuel, placing at the disposal of the farmer a much larger proportion of the enormous stores of combined nitrogen existing therein in every one of the continents. What that might be can be gathered from the figures quoted above. An annual output of 200 million tons of coal, containing on an average 1 per cent. of nitrogen, represents two million tons of combined nitrogen, equivalent to nearly $9\frac{1}{2}$ million tons of sulphate of ammonia from the United Kingdom alone, if it were possible to recover it completely in that form. But even the present imperfect methods of recovery would be capable of producing about $1\frac{1}{4}$ million tons annually in this country, if the whole of the output were carbonised, and the ammonia recovered, as is done in the gas-works and blast furnaces at the present moment. The production of Continental Europe would be probably as much, while America, India, China, and many other countries will furnish for a much longer period than 30 years contributions of no inconsiderable amount.

At the same time, while very much remains to be done, it cannot be said that no effort has been put forth towards a more economical and wholesome employment of fuel. The technical treatment of coal, both in distillation and in combustion, has made enormous advances, and genuine improvements in processes have frequently yielded to their fortunate devisers the profits that rightly belong to those, who, by the application of skill and ingenuity, have increased the material wealth of the whole community.

But, in dealing with the subject of coal, technical methods are beset with the difficulties that arise from the material which they treat being almost infinitely various in composition and in properties. A system, proved efficacious in one instance, has to be modified to suit another class of material, even analytically of very similar composition, or may fail to treat it successfully altogether. We come across coals in which the percentages of the elementary constituents are almost identical,—isomeric they have been called—but whose properties are totally dissimilar, the products of their destructive distillation are different, and their action towards reagents is diverse.

*General Section of the Clyde Coal Basin.*


Upper Coal Measures.
Non-Coking Coals.
Red sandstones.
Palace Craig ironstone.
Upper coal.
Ell coal.
Pyotshaw coal.
Main coal. (A rib of cannel coal comes in here occasionally.)
Humph coal.
Splint coal. Gas coal in Hamilton district.
Virgin coal.
Position of Airdrie blackband ironstone.
Virtuewell coal.
Semi-Coking Coals.
Kiltongue coal. (A rib of cann coal comes in here occasionally.)
Upper Drumgray coal.
Lower Drumgray coal.
Three Seams, sometimes more, seldom well developed.
(An intrusive bed of whin is found in parts of the field at about this position, thickness variable.)
Position of slatyband ironstone.
Millstone.
Grit.
Thick beds of sandstone, with several beds of valuable fire-clay.
Limestone, Castlecary, &c.
Sandstones.
Limestone, Arden, &c.
Hirst coals.
Carboniferous Limestone Measures.
Strongly-Coking Coals.
Hard sandstones, with a few thin coal seams.
Limestones, Index, Cowglen, &c.
Three or more coals, including Bannockburn Main, Kilsyth Haughrigg, Possil Main, and position of the Lesmahagow cannel.
(An intrusive bed of whin is found in parts of the field at about this position, thickness variable.)
Sandstones and shales, with thin coals.
Kilsyth coking coal.
Sandstones and shales, with thin coals.
Limestone and coal, Hurlet, Hosie, &c.
(On the northern limits of the basin a thick bed of whin is found here.)
Calciferous Sandstone Measures.

The remedy that naturally suggests itself for this state of things, and the only remedy that can be completely satisfactory, is to endeavour, even though it may be by slow and difficult stages, to arrive at some clear conception of what coals are, of what it is that conditions the difference in their behaviour even when similar in elementary composition, so that thereafter the means can be with certainty adapted to secure the most economical employment of them for specific purposes.

In the belief that this end will be materially furthered by the accumulation and comparison of evidence drawn from the different localities where coal is found, we desire to give a description of some of the experiments which in the course of the last two years we have made on certain typical coals of the West Central Coalfield of Scotland.

In a paper read by one of the present authors before the Philosophical Society of Glasgow, and published in the "Transactions for 1897–98" (this Journal, 189 ), a description was given of the physical characters and technical applications of the products from the principal seams of this field. The names and relative positions of the seams are indicated in the accompanying section, which we reproduce from the paper referred to.

With regard to the section it ought to be kept in view that it is not known to be completely developed in any one part of the field; the upper strata are represented in the southern, and the middle and lowest only in the northern and eastern area of the basin, at least to a payable extent. The character of the seams themselves varies to some extent from point to point, and their chemical composition is by no means even approximately constant; yet the figures given below may be regarded as fairly typical over a large area, and represent the composition of the samples dealt with in the experiments described on next page.

Table II. shows the composition of the organic matter in these well-known coals. They were selected for investigation from the most various localities of Lanarkshire and Stirlingshire as typical products of the coalfield, and with no intention of using them to support any preconceived theory or view regarding either their formation or their constitution. In these circumstances, one cannot fail to be struck with the close agreement in composition which they present. Sample B, for example, is identical almost to within the limits of experimental error* with that marked "K." Sample "D," the gas coal associated with the splint in the Hamilton district, is exactly similar to that marked "H," the first of the Drumgray coals; samples M and O are not less remarkable in point of similarity. The ultimate composition of the famous Lanarkshire splint shows no divergence from that of the soft coals above and below it, such as one might reasonably anticipate from the remarkable properties that render it so valuable from an industrial point of view; and in particularising these we wish merely to emphasise the outstanding feature of the list, the close approximation which these bodies, drawn from localities geographically far apart and from strata of different geological age, present in chemical composition. Taken by themselves, they seem to afford abundant support for the theory advanced many years ago by Baltzer (Vierteljahresschrift der Züricher naturforschenden Gesellschaft, 1872), to the effect that, leaving out of account the water, resinous bodies and enclosed gases which they contain, coals are essentially mixtures of complex carbon compounds, which form a genetic and probably homologous series, of complex structure analogous to that of the aromatic series.

It has been known, however, for a very long time, that such similarity, and even identity, of chemical composition may be the concomitant of widely different chemical and physical properties. As far back as 1857 it was pointed out by Stein that two samples of coal identical in ultimate composition may give entirely different results when ignited in absence of air. One may yield a firm, metallic-looking cake, a true "coke," the other only a soft, partially coherent or even a pulverulent residue. And the percentage weights of so-called "fixed carbon" thus obtained, may be equally unlike. The latter point is shown by the figures given in Table III. below.

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* We may say that for the purposes of this investigation the ultimate analysis of these coals was done with the greatest care, triplicate assays being made in each case.

### Table I.

*Ultimate Analyses of Coals.*

| | Hamilton District. | | | | | Airdrie District. | | |
|---|---|---|---|---|---|---|---|---|
| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. | H. Upper Drumgray. |
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 4·52 | 4·98 | 4·82 | 5·54 | 5·10 | 4·97 | 5·37 | 5·68 |
| Carbon | 71·88 | 73·62 | 75·50 | 76·16 | 74·67 | 71·83 | 73·30 | 77·14 |
| Oxygen (and organic sulphur). | 11·10 | 9·50 | 8·71 | 7·52 | 8·62 | 8·80 | 7·83 | 7·71 |
| Nitrogen | 1·53 | 1·54 | 1·50 | 1·52 | 1·54 | 1·68 | 1·65 | 1·65 |
| Moisture | 9·99 | 9·08 | 7·27 | 5·56 | 7·77 | 6·90 | 10·45 | 4·70 |
| Ash | 0·98 | 1·28 | 2·20 | 3·70 | 2·30 | 5·82 | 1·40 | 3·12 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

| | Airdrie District. | | | Slamannan. | | | |
|---|---|---|---|---|---|---|---|
| — | I. Upper Drumgray. | J. Lower Drumgray. | K. Lower Drumgray. | L. Lower Drumgray (iii.) | M. Kilsyth Haughrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 4·66 | 5·02 | 5·00 | 4·37 | 5·06 | 5·14 | 5·20 |
| Carbon | 77·17 | 75·17 | 77·33 | 83·87 | 80·67 | 82·80 | 81·50 |
| Oxygen (and organic sulphur). | 9·57 | 9·92 | 9·78 | 3·81 | 7·50 | 5·67 | 7·53 |
| Nitrogen | 1·88 | 1·45 | 1·90 | 2·12 | 1·84 | 1·89 | 2·04 |
| Moisture | 3·82 | 7·14 | 4·02 | 1·83 | 1·98 | 1·75 | 1·72 |
| Ash | 2·90 | 1·30 | 1·97 | 4·00 | 2·95 | 2·75 | 2·01 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

### Table II.

*Ultimate Analyses of Dry Organic Matter.*

| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. | H. Upper Drumgray (i.) |
|---|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 5·07 | 5·56 | 5·32 | 6·11 | 5·67 | 5·70 | 6·09 | 6·16 |
| Carbon | 80·74 | 82·13 | 83·41 | 83·92 | 83·03 | 82·30 | 83·16 | 83·69 |
| Oxygen (and organic sulphur). | 12·47 | 10·59 | 9·62 | 8·29 | 9·59 | 10·08 | 8·88 | 8·36 |
| Nitrogen | 1·72 | 1·72 | 1·65 | 1·68 | 1·71 | 1·92 | 1·87 | 1·79 |
| | 100.00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

| — | I. Upper Drumgray (ii.) | J. Lower Drumgray (i.) | K. Lower Drumgray (ii.) | L. Lower Drumgray (iii.) (Slamannan). | M. Kilsyth Haugbrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 5·00 | 5·48 | 5·32 | 4·64 | 5·32 | 5·38 | 5·40 |
| Carbon | 82·73 | 82·10 | 82·26 | 89·06 | 84·86 | 86·70 | 84·67 |
| Oxygen (and organic sulphur). | 10·26 | 10·83 | 10·40 | 4·05 | 7·89 | 5·94 | 7·82 |
| Nitrogen | 2·01 | 1·59 | 2·02 | 2·25 | 1·93 | 1·98 | 2·11 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

### Table III.

*Proximate Analyses of above Coals.*

| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. | H. Upper Drumgray (i.) |
|---|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Moisture | 9·99 | 9·09 | 7·27 | 5·56 | 7·77 | 6·90 | 10·45 | 4·70 |
| Ash | 0·98 | 1·28 | 2·20 | 3·70 | 2·30 | 5·79 | 1·39 | 3·12 |
| Volatile matter | 33·33 | 35·84 | 36·22 | 38·25 | 33·53 | 37·73 | 35·25 | 40·27 |
| Fixed carbon | 55·70 | 53·79 | 54·31 | 52·49 | 56·40 | 49·58 | 52·91 | 51·91 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

TABLE III.—*continued.*

| — | I. Upper Drumgray (ii.) | J. Lower Drumgray (i.) | K. Lower Drumgray (ii.) | L. Lower Drumgray (iii.) (Slamannan). | M. Kilsyth Haughrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Moisture | 3·82 | 7·14 | 4·02 | 1·83 | 1·98 | 1·75 | 1·72 |
| Ash | 2·81 | 1·57 | 2·01 | 4·00 | 2·95 | 2·75 | 2·01 |
| Volatile matter | 35·83 | 36·78 | 33·81 | 20·61 | 32·55 | 27·40 | 28·45 |
| Fixed carbon | 57·54 | 54·51 | 60·16 | 73·56 | 62·52 | 68·10 | 67·82 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

TABLE IV.

| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. | H. Upper Drumgray (i.) |
|---|---|---|---|---|---|---|---|---|
| Specific gravities | 1·268 | 1·261 | 1·292 | 1·290 | 1·286 | 1·322 | 1·275 | 1·308 |
| Calorific power (Thompson's calorimeter) | 7,480 | 7,590 | 7,425 | 7,370 | 7,480 | 6,957 | 6,930 | 7,507 |

| — | I. Upper Drumgray (ii.) | J. Lower Drumgray (i.) | K. Lower Drumgray (ii.) | L. Lower Drumgray (iii.) (Slamannan). | M. Kilsyth Haughrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| Specific gravities | 1·305 | 1·279 | 1·289 | 1·322 | 1·291 | 1·306 | 1·275 |
| Calorific power (Thompson's calorimeter) | 7,370 | 7,177 | 7,508 | 7,810 | 7,535 | 7,865 | 7,810 |

In the paper to which reference has already been made (W. Carrick Anderson, Trans. Phil. Soc. of Glasgow, 1897–98), it was pointed out, that, of the coals indicated in the accompanying section, those situated above the position of the Airdrie Blackband Ironstone, although on ignition in the crucible they give residues of fixed carbon that cohere together, are non-coking coals in the sense that the residues they yield are soft, so that they are valueless for the purpose of manufacturing cokes suitable for furnace purposes. Those lying between the horizon of the Airdrie Blackband and that of the Slatyband Ironstone are frequently affected by the more or less close proximity of intrusive volcanic rocks and their qualities thereby changed. This is particularly noticeable in the Drumgray coals, and for this reason we have included in the above tables two samples of Upper and three of Lower Drumgray somewhat different in composition. From the Virtuewell coal, an inferior coke is occasionally manufactured, and both the Upper and Lower Drumgray are, in certain localities, valuable coking coals. Where the intrusive volcanic rocks have come into intimate contact with these seams, they have been converted into semi-anthracites (sample L in foregoing tables), and in this condition constitute valuable steam coals. The lowest or limestone series (below the position of the index limestone) contains a set of coals the chief characteristic of which is their strong caking property. The Possil Main, Kilsyth Haughrigg, Bannockburn Main, Kinneil and Wilsontown Main and Kilsyth Coking seams, which are developed in different localities on the northern edge of the basin, are all worked for the manufacture of coke. The products, while variable in quality, are as a rule hard more or less silvery cokes with a strong metallic ring, for which there is an active demand for foundry purposes throughout the district.

It would appear from this, that, whatever may be the points in which the coals of this field resemble and differ from one another, there is an increasing development of the caking property as we pass downward in the series, and this is confirmed by the following experiments, in which the caking power is measured by the number of units of fine white sand that can be caused to unite into a cake that just coheres by ignition in a closed platinum crucible with unit weight (1 grm.) of the finely powdered coal. This number is the caking index :—

TABLE V.

*Caking Indices of Coal.*

| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. | H. Upper Drumgray (i.) |
|---|---|---|---|---|---|---|---|---|
| Caking Index | 4 + | 4 | 3·5 | 3·5 − | 4 | 4 | 4 | 4 − |

| — | I. Upper Drumgray (ii.) | J. Lower Drumgray (i.) | K. Lower Drumgray (ii.) | L. Lower Drumgray (iii.) (Slamannan). | M. Kilsyth Haughrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| Caking Index | 6·5 | 4·5 + | 6·5 + | 0 | 9 + | 15·5 | 10 + |

Measured by this convenient, if somewhat rough, test, it appears that, until we come down to the Drumgray coals, the caking capacity shows little difference in degree; but in the Drumgrays, and especially those indicated as "I" and "K" there is noticeable a marked advance towards the strongly caking quality characteristic of the limestone series, represented by the last three samples.

The reasons for this marked difference in property between substances which show comparatively little variation in ultimate analysis were considered at some length in the previous paper, and it was shown by experiment that in the non-coking coals of the uppermost seams the formation of coherent residues on ignition was due entirely to the presence in them of bodies extractable with caustic potash. The result was that, after boiling for 1—2 hours with 5 per cent. solution of the alkali, all of these coals lost the tendency to cohere on ignition which they formerly possessed, and gave only loose dull earthy powders. The strongly caking coals from the limestone series, on the other hand, continued, even after three hours boiling with alkali, to cohere firmly on ignition, forming true cokes, which were hard, although not so bright as those from the original coals. In the intermediate series the state of things forms a connecting link between these two extremes. The Virtuewell coal after boiling with caustic potash gave a residue that cohered slightly on ignition, forming a brittle soft coke. Kiltongue yielded only a loose dull powder, similar to those of the uppermost series. Upper Drumgray "H" just cohered, while Upper Drumgray "I" gave a dull but fairly strong coherent coke. The tendency becomes more pronounced in the Lower Drumgray coals, of which that marked "J" yielded a residue that cohered fairly well, but was dull and earthy in appearance, while "K" gave a hard, strong, and fairly bright coke, differing very little from that of Kilsyth Haughrigg. In these intermediate coals therefore, except Kiltongue, there is present to a greater or lesser extent, in addition to resinous bodies, a non-saponifiable constituent capable of inducing a certain amount of coking in the oven, although not to the extent that is characteristic of the coals of the lowest series; but at their best they are true caking coals.

The question therefore arises, what is it that conditions this difference in the degree of coking power? Is the binding constituent the same in each of the true caking coals, and is the difference in degree of caking, which they exhibit, due entirely to the relative amount present in each case, or are the binding constituents different?

To gain some light on this point we heated samples of each of these coals, coking and non-coking, to a temperature of 300°—305° C. in a current of dry carbon dioxide for three hours, and afterwards tested the coking quality by ignition in a closed platinum crucible. The results are shown in the following table:—

Table VI.

*Losses at* 300° *C.*

| — | A. Ell. | B. Main. | C. Splint. | D. Gas. | E. Virgin. | F. Virtuewell. | G. Kiltongue. |
|---|---|---|---|---|---|---|---|
| Percentage loss of dry coal at 300° C. | 9·5 | 16·2 | 16·2 | 21·8 | 17·7 | 16·2 | 11·9 |
| Physical condition of residue. | A considerable part formed a dull earthy coke, the rest in powder. | Residues just cohered and not more. | | | | | |

| — | H. Upper Drumgray (i.) | I. Upper Drumgray (ii.) | J. Lower Drumgray (i.) | K. Lower Drumgray (ii.) | M. Kilsyth Haughrigg. | N. Bannockburn Main. | O. Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| Percentage loss of dry coal at 300° C. | 14·7 | 8·1 | 12·6 | 7·9 | 13·1 | 7·3 | 6·4 |
| Physical condition of residue. | Residue cohered slightly, but more than the preceding. | Residue coked and fairly hard in the lower part of the crucible. | Residue cohered, but no more. | Residues, dull but fairly strong hard cokes. | | Dull, but good hard strong cokes. | |

Comparing these results with those got from the experiments with caustic potash, we are led to the following conclusions with regard to the whole series:—

(1.) In the Main, Splint, Gas, Virgin, and Kiltongue coals the cohesion of the residues on ignition is due entirely to the presence of small quantities of resinous matter, which can be expelled completely on slowly heating the powdered coals to 300° C. The breakdown of these resins on rapid ignition causes the binding together of the residues.

(2.) In Ell the tendency towards cohesion is due to the same cause, but a small quantity of the resinous matter remains in the coal at 300° C.

(3.) Virtuewell owes its tendency to cohere, partly to the presence of a constituent or series of constituents not extractable with caustic potash, but completely volatile on heating for three hours at 300° C.

(4.) In both samples of the Upper and Lower Drumgray (i. and ii.) the caking property proceeds partly from the resinous constituents and partly from non-resinous substances; in the coals marked H and J, chiefly from the former, and more so in Upper than in Lower; in those marked I and K, principally from the latter; and in K almost entirely so. In H and J the caking constituents can be almost completely volatilised at 300° C. in absence of air. The coals I and K form hard bright cokes on ignition, even after exposure to a temperature of 300°—305° C. for three hours, in absence of air.

(5.) In the strongly caking coals of the limestone series the firmly coherent character of the ignited residues is the result almost exclusively of the breakdown of non-saponifiable bodies. In Kilsyth Haughrigg, a larger proportion of these is volatile at 300° than is the case in Bannockburn Main and Kilsyth Coking coals, which suffered little deterioration in coking property by exposure for three hours to that temperature.

Two general remarks we would make upon these conclusions. The first, with reference to the statement that the resinous constituents exercise no influence upon the coking of a coal, is that, while they do not condition its suitability in most cases for coking, they distinctly contribute to the caking property. The second bears upon the statements of Marsilly and Percy (Annales de Mines S. 5, 1857, **12**, 347, *et seq.*; Percy, "Metallurgy," **1**, "Fuel," p. 309), that heating for from one or two hours at 300—304 C. destroys the caking property through volatilisation of constituents. We find that in certain coals of the Clyde series it does do so, but that in the strongly caking coals from the lowest strata mere heating to 305°, even for three hours (air being excluded), does not materially affect their tendency to cohere firmly on ignition.

The next point we had to determine, if possible, was the nature of the substance that causes the fusibility of the latter class of coals, and so renders them valuable for the manufacture of coke. The action of caustic potash solution afforded a criterion by which we were able to differentiate between a coking and a non-coking coal; and it may be added that we have proved the validity of the test in respect to both classes of mineral from other localities as well. But it does not enable us to say, whether the character of caking or not-caking depends upon the constitution of the whole body, or whether it depends upon the nature of a constituent or series of constituents forming merely a part of the total bulk of the coal.

In the attempt to ascertain this, recourse was had in the first instance to atmospheric oxidation. The results obtained for both descriptions are described at length in the paper, containing the first instalment of these researches, already referred to. It is sufficient here to say that, while of very great interest, they did not appear to offer any means of arriving at a solution of the question. We then turned our attention to the effect of nitric acid oxidation.

In 1891–92 it was pointed out by R. J. Friswell (Proc. Chem. Soc. 1891–92, 9), that the effect of dilute nitric acid upon coals was to convert them into substances which, on drying, were black, lustrous and very brittle, insoluble in water, but very easily soluble in ammonia and other alkaline fluids. From the intensely dark coloured solutions thus obtained, acids precipitate bulky brown flocculent substances, which are insoluble in solutions of acids and mineral salts, but are soluble to some extent in pure water.

So far as we have been able to learn, no one has up to the present time attempted to investigate the character of these bodies, or to ascertain how far the substances obtained in this way from different coals agree in composition. In the present case it seemed to us a matter of importance to discover, first of all, the qualitative composition of the bodies, and, secondly, whether those obtained from the different classes of coal were the same or different. The method of procedure adopted in their preparation is described in the previous paper and need not be repeated. Qualitatively, they were found to consist in each case of carbon, hydrogen, oxygen, nitrogen and sulphur, with a small quantity of mineral matter, principally oxide of iron, amounting as a rule to from 0·5 to 1 per cent.

Provided excess of acid was used for the oxidation of the coal, it appeared to be immaterial whether the excess was much or little, so far as the ultimate composition of the purified residue was concerned, and from a given coal the oxidized body, or "coal acid" as we shall call it, was always one and the same. This may be illustrated by the following results obtained from the combustion of two samples of the acid from Bannockburn Main coal, prepared on different occasions.

TABLE VII.

| —— | Sample "A." | Sample "B." |
|---|---|---|
| | Per Cent. | Per Cent. |
| Hydrogen | 3·33 | 3·31 |
| Carbon | 62·61 | 62·45 |
| Oxygen | 28·55 | 28·50 |
| Nitrogen | 4·41 | 4·45 |
| Sulphur | 0·44 | 0·44 |
| Ash | 0·63 | 0·85 |
| | 100·00 | 100·00 |

The coals which we examined went into solution almost completely when evaporated with nitric acid and subsequently treated with ammonia. The small residue left was investigated in one instance and found to consist largely of mineral matter (36 per cent.) surrounding particles of a compound similar in composition to that of the coal acid prepared, and probably only prevented from going into solution by being intimately mixed with the ash residue.

Having proved that in these coal acids we had to deal with bodies, which, whether chemical individuals or not, were at least of definite composition, we proceeded to prepare specimens from typical coals of the caking and non-caking type, and to subject them to careful analysis. The following table shows the figures obtained:—

TABLE VIII.

*Composition of Acid Bodies got by Oxidation of Coal.*

| —— | Ell. | Splint. | Gas. | Virgin. | Lower Dromgray (Slamannan). | Bannockburn Main. | Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 3·15 | 3·20 | 3·17 | 2·74 | 2·96 | 3·33 | 3·16 |
| Carbon | 58·45 | 62·31 | 61·35 | 58·97 | 63·35 | 62·61 | 61·32 |
| Oxygen | 32·36 | 28·04 | 29·16 | 32·67 | 28·00 | 28·55 | 30·80 |
| Nitrogen | 4·79 | 4·79 | 4·71 | 4·00 | 4·27 | 4·42 | 3·30 |
| Sulphur | 0·72 | 0·31 | 0·68 | 0·27 | 0·51 | 0·43 | 0·73 |
| Ash | 0·53 | 1·35 | 0·93 | 1·35 | 0·91 | 0·63 | 0·69 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

TABLE IX.

*Composition of Organic Matter in above Acids.*

| —— | Ell. | Splint. | Gas. | Virgin. | Lower Dromgray (Slamannan). | Bannockburn Main. | Kilsyth Coking. |
|---|---|---|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Hydrogen | 3·17 | 3·24 | 3·20 | 2·78 | 2·99 | 3·35 | 3·18 |
| Carbon | 58·76 | 63·17 | 61·93 | 59·78 | 63·93 | 63·05 | 61·75 |
| Oxygen | 32·53 | 28·42 | 29·43 | 33·11 | 28·25 | 28·72 | 31·01 |
| Nitrogen | 4·82 | 4·85 | 4·75 | 4·05 | 4·31 | 4·45 | 3·32 |
| Sulphur | 0·72 | 0·32 | 0·69 | 0·28 | 0·52 | 0·43 | 0·74 |
| | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 | 100·00 |

These results, so far as they go, are of very high interest. Incidentally, we may observe, that they supply direct proof of the presence of "organic sulphur" in coals, the existence of which had been asserted for some time on circumstantial evidence alone. They show, moreover, that, whatever may be the cause of the chemical and physical differences between these caking and non-caking coals, they yield on oxidation bodies which, although different from one another, show no distinct division corresponding to the coals themselves. The acids obtained from the soft non-caking coals (Ell and Virgin) certainly contain more oxygen than those from Bannockburn Main and Kilsyth Coking, but, on the other hand, the latter are almost identical with those from Splint and Gas coals, which are non-caking.

There is then the further question, do these acid bodies owe their origin to the oxidation of the organic matter of the coal as a whole, or is each the product of a constituent merely? In the first case, coals, whether coking or non-coking, will be made up, if we neglect resins and ash, entirely of bodies containing—in addition to hydrogen, oxygen, and carbon—both nitrogen and sulphur; in the second case, of a mixture of such constituents with others of different composition.

In every case, heating coal to 300°—310° C. causes a considerable loss in weight, and part of the organic matter is gasified in addition to that which distils over. If this gasification is due to the decomposition of a complex molecule containing nitrogen and sulphur and constituting the bulk of the coal, then oxidation with nitric acid subsequent to the heating might be expected to yield an acid of different composition from that previously obtained. Conversely, should the product of oxidation be the same as before, it might be argued that the gasification proceeded from the decomposition of another constituent existing over and above the nitrogenous one which yields the acid.

To ascertain which took place, we heated Ell coal for four hours in a current of dry carbon dioxide at a temperature of 300°—315° C. and from the residue prepared an acid by oxidation as before. The dry Ell coal lost about 9·5 per cent. by weight on heating, and the change in composition caused thereby is indicated by the following comparison.

Table X.

| A.—Composition of Organic Matter in Ell Coal. | | B.—Composition of Residue left on heating Ell to 300°—315° C. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Hydrogen | 5·07 | 5·32 |
| Carbon | 80·74 | 82·67 |
| Oxygen and org. sulphur | 12·47 | 10·00 |
| Nitrogen | 1·72 | 2·01 |
| | 100·00 | 100·00 |

Five grms. of this residue was converted into the acid by oxidation with nitric acid (1·2), and this was purified by the same methods as before. On analysis it gave the following result, which we compare with the figures representing the original Ell acid.

Table XI.

| Acid from Original Ell Coal. | | Acid from Ell Residue. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Hydrogen | 3·15 | 3·01 |
| Carbon | 58·45 | 58·57 |
| Oxygen | 32·36 | 32·28 |
| Sulphur | 0·72 | 0·71 |
| Nitrogen | 4·79 | 4·60 |
| Ash | 0·53 | 0·83 |
| | 100·00 | 100·00 |

The bodies are thus clearly identical. The inference we draw from this result is that the nitrogenous molecule in Ell coal remains intact even when the coal is subjected to a temperature of 300°—315° C. for a lengthened period of time, and that the loss in weight, amounting to 9·5 per cent., and only to a small extent resulting from the volatilisation of resinous substances, is due chiefly to the (volatilisation and) breakdown of another constituent or series of constituents.

From the very close resemblance, in some cases (Splint and Bannockburn Main) approaching identity, between the acid bodies got from caking and non-caking coals, we incline to the belief that the cause of the difference in the coals themselves is not to be found in the nitrogenous constituents that give rise to these, but in the apparently nitrogen-free bodies associated with them. The greater or less ease with which these are volatilised and decomposed in different coals would thus condition their non-caking or caking character. In Ell coal they do not melt, but break up at once into gaseous bodies. In Virtuewell and the Drumgray H and J coals they melt on rapid heating, and cause partial fusion, so that it is possible to manufacture weak cokes from these. The increased caking power of the latter coals does not come out in the table of "caking indices," because the admixture of sand, which has a low conductivity for heat, causes practically the whole of this potentially fusible constituent to be volatilised before it melts, and the "caking index" as given is thus, as in the case of the preceding coals, merely that of the resinous substances contained in them. Bannockburn Main coal, we have ascertained, melts at about 317° C., and, the fusible constituent being decomposed at a still higher temperature, deposits much carbon which acts as a cementing material. This large deposition of carbon it is that occasions the phenomenon, often noted and commented on, that the percentage weight of fixed carbon got from caking coals is greater than that obtained from those that do not cake.

The view as to the composition of coal, which we have been thus led to adopt, as the result of our experimental investigations, differs materially from the theory already mentioned as having been formulated many years ago by Baltzer. We conclude that, whether or not different coals contain bodies that form a genetic or homologous series, such bodies form, at the most, only a part, and, it may be, in many cases a minor part of the total organic matter of the minerals; further, that a considerable part of the organic matter consists of a complex compound comparatively rich in nitrogen, and containing sulphur as well; and that, over and above these, resinous material is always present to a small but fairly constant extent.

The composition of the mixture of resinous and other bodies, which can be driven off by heating a coal to 300° C., cannot, as is evident from the figures in Table X., differ very widely from that of the residue left. Several of these resins, or mixtures of them, have been examined and their ultimate composition is not unlike that of ordinary pine resin, *i.e.*, say C = 80·7 per cent.; H = 9·5 per cent.; O = 9·8 per cent. (Muck, "Die Chemie der Steinkohle," 2nd Edition, 1891, pp. 66, 67.)

The nitrogenous bodies obviously owe their origin to the proteid substances of the vegetable matter from which the coal was formed. Indeed the very discrepancies, which they show in ultimate composition, may be regarded as confirmation of their being derived from some such source, for the proteids themselves show like differences on analysis.

The limits of variation in the class have been set down as these:—

Carbon, from 51·5 per cent. to 54·5 per cent.
Hydrogen, from 6·9 per cent. to 7·3 per cent.
Nitrogen, from 15·2 per cent. to 17·0 per cent.
Sulphur, from 0·3 per cent. to 2·0 per cent.
Oxygen, from 20·9 per cent. to 23·5 per cent.

The remaining constituent, or constituents, would then represent in each case degradation products of the original carbohydrates of the coal plants. Whether these constituents will be partially fusible or not, may possibly be determined to some extent by the nature of the original carbohydrates, which have undergone degradation. We know that the isomeric bodies, starch, cellulose and gum, when ignited in a closed crucible, give fixed residues that are quite different in appearance, and it is at least conceivable that the products of their degradation, even assuming the process to have gone on to an equal extent in each case. might show a like difference in behaviour. But it is equally possible, and perhaps on the whole more probable, that the fusible or non-fusible character of the constituents, and consequently of the coals themselves, is determined, ultimately at least, by the degree to which, under varying geological conditions, the degradation process has advanced.

### Discussion.

Mr. C. A. Fawsitt said that he was sorry that he was not qualified to speak of coal as regards its capability of coking, but he was sure the members of the Society were under a debt of gratitude to the authors for having brought the subject before them. He had often thought it astonishing that the general public, and even manufacturers, knew so little about coal. It was still more surprising to him that dealers in coal had so little knowledge of its composition, and he thought that the time was approaching when an analysis, along with the calorific value of a coal, would be submitted by the coalmaster to the intending purchaser. In the case of a well-known coal the name was a sufficient guarantee of its quality, but beyond this the users of coal had very little to guide them. If the calorific value were given in the case of an unknown coal, one would have very little hesitation in trying it. It seemed to him that there was a great waste in their methods of using coal. As Mr. Anderson had stated, the atmosphere was polluted by black smoke, against which letters were continually appearing in the newspapers. The manufacturer, however, knew his own business as well as any one, and was not likely to throw away coal if he could devise any proper method of saving it. He thought that the question of organic sulphur in coal was of very great importance. Whether it was the organic sulphur that in distillation combined to form sulphocyanide, he was not in a position to state, but he knew that the production of this by-product, in which nitrogen and sulphur were recovered as sulphocyanide, in the distillation of coal in the gasworks was of great importance, and had a marked effect in increasing the supply and in reducing the price of sulphocyanide. If some means could be adopted of increasing the amount of ferrocyanide it would add very much to the value of coal. He understood Mr. Anderson to state that in blast furnaces the total nitrogen was utilised, and he would like to ask him if that was correct. If it was not, then a large proportion would escape through the ammonia condensing apparatus, and thus be lost.

Mr. D. J. Playfair said he thought it would be interesting to know if the products of the distillation of the "coal acids" resembled those of the original coal.

A Gentleman present said that, as he was perhaps the only one there connected with blast furnaces, he would like to answer the question which Mr. Fawsitt had raised. He thought that in the ironworks at present from one-fourth to one-fifth of the nitrogen in the coal was recovered. The quantity of ammonia lost through the condensing apparatus did not amount to 0·5 per cent. The remainder was not given off as free nitrogen or in the form of nitrogen compounds, which at present could not be converted into ammonia. The question of the form in which the nitrogen was combined was one of very great practical importance. He did not know if Mr. Anderson had given much attention to the matter and was not aware of any work done on the subject, but, from its behaviour in the process of destructive distillation, it must occur in more forms than one. In some places in the Midland counties apparatus had been erected on the same principles as those used in the Scottish iron works. In the latter all the ammonia that was available came off as free ammonia and lime treatment was not necessary. In the case of the former he did not think that the process was a success at all.

Mr. Howden said that he had made, some years ago, a considerable number of experiments on the evaporating values of different kinds of coal, and might therefore comment on Mr. Fawsitt's suggestion as to the advisibility of submitting analyses of coal. He thought that analyses were rather deceptive on the whole, unless they referred to coals of the same kind, as he had found some coals containing very high percentages of carbon to give very low evaporating values. One of these coals, which approximated to a pure anthracite and was very rich in carbon, would not burn at all; steam could not be raised after trying it with a hot fire and close combustion.

Mr. James McLeod said he would like to ask Mr. Anderson what effect long exposure to atmospheric influences had upon coal with regard to its employment for gas making. He had occasion to test some Cairntable Boghead coal that had lain in the Glasgow Gas Works for 30 years. It was a coal only inferior to the Torbanehill mineral, and yielded the other day about 14,000 cubic feet of 40-candle power gas per ton, showing that its deterioration had been trifling.

The Chairman feared that most of the members present felt, like himself, unable to discuss the paper fully at a first hearing. He might, however, be permitted to say that in his opinion the authors had attacked the problem from the right side, if only with the object of ascertaining the differences in composition between various kinds of coal. Another method had been to study the products of the destructive distillation of coals, but this seemed to him a method which must fail to throw much light upon the question of the ultimate composition of coals, if only because the nature of the distillation products depended so much upon external conditions. The authors had apparently established at least one reason for the difference between a coking and a non-coking coal, and he hoped they would continue their investigations.

Mr. Anderson in reply said that there were one or two points in the discussion to which he would like to refer. With regard to Mr. Fawsitt's question as to the nitrogen recovery in blast furnaces, that was based on a misapprehension. In the paper he had read last year, he had quoted the statement of Knublauch (this Journal, 1896, 106, 107) to the effect that, in distilling Westphalian coal, only 12—14 per cent. of the nitrogen present was recovered as ammonia, and not quite 2 per cent. as hydrocyanic acid. A gentleman, who had spoken, stated that in the blast furnaces they counted on a recovery of $\frac{1}{4}$th or $\frac{1}{5}$th of the nitrogen as ammonia; he thought that was a liberal estimate. Taking these figures as correct, they meant that only a small proportion of the available nitrogen was at present being recovered in marketable form. They had done a great deal in Scotland in connection with the recovery of residuals, but he thought that processes which succeeded in obtaining less than 25 per cent. of the nitrogen in the coal, while they could not be called failures, at least left very much to be desired, and were capable of considerable improvement. With regard to Mr. Playfair's question as to the products of distillation, that was a point which they had not yet investigated, and one which they would have to attack separately. A question had been asked as to the manner in which the nitrogen was combined. In the distillation of coal there were at least two forms of combined nitrogen. In all coals a certain amount came off as ammonia and as cyanide; but a remarkable fact was that in coal minerals from any part of the world there was a certain percentage of nitrogen that always remained behind in the coke. In the statement he quoted previously, 50 per cent. was given as the average quantity remaining in the coke. He had found a like percentage in coke made from Scotch coals. Different figures were on record but he thought that all pointed to something definite in the condition of the combined nitrogen. He quite agreed with Mr. Howden as to the limited value of the ordinary coal analysis. A chemist only gave a proximate analysis, which was a very fair index of the yield of coke to be expected from it, though even this would vary according as it was treated in one type of oven or another. It would also show the content of moisture and ash, but beyond that its value was comparatively slight as far as affording an indication of its suitability for steam raising was concerned. The effect which long exposure to the atmosphere had upon coals was an interesting subject by itself. It was investigated first about 1870 by Prof. Richters, who ascertained that coal absorbed a certain amount of oxygen when heated in air, and, curiously enough, when it took up oxygen in that form the percentage yield of fixed residue rose very extraordinarily. He had not experimented on Cairntable coal, but he knew that some of the gas coals resisted atmospheric action for a long time. This might be said of the splint and gas coal from Hamilton, which were extremely difficult to attack by the atmosphere. He entirely agreed with what Prof. Henderson had said with regard to the study of the constitution of coal. He would only further add that, if any of those present were in the habit of coming across coals of different kinds, and were confronted with knotty points or apparent freaks of nature in the form of coal, he would be glad if they would communicate with him at the University, in order that he might continue his investigations upon the subject. While working in the laboratory one was sometimes apt to get out of touch with the practical problems, and he would thank any gentleman, who had such a problem, if he would let him know the difficulty. All such evidence had its value in the search after a general law.

## Yorkshire Section.

*Meeting held on Monday, January 31st, 1898.*

MR. THOS. FAIRLEY IN THE CHAIR.

### THE REFRACTIVE CONSTANT IN OIL AND FAT ANALYSIS.

BY PROF. H. R. PROCTER, F.I.C.

(*Contribution from the Leather Industries Laboratory of the Yorkshire College, Leeds.*)

The employment of the refractive index of oils and fats as one of the physical constants available for their recognition is of comparatively recent date. According to Benedikt, Leone and Longi were the first to use it in the detection of the adulteration of olive oil with sesame and cotton-seed; and in 1889 Strohmer determined it with considerable accuracy for a number of oils by means of the Abbe refractometer, and found that olive oil had the lowest, and castor and the drying oils the highest, indices. He concluded, however, that it was not of much value for analytical purposes. About the same date, Amagat and Jean (Comptes Rend. **109**, 116) described their oleorefractometer, which permitted of the very delicate comparison of the refractivity of an oil with that of a second sample, or of an "*huile type*," but possessed only an arbitrary scale, and did not permit of the determination of an absolute refractive index. They showed that in many cases its indications were of much value in affording *primâ facie* evidence of adulteration in olive oils and butters; but the value of the large number of determinations which have been made with the instrument are greatly diminished by the fact that they cannot be compared with those of any other instrument; and even the composition of the "*huile type*" has not been published, though it is understood to be sheepsfoot oil. It should, however, be by no means impossible to construct an empirical table by which its indications could be reduced to refractive index.

More recently, considerable attention has been paid in Germany to the use of refractivity in detecting adulteration in butter and lard (Hefelmann, "Pharmaceutischen Centralhalle," 1894, Nos. 33 and 35; Zeiss, *ibid.* No. 31; Mansfield, "Forschungsberichte ueber Lebensmittel," Vol. I., Heft 3), and a simple and convenient form of the Abbe refractometer is now made for the purpose by Zeiss, which is provided with a water-jacket to the prisms, so that determinations can be made at various temperatures. The instrument, like the Abbe refractometer in its original form, depends on "total reflection," and the optical part consists essentially of two prisms of acute angle, and highly refracting glass, which, when placed together, form a solid block of rectangular section. The angle of the prisms is such that, so long as they are separated by a film of air, the whole of the light passing in at one end of the block is totally reflected from the oblique face, none passing into the second prism, or, consequently, to the eye of the observer. If, however, a film of oil be introduced between them, all light falling on the reflective surface at less than a certain angle can pass through, and on looking at the prism through a suitable eyepiece, the field of view will be divided into a light and a dark portion, the position of the limiting line being dependent on the relation between the refractivity of the oil and that of the prism. This position is read on a graduated scale fixed immediately above the prism, and from it the index of refraction may be obtained by reference to a table supplied with the instrument, or it may be simply recorded in the arbitrary scale-divisions, which range from 0 to 100. Since light of different colour and wave-length differs in its refrangibility, and, consequently, in the angle at which, under given conditions, it would be totally reflected, the edge of the light field would, naturally, be bounded by a coloured fringe which would render accurate reading difficult, and this has been provided for in the case of butters by a special achromatisation of the prisms, which renders the line sharp and colourless. With fats which differ materially from butter in dispersive power, coloured fringes will still appear, which are blue when the dispersion is higher, and red when it is lower, than that of butter, and which are frequently useful in indicating a mixture of other fats, even when the refraction is identical with that of butter. In such cases it is often necessary to employ sodium light in order to obtain sharp readings, when the refraction measured will be that of the sodium or "D" line. An orange glass fitted into grooves provided below the lower prism may also be substituted for the sodium light with convenience, but some loss of accuracy. For adjusting the instrument in case of accidental shifting of the zero, a "normal fluid" is supplied which gives a colourless margin, and of which the refractivity at various temperatures has been accurately determined by the makers. The lower prism is hinged, so that it can be turned back to permit of the application of the film of oil, which should be spread on the face with a camel's hair brush, or a slip of soft wood, in order to avoid risk of scratching the somewhat soft glass. The temperature of the instrument is regulated by connecting the water-jacket which surrounds both prisms with a supply of water of suitable temperature. (It is important that the water should flow in at the lower nozzle, as directed in the instructions, or irregular heating of the prisms, and inexact measurements, will result.) The thermometer which is furnished with the instrument usually reads only to 50°, which is insufficient for the fatty acids and harder fats, and as, with care, the instrument may safely be raised to 75°, it is desirable to obtain a thermometer graduated to this temperature. Of course, in working at high temperatures, care must be taken to avoid sudden changes, which might crack or loosen the prisms. The instrument is so convenient in use, that it is much easier to take a refractive index of an oil or fat with it than to determine the specific gravity with the pycnometer, and it has the further advantage that two or three drops are sufficient for the purpose. It has been used in all the work described in this paper, but, of course, the larger Abbe refractometer, if fitted with a water-jacket, is equally applicable, and has the advantage of giving the refractive index and dispersion by direct reading. One or two new instruments have been recently introduced, which the writer has not yet seen, but which appear to deserve mention, which do not depend on the total reflection, but on the deviation of the light, as in the case of the instrument of Amagat and Jean. Instead, however, of using an oil as a standard fluid, water or glass prisms are employed, to approximately compensate the refraction of the oil, and the scales are so constructed as to give the refractive index. One of these is made by Dubosq, another by Ph. Pellin, of Paris.

On comparing tables of the specific gravities and refractive indices of oils and fats, it will be noted that they are to a considerable extent proportional to each other and that both increase as the temperature is raised. It is obvious that, if this proportionality were strictly constant, no information could be obtained from the one which might not equally be deduced from the other. There is, therefore, no advantage in determining both constants, unless the two are so combined as to bring out the deviations from strict proportionality, which in a mere rough comparison are quite masked. Another consideration, which points to the desirability of the calculation of such a mutually dependent constant, is that both density and refractive index decrease with rise of temperature, and that both must be determined at a fixed and uniform temperature to allow of any general comparison. As many fats are solid or turbid at 15°, a higher temperature must often be used. For specific gravity that of the boiling-point of water has been largely adopted, but it is too high to be conveniently used for refraction determinations, and for butter and lard 40° has been generally adopted, which, in its turn, is too low for tallows and for the saturated fatty acids. As both density and refractive index decrease *pari passu* with a rise of temperature, it is probable that a refraction constant from which the effects of alteration in density were eliminated would also be independent of temperature.

The first attempt to find such an expression was made by Newton, based on theoretical deductions from his emission theory of light, but, though it showed good agreement with observation in the cases which he investigated, it failed to do so on more extended experiment, and since it has also been deprived, by the general acceptance of the undulatory theory, of its theoretical basis, it has been entirely abandoned. In 1858 the subject was again closely investigated by Gladstone and Dale, who proposed a new formula, based on the results of very extensive experiment, and which still agrees with the results of later investigation. They offered no theoretical explanation of their expression, though some subsequent attempts have been made to do so. (See Preston, "Theory of Light," 2nd Edition, p. 132.) Their formula is a very simple one. Where $n$ is the refractive index and $d$ the density, the Gladstone and Dale constant $R_G = \frac{n-1}{d}$, or, in other words, they merely divide the refractive index, less 1, by the specific gravity.

More recently a fresh formula, based on theoretical considerations, was proposed almost simultaneously by two independent investigators, L. Lorenz and H. Lorentz, and as it agreed well with experiment, it has been used in most of the more recent work. Adopting the same notation as before, their constant $R_L = \frac{n^2-1}{(n^2+2)d}$. In certain extreme cases, such as the comparison of the refractivity of a vapour with that of the same substance in the liquid form, it gives wonderfully accurate results, where the older formula is widely at fault, but in others, where the range is small, and especially in the case of mixtures of liquids, the latter is frequently the closer of the two, and it is much simpler in calculation and more adopted for logarithmic reckoning. In the special cases which are dealt with in this paper either formula gives good results, and both are calculated in the table of the refractions of oils and fats which is subsequently given; but, on the whole, that of Lorenz and Lorentz has been found most accurate, and it is also more easily compared with the theoretical results deduced from chemical constitution.

The two formulæ, though approximately proportional to each other over a small range, differ pretty widely in extreme cases, and we cannot, therefore, pass from one to the other by simple multiplication by a constant factor. The ratio between the two is algebraically $\frac{R_L}{R_G} = \frac{n+1}{n^2+2}$, and therefore depends on the index of refraction only. Its numerical value at $n = 1·40$ is 0·6060, and at $n = 1·50$ is 0·5882, and these include the values of all fats and oils. When $n = 1·4345$, or at about 16° of the scale of the "butter refractometer," the ratio is exactly 0·6, and this value may be used in most cases for rough comparison between the two. The following tables, giving the refractive constants by both formulæ for oleic acid and for a sample of olive oil, will give some idea of the relative constancy to be expected. The oleic acid, though obtained from Kahlbaum specially for the purpose, was not quite pure, and at a temperature somewhat above the solidifying point of oleic acid showed some crystallisation of palmitic or stearic acid, which was filtered off before making the determinations.

### TABLE I.

*Oleic Acid (Kahlbaum).*

| Temperature. | Sp. Gr. | Refractive Index. | Refractive Constants, $\frac{n-1}{d}$ | Refractive Constants, $\frac{n^2-1}{(n^2+2)d}$ |
|---|---|---|---|---|
| ° C. | | | | |
| 15 | 0·898 | 1·4638 | 0·5165 | 0·3072 |
| 20 | 0·895 | 1·4620 | 0·5159 | 0·3070 |
| 25 | 0·893 | 1·4603 | 0·5154 | 0·3069 |
| 30 | 0·889 | 1·4585 | 0·5158 | 0·3072 |
| 35 | 0·885 | 1·4566 | 0·5160 | 0·3075 |
| 40 | 0·882 | 1·4546 | 0·5154 | 0·3074 |
| 45 | 0·880 | 1·4528 | 0·5145 | 0·3071 |
| 50 | 0·875 | 1·4509 | 0·5153 | 0·3077 |
| 60 | 0·870 | 1·4471 | 0·5140 | 0·3072 |
| | | | | Average = 0·3072 |

### TABLE II.

*Olive Oil (H. B. and H.).*

| Temperature. | Sp. Gr. | Refractive Index. | Refractive Constants, $\frac{n-1}{d}$ | Refractive Constants, $\frac{n^2-1}{(n^2+2)d}$ |
|---|---|---|---|---|
| ° C. | | | | |
| 15 | 0·9201 | 1·4718 | 0·5127 | 0·3042 |
| 20 | 0·9150 | 1·4691 | 0·5127 | 0·3044 |
| 30 | 0·9084 | 1·4657 | 0·5126 | 0·3047 |
| 40 | 0·9019 | 1·4619 | 0·5121 | 0·3048 |
| 50 | 0·8961 | 1·4584 | 0·5115 | 0·3047 |
| 60 | 0·8894 | 1·4543 | 0·5108 | 0·3047 |
| | | | | Average = 0·3046 |

It will be noted that in the above tables the constancy of the Lorenz constant at different temperatures is decidedly greater than that of the Gladstone, and this is confirmed by the subsequent table of the refractive constants of oils, in which both are determined for the temperatures of 15° and 60° C.; though in individual cases the reverse is the case. The great objection to the Lorenz formula is the difficulty of calculating it; and to overcome this difficulty, Table III. has been computed, which gives the value of $\frac{n^2-1}{n^2+2}$ for all values of $n$ between 1·4 and 1·5. With its aid the Lorenz constant is found by merely taking out the value corresponding to the observed refractive index, and dividing it by the density; so that the labour involved is scarcely greater than that required in the reckoning of that of Gladstone.

### TABLE III.

*For Calculating Lorenz Specific Refractions.*

| $n$. | $\frac{n^2-1}{n^2+2}$ | Diff. for $n = 0·01$. | $n$. | $\frac{n^2-1}{n^2+2}$ | Diff. for $n = 0·01$. |
|---|---|---|---|---|---|
| 1·400 | 0·24242 | | 1·421 | 0·25359 | |
| 1 | 296 | | 2 | 412 | |
| 2 | 350 | | 3 | 464 | |
| 3 | 403 | | 4 | 517 | |
| 4 | 457 | | 5 | 570 | 0·00526 |
| 5 | 510 | 0·00534 | 6 | 622 | |
| 6 | 564 | | 7 | 675 | |
| 7 | 617 | | 8 | 717 | |
| 8 | 671 | | 9 | 780 | |
| 9 | 724 | | 1·430 | 0·25832 | |
| 1·410 | 0·24776 | | 1 | 884 | |
| 1 | 829 | | 2 | 937 | |
| 2 | 882 | | 3 | 989 | |
| 3 | 935 | | 4 | 0·26042 | |
| 4 | 989 | | 5 | 094 | 0·00523 |
| 5 | 0·25042 | 0·00530 | 6 | 146 | |
| 6 | 095 | | 7 | 198 | |
| 7 | 148 | | 8 | 251 | |
| 8 | 201 | | 8 | 303 | |
| 9 | 254 | | 1·440 | 0·26355 | |
| 1·420 | 0·25306 | | 1 | 407 | |

Table III.—*continued.*

| $n$. | $\frac{n^2-1}{n^2+2}$ | Diff. for $n = 0·01$. | $n$. | $\frac{n^2-1}{n^2+2}$ | Diff. for $n = 0·01$. |
|---|---|---|---|---|---|
| 1·442 | 0·26459 | | 1·467 | 0·27747 | |
| 3 | 511 | | 8 | 798 | |
| 4 | 563 | | 9 | 849 | |
| 5 | 615 | 0·00519 | 1·470 | 0·27900 | |
| 6 | 667 | | 1 | 951 | |
| 7 | 719 | | 2 | 0·28002 | |
| 8 | 771 | | 3 | 053 | |
| 9 | 822 | | 4 | 104 | |
| 1·450 | 0·26874 | | 5 | 154 | 0·00508 |
| 1 | 926 | | 6 | 205 | |
| 2 | 977 | | 7 | 256 | |
| 3 | 0·27029 | | 8 | 307 | |
| 4 | 081 | | 9 | 357 | |
| 5 | 132 | 0·00515 | 1·480 | 0·28408 | |
| 6 | 184 | | 1 | 459 | |
| 7 | 235 | | 2 | 509 | |
| 8 | 287 | | 8 | 560 | |
| 9 | 338 | | 4 | 610 | |
| 1·460 | 0·27389 | | 5 | 661 | 0·00504 |
| 1 | 440 | | 6 | 711 | |
| 2 | 492 | | 7 | 761 | |
| 3 | 543 | | 8 | 812 | |
| 4 | 594 | | 9 | 862 | |
| 5 | 645 | 0·00511 | 1·490 | 0·28912 | |
| 6 | 696 | | 1 | 962 | |

Table III.—*continued.*

| $n$. | $\frac{n^2-1}{n^2+2}$ | Diff. for $n = 0·01$. | Add for $n^2$ | Diff. 0·000534 | Diff. 0·000500 |
|---|---|---|---|---|---|
| 1·492 | 0·29012 | | 0·0001 | 0·00005 | 0·00005 |
| 3 | 062 | | 2 | 11 | 10 |
| 4 | 112 | | 3 | 16 | 15 |
| 5 | 162 | 0·00500 | 4 | 21 | 20 |
| 6 | 212 | | 5 | 27 | 25 |
| 7 | 262 | | 6 | 32 | 30 |
| 8 | 312 | | 7 | 37 | 35 |
| 9 | 362 | | 8 | 43 | 40 |
| 1·500 | 0·29412 | | 9 | 48 | 45 |

Tables IV., V., and VI. give the indices of refraction, the specific gravities, and the corresponding Gladstone and Lorenz constants for a number of oils and solid fats, and a few of the fatty acids, as determined in the Leather Industries Laboratory. Where the fat was liquid, both at 15° and 60°, the constants have been determined at both temperatures. The numbers given must in many cases be taken as merely provisional, since the writer has not been able to repeat them on a sufficient number of samples of known authenticity to give them any authoritative value, and he hopes that the subject will be taken up by those analysts who make a specialty of oils and fats, and who have, consequently, the material available for the determination of general constants.

Table IV.

| Name of Oil or Fat. | Sp. Gr. at 15° C. | Refractive Index at 15° C. | Refractive Constants for 15° C. | | Sp. Gr. at 60° C. | Refractive Index at 60° C. | Refractive Constants at 60° C. | |
|---|---|---|---|---|---|---|---|---|
| | | | $R_G = \frac{n-1}{d}$ | $R_L = \frac{n^2-1}{d(n^2+2)}$ | | | $R_G = \frac{n-1}{d}$ | $R_L = \frac{n^2-1}{d(n^2+2)}$ |
| Castor (average, 6 samples) | 0·963 | 1·4803 | 0·4987 | 0·2951 | 0·934 | 1·4647 | 0·4975 | 0·2958 |
| Raw linseed, old | 0·935 | 1·484 | 0·5176 | 0·3060 | 0·8997 | 1·4674 | 0·5195 | 0·3086 |
| „ new | 0·930 | 1·481 | 0·5172 | 0·3060 | 0·9008 | 1·4657 | 0·5170 | 0·3073 |
| „ blown 48 hours at 100° | 0·9481 | 1·4862 | 0·5128 | 0·3029 | 0·9226 | 1·4698 | 0·5091 | 0·3023 |
| Cod liver, Möller's | 0·928 | 1·481 | 0·518 | 0·3068 | 0·8998 | 1·4652 | 0·5171 | 0·3073 |
| „ medicinal (average, 3 samples). | 0·9359 | 1·482 | 0·5154 | 0·3049 | 0·9075 | 1·4659 | 0·5134 | 0·3051 |
| „ medicinal, blown 48 hours. | 0·9812 | 1·4865 | 0·4955 | 0·2915 | 0·9594 | 1·4705 | 0·4904 | 0·2911 |
| Cod oil (brown) | 0·928 | 1·482 | 0·523 | 0·3072 | .. | .. | .. | .. |
| Coast cod (average, 4 samples) | 0·9300 | 1·481 | 0·5172 | 0·3060 | 0·900 | 1·4635 | 0·515 | 0·3063 |
| Commercial cod, 1 | 0·9341 | 1·4831 | 0·5172 | 0·3058 | .. | .. | .. | .. |
| „ „ 2 | 0·9343 | 1·4808 | 0·5146 | 0·3045 | .. | .. | .. | .. |
| Whale (average, 4 samples) | 0·9203 | 1·476 | 0·5162 | 0·3065 | 0·892 | 1·4603 | 0·5158 | 0·3072 |
| Sardine (Jap) | 0·9256 | 1·479 | 0·5175 | 0·3063 | .. | .. | .. | .. |
| Seal (pale) | 0·9249 | 1·4784 | 0·5173 | 0·3063 | 0·8987 | 1·4619 | 0·5139 | 0·3059 |
| Shark liver | 0·917 | 1·4783 | 0·5216 | 0·3089 | 0·8918 | 1·462 | 0·518 | 0·3083 |
| Mixed fish (average, 4 samples) | 0·929 | 1·4804 | 0·5171 | 0·3060 | 0·900 | 1·4635 | 0·515 | 0·3063 |
| Cotton-seed | 0·9247 | 1·475 | 0·5137 | 0·3045 | 0·8943 | 1·4586 | 0·5127 | 0·3055 |
| Arachis (average, 4 samples) | 0·9212 | 1·4731 | 0·5136 | 0·3046 | 0·8899 | 1·4564 | 0·5128 | 0·3057 |
| Maize (average, 3 samples) | 0·9216 | 1·4765 | 0·5170 | 0·3063 | 0·8946 | 1·4605 | 0·5127 | 0·3065 |
| Sesame | 0·921 | 1·4746 | 0·5153 | 0·3057 | 0·8953 | 1·4580 | 0·5115 | 0·3048 |
| Olive (average, 4 samples) | 0·9166 | 1·4715 | 0·5144 | 0·3052 | 0·8903 | 1·4546 | 0·5106 | 0·3045 |
| Rape (colza) (average, 4 samples) | 0·9142 | 1·4745 | 0·519 | 0·3077 | 0·8854 | 1·4585 | 0·5201 | 0·3085 |
| Neatsfoot | 0·9163 | 1·473 | 0·5162 | 0·3062 | 0·8875 | 1·4559 | 0·5137 | 0·3062 |
| Sperm | 0·882 | 1·4675 | 0·530 | 0·3149 | 0·8522 | 1·4508 | 0·529 | 0·3158 |
| Mineral oil | 0·8654 | 1·4759 | 0·5499 | 0·3259 | 0·8383 | 1·4606 | 0·5494 | 0·3271 |
| Vacuum mineral oil | 0·8625 | 1·477 | 0·5530 | 0·3276 | .. | .. | .. | .. |

Referring to Tables IV., V., and VI., it will be noted that the refractive constants (by either formula) are by no means simply proportional to the refractive indices or to the specific gravities of the fats. Castor oil and "blown" oils show the lowest refractive constants of any liquid oil, though both their gravities and refractive indices are high, while raw linseed oil and the fish oils, which approach them in both these particulars, have very high refractive constants. On the other hand, sperm oil, with low gravity and low refractive index, has the highest refractive constant of any known oil, if we except the hydrocarbon, petroleum, and rosin oils, which are still higher. Turning to solid fats, we find that lard and palm oil are high, while hard tallows and cocoanut and palm-nut oils are low.

Before discussing the practical application of these facts to oil analysis, it is worth while to examine their chemical meaning. The dependence of the refractive constant—or, as it is conveniently called, the "specific refraction"—of a body on its chemical composition was early pointed out by Gladstone. If two liquids of different specific refraction are mixed together, the specific refraction of the mixture is equal to the sum of the refractions of the constituents, each multiplied by the fractional part which it forms of the mixture. Thus, using the Gladstone constant, and the gravities and indices for water and glycerol given by Skalweit, we have for 100 per cent. glycerol a specific refraction of 0·3748, and for water of 0·330. Multiplying each of these by 0·5, and adding, we have 0·3539—exactly the specific refraction obtained by employing the values given in the same table for 50 per cent. glycerol. (It is possible that this very close agreement is due to the Skalweit table being calculated by the Gladstone formula, as in this case the Lorenz formula gives a decidedly less accurate result. Ostwald, however, states that for such purposes the Gladstone formula is generally the more accurate, and it is certainly more convenient in reckoning.) It was shown by Gladstone and Landolt that, speaking in general terms, the same process could be applied to determining the specific refraction of chemical compounds; and they calculated specific refractions for the elements, which, multiplied by the number of atoms of each present in a compound and divided by its atomic weight, would give approximately its specific refraction according to their formula. They found, however, that the numbers were

TABLES V. AND VI.

| —— | Sp. Gr. at 60° C. | Refractive Index at 60° C. | Refractive Constant at 60° C. | |
|---|---|---|---|---|
| | | | $R_G = \frac{n-1}{d}$ | $R_a = \frac{n^2-1}{(n^2+2)d}$ |
| *Fats.* | | | | |
| Wood fat (purified) | 0·885 | 1·465 | 0·525 | 0·3124 |
| Japan wax | 0·907 | 1·450 | 0·495 | 0·2963 |
| Oleostearine | 0·907 | 1·449 | 0·495 | 0·2957 |
| Cocoanut oil | 0·897 | 1·442 | 0·493 | 0·2950 |
| Palm-nut oil | 0·896 | 1·443 | 0·495 | 0·2959 |
| Butter fat (average 5 samples). | 0·897 | 1·4503 | 0·502 | 0·2998 |
| Mutton tallow | 0·9074 | 1·4531 | 0·499 | 0·2979 |
| Beef tallow | 0·901 | 1·442 | 0·490 | 0·2937 |
| Bock tallow | .. | 1·4495 | .. | .. |
| Horse fat | 0·894 | 1·455 | 0·509 | 0·3035 |
| Bone fat | 0·894 | 1·451 | 0·504 | 0·3012 |
| Lard | 0·886 | 1·452 | 0·510 | 0·3044 |
| Palm oil | 0·8837 | 1·451 | 0·510 | 0·3047 |
| Cacao butter | 0·887 | 1·450 | 0·507 | 0·3030 |
| Vaseline | 0·851 | 1·470 | 0·553 | 0·3070 |
| Paraffin wax | 0·776 | 1·434 | 0·559 | 0·3356 |
| *Fatty Acids.* | | | | |
| Stearic acid | 0·8469 | 1·4361 | 0·5149 | 0·3088 |
| Palmitic acid | 0·851 | 1·434 | 0·510 | 0·3060 |
| Oleic acid | 0·870 | 1·447 | 0·514 | 0·3072 |
| Mixed tallow acids | 0·875 | 1·442 | 0·5051 | 0·3024 |
| Mutton tallow acids. | 0·875 | 1·442 | 0·5051 | 0·3024 |
| Mixed olive oil acids (3 samples). | 0·878 | 1·446 | 0·508 | 0·3037 |
| Mixed cod oil acids | 0·882 | 1·462 | 0·5238 | 0·3117 |
| Moller's cod oil acids. | 0·890 | 1·4615 | 0·5162 | 0·3069 |
| Medicinal cod oil acids. | 0·882 | 1·4578 | 0·519 | 0·3092 |

not in all cases quite independent of the mode of combination of the compound, and that especially it was necessary to assign higher values to the double-linked carbon of unsaturated compounds, and the double linked oxygen of aldehydes and carbonyl. This discovery has been amply confirmed by later investigators, and numbers have been determined by Brühl and Conrady, using the Lorenz constant, which conform with much greater accuracy than the original figures of Gladstone and Landolt to the results of direct determination.

(*Note.*—As has been pointed out, the Gladstone and Landolt figures may be approximately reduced to correspond with the Lorenz formula by multiplication by 0·6, but the numbers thus obtained for carbon and hydrogen diverge considerably from the results of later investigators. If, however, they are calculated for $CH_2$ in each case, the correspondence is good, and, as a large part of the organic carbon exists in this or equivalent combinations, but little difference arises in the final results of a calculation. It is obvious that such divergences inevitably arise from the indirect mode in which the figures are obtained, and they can only be gradually eliminated by the comparison of the refractions of a large number of compounds of different types.)

For convenient reference, the most important of these values of the atomic refractions have been brought together in Table VII. Gladstone and Landolt's figures refer to the Gladstone-Dale formula $R_G = \frac{n-1}{d}$, and the *a* or red-hydrogen line, Brühl's to the Lorenz formula $R_L = \frac{n^2-1}{n^2+2} \cdot \frac{1}{d}$ and the *a* line, and Conrady's to the same formula and the D or sodium line. The differences introduced by these variations in the spectral line referred to are not very large. Conrady's determinations are the latest and most accurate, and will be used in the calculations which follow, especially as they refer to the D line, for which the writer's determinations have been made.

To make the use of these figures perfectly clear, it may be worth while to give, as an example, a calculation of the specific refraction of oleic acid, using Conrady's figures:—

$$
\begin{array}{lll}
C_{18} = 216 \ldots\ldots\ldots & 18 \times 2·501 = 45·018. & \\
H_{34} = \phantom{0}34 \ldots\ldots\ldots & 34 \times 1·051 = 35·734. & \\
O_2 \phantom{0} = \phantom{0}32 \ldots\ldots\ldots & \left\{ \begin{array}{l} 1 \times 1·521 = \phantom{0}1·521 \text{ (alcoholic).} \\ 1 \times 2·287 = \phantom{0}2·287 \text{ (carboxyl).} \end{array} \right. & \\
 & \text{Add for ethylene bond} \quad 1·707 & \\
\overline{\text{Mol. wt.} = 282.} & \text{Atom refract.} = \overline{86·267} \div 282 = 0·3059 = R_L. &
\end{array}
$$

TABLE VII.

*Atom-Refractions of the Elements.*

| —— | Gladstone and Landolt. $R_{Ga.}$ | Bruhl. $R_{La.}$ | Conrady. $R_{LD.}$ |
|---|---|---|---|
| Carbon | 5·0 | 2·365 | 2·501 |
| Add for double (ethylene) bond | 2·4 | 1·836 | 1·707 |
| " triple (acetylene) bond | .. | 2·220 | .. |
| Hydrogen | 1·3 | 1·103 | 1·051 |
| Oxygen (alcoholic) | 2·8 | 1·506 | 1·521 |
| " (ethereal) | .. | 1·655 | 1·683 |
| " (aldehydic or carboxyl) | 3·4 | 2·328 | 2·287 |
| Chlorine | 9·8 | 6·014 | 5·998 |
| Bromine | .. | 8·95 | 8·927 |
| Iodine | .. | 13·99 | 14·12 |

The average value for R deduced from Table I. is 0·3072. The difference from the theoretical value may be due to want of purity in the sample, to some inaccuracy of Conrady's figures, or to some unexplained peculiarity in the structure of oleic acid. The presence of less saturated acids would raise the observed specific refraction of the sample, but it may be remarked that even for linolenic acid the theoretical value is only 0·3075. It is not probable that the experimental errors extend beyond the fourth place of decimals. It may be noted that the observed values for stearic and palmitic acids (Kahlbaum's) are also higher than the theoretical, the former being 0·3088 and 0·3060, and the latter 0·3051 and 0·3026, respectively, while a specimen of paraffin wax gave 0·3356, as compared to the calculated value for the paraffin, $C_{18}H_{38}$, of 0·3314. Of course the mean molecular weight of the paraffin observed is only conjectural, and the observed number would lie between the calculated values for $C_{15}H_{32}$ and $C_{16}H_{34}$. The observed values for stearic acid and palmitic acid are clearly too high, and it is not improbable that they are influenced by the nearness of the temperature at which they were taken (60° C.) to their melting-point.

In calculating theoretical values for the specific refraction of complicated substances like triolein, it is frequently a saving of time to follow the equation of their formation; *e.g.*:—triolein = glycerol + 3 oleic acid − 3 water. Thus, again using Conrady's figures—

| —— | Molecular Weight. | Atomic Refraction. |
|---|---|---|
| Glycerol | 92 | 20·474 |
| 3 Oleic acid | 846 | 258·801 |
| | 938 | 279·275 |
| − 3 Water | − 54 | −10·869 |
| | 884 | 268·406 ÷ 884 = 0·3036 = $R_L$ |

The observed value for olive oil deduced from Table II. is 0·3046, but it must be remembered that olive oil contains not only traces of palmitin, but of acids less saturated than oleic, its iodine value sometimes exceeding that calculated for pure triolein.

Table VIII. (see next page) gives the calculated values of specific refractions for the principal fatty acids and glycerides, and for some allied substances.

The influence of the different elements on the specific refraction of a compound, as shown in Table VII., is somewhat masked by the difference of atomic weight, and will be more obvious if calculated for unit weight, as in the following table, instead of for that of the atom:—

TABLE IX.

*Specific Refractions of some Elements for Unit Weight (from Conrady).*

| | |
|---|---|
| Carbon (saturated) | 0·1709 |
| " (ethylene) | 0·3507 |
| " (acetylene) | 0·3934 |
| Hydrogen | 1·0510 |
| Oxygen (hydroxyl) | 0·0951 |
| " (ethereal) | 0·1052 |
| " (carboxyl) | 0·1429 |
| Chlorine | 0·1689 |
| Bromine | 0·1116 |
| Iodine | 0·1112 |

Table VIII.

*Specific Refractions from Conrady's Values.*

| Substance. | Formula. | Molecular Weight. | Specific Refractions $\frac{n^2-1}{(n^2+2)d}$ |
|---|---|---|---|
| Butyric acid ......... | $C_4H_8O_2$ | 88 | 0·2525 |
| Tributyrin ........... | $C_3H_5(C_4H_7O_2)_3$ | 302 | 0·2526 |
| Lauric acid .......... | $C_{12}H_{24}O_2$ | 200 | 0·2952 |
| Trilaurin............. | $C_3H_5(C_{12}H_{23}O_2)_3$ | 638 | 0·2927 |
| Myristic acid......... | $C_{14}H_{28}O_2$ | 228 | 0·2995 |
| Trimyristin .......... | $C_3H_5(C_{14}H_{27}O_2)_3$ | 722 | 0·2970 |
| Palmitic acid ........ | $C_{16}H_{32}O_2$ | 256 | 0·3026 |
| Tripalmitin .......... | $C_3H_5(C_{16}H_{31}O_2)_3$ | 806 | 0·3002 |
| Stearic acid .......... | $C_{18}H_{36}O_2$ | 284 | 0·3051 |
| Tristearin............ | $C_3H_5(C_{18}H_{35}O_2)_3$ | 890 | 0·3029 |
| Arachidic acid ....... | $C_{20}H_{40}O_2$ | 312 | 0·3073 |
| Triarachidin ......... | $C_3H_5(C_{20}H_{39}O_2)_3$ | 974 | 0·3051 |
| Oleic acid............ | $C_{18}H_{34}O_2$ | 282 | 0·3059 |
| Triolein.............. | $C_3H_5(C_{18}H_{33}O_2)_3$ | 884 | 0·3036 |
| Linolic acid.......... | $C_{18}H_{32}O_2$ | 280 | 0·3067 |
| Trilinolin ............ | $C_3H_5(C_{18}H_{31}O_2)_3$ | 878 | 0·3043 |
| Linolenic acid ....... | $C_{18}H_{30}O_2$ | 278 | 0·3075 |
| Trilinolenin.......... | $C_3H_5(C_{18}H_{29}O_2)_3$ | 872 | 0·3055 |
| Ricinolic acid ........ | $C_{18}H_{34}O_3$ | 298 | 0·2946 |
| Triricinolin .......... | $C_3H_5(C_{18}H_{33}O_3)_3$ | 932 | 0·2929 |
| Hydroxystearic acid.. | $C_{18}H_{35}(OH)O_2$ | 300 | 0·2939 |
| Dihydroxystearic acid | $C_{18}H_{34}(OH)_2O_2$ | 316 | 0·2854 |
| Glycerol ............ | $C_3H_8O_3$ | 92 | 0·2225 |
| Paraffin.............. | $C_{18}H_{38}$ | 254 | 0·3344 |

The effect of hydrogen, therefore, in raising the specific refraction of a compound is much higher than that of any other element; carbon occupies an intermediate place; while hydroxyl oxygen is lowest of all, and has only about 1/11th the value of an equal percentage of hydrogen. The effect is equally clear in the calculated values of compounds in Table XI., the influence of oxygen in glycerol being especially striking; while the saturated hydrocarbons, as instanced in paraffin, are much higher than any fatty acid. The lowering effect of oxygen is also very clearly shown in hydroxy- and dihydroxy-stearic and ricinolic acids as compared to stearic and oleic, and in the "blown" oils of Table IV. as compared to similar oils unoxidised. The larger proportion of carbon causes the lower specific refraction of the glycerides as compared to the corresponding fatty acids, the latter being about 0·002 higher on the average.

The effect of high iodine value in increasing the refractive index, or, in other words, the influence of the double bond, is by no means so marked as might have been expected, and is probably more obvious when the simple refractive index only is considered, than it is in the specific refraction, as the tendency of the double linkage is to raise the gravity as well as the refractive power due to constitution, and so produce a double effect on the index, which is partially neutralised in the specific refraction. The difference of calculated values of 0·0008 between stearic and oleic acid is scarcely beyond the limits of experimental error, and the actual results of observation quoted in Table VI. actually make stearic acid the higher of the two. The effect is more obvious in the drying oils, though even there it is hardly marked enough for useful analytical distinction.

Summing up results, it may be remarked that a specific refraction higher than that normal to any particular oil usually points to the presence of unsaponifiable hydrocarbons, or of higher alcohols, such as those of sperm oil and wool fat, especially if the gravity be low. Moderately high specific refraction with high gravity may be due to unsaturated acids. These cases are at once distinguished by the saponification and iodine values. Low specific refraction seems in most cases to be due to the large proportion of oxygen present, but this may either arise from the fatty acids being of low molecular weight, so that the carboxyl and hydroxyl oxygen bears a large proportion to the alkyl group, or from the latter being hydroxylated or otherwise oxidised. Thus, in fatty acids of high molecular weight, low specific refraction points to the presence of ricinolic or hydroxy acids, or of the oxidised products present in blown oils; and, if the refractive constant of the original oil be known, is a measure of the oxidation which has taken place. As such, it may prove useful in the technology of oil oxidation. It may be pointed out that the presence of bromine, iodine, and, in a somewhat less degree, of chlorine, would have a similar lowering effect on the specific refraction; and it is possible that the method might be applied to the examination of the products of the action of these elements upon oils. Turning to the solid fats, the lower specific refractions of the harder as compared to the softer fats are noteworthy, and in several cases seem lower than can easily be accounted for on theoretical considerations, though in others they may be explained by the presence of acids of low molecular weight, such as myristic and lauric. The low constant of Japan wax would probably render its detection in mixture with true waxes easy; while the marked contrast between cacao butter and cocoanut and palm-nut oils is rather difficult of explanation. Cow butter has a low constant, in spite of its oleic acid, which is, no doubt, due to the presence of butyrin.

It is fair to remark that the foregoing paper has been somewhat altered and amplified since the meeting at which it was discussed, so that possibly some criticisms are less applicable than they were at the time they were made. I must express my obligation to my assistants, Mr. M. C. Lamb and Dr. T. Guthrie, for much help, both in the many determinations involved and in the subsequent arrangement of their results.

## Discussion.

Dr. Lewkowitsch said he considered Amagat and Jean's a fairly good instrument, because it worked on a differential principle. Changes of temperature were not likely to give much trouble, since the oil under examination and the type oil were equally influenced. It was necessary to notice, however, as had been pointed out by Mr. Allen, that, owing to faulty manufacture, not all instruments gave identical results, so that two observers working with different instruments might obtain different figures for the same substance. Although the identity of the type oil supplied with Amagat and Jean's instrument was kept secret by the makers, it was known to be sheep's-foot oil. Of course, the apparatus being a differential one, all values referred to the zero adopted for that oil. He had arranged all the oils and fats the refractive index of which had been determined by means of the oleorefractometer, with a view to seeing whether any correlation could be established between the refractive index and the iodine value. At first sight it would appear as if—speaking broadly—this interdependence did exist—that the higher the iodine value, the greater the deviation to the right. Castor oil, however, did not fit in this rule, and it was certainly interesting to have some light thrown on this exception by Prof. Procter's paper. The terrestrial animal oils and fats all deviated to the right, but, unfortunately, no differentiation could be established between animal and vegetable oils with the help of the refractive index, inasmuch as the fish, liver, and blubber oils—in agreement with their high iodine values—showed strong deviations to the right.

By means of a refractometer like Abbe's the refractive index could certainly be expressed in more strict accordance with scientific parlance, but it was rather difficult in practice to remember a set of figures having four decimals, and differing only in the third or fourth decimal. Therefore the butyro refractometer, practically a differential apparatus, had so quickly found favour with chemists. He quite agreed with what Prof. Procter had said regarding this instrument, and the convenient adjustment of temperature. It should, however, be pointed out that it is not quite correct to *calculate* observations made at one temperature to another; it had therefore become customary on the Continent to take all readings at 40° C., at any rate in the case of fats. The refractive index certainly gave very valuable indications, but it should be remembered that too far-reaching conclusions as to identity or impurity of an oil under examination must not be drawn. It is true one could calculate the refractive index of a mixture from the known indices of the components, but the most one should expect at present of refractometric examination was to employ it as a sorting test, say, for detecting gross adulterations, or for singling out suspected samples for further examination by chemical methods. Thus, in the examination of lards wrong conclusions might be arrived at, according as they were of European or American origin, as they not only differ in iodine value (no doubt because of the different feeding of the pigs), but also in the refractive index. Similarly, in the case of butter, if the refractive index alone were accorded decisive value, there might easily arise cause for unfounded suspicion. Coming now to the proposed value, *viz.*, the specific refractivity, the new constant certainly required the determination of two constants; but, as the specific gravity was generally taken, no inconvenience would be caused if additional information was obtained thereby. He had already referred to the satisfactory explanation of the apparently abnormal position of castor oil. But, as Prof. Procter had, unfortunately, been unable to place all his figures before the meeting, he should like to ask for further information as to the utility of this new constant in practical oil and fat analysis. In the absence of these figures he could hardly help making the remark that Prof. Procter was ahead of the time. Important information for practical purposes would only be gained if we knew more of the composition of the oils and fats, constituting as they do an intricate mixture of various glycerides, the nature of which was not understood yet, not to speak of the quantitative relation of the components. If this were known, no doubt a valuable help to fat analysis would be afforded. Thus, in the case of a dilute glycerin solution, where we had two definite chemical individuals, water and glycerol, the proportion of the two components might be calculated; in fact, the determination of glycerol in an aqueous glycerin solution by means of the refractive index was a well-known method. But in the case of, say, olive oil one had a certain proportion of less saturated acids than oleic—acids with perhaps two double bonds or one triple bond; and this would explain Prof. Procter's observation, that the specific refractivity of olive oil was greater than that calculated for triolein. Again, in the case of linseed oil, one had mixtures of oleic, linolic, linolenic, and isolinolenic acids (the last two of which, no doubt, would have the same specific gravity and refractivity), and it would appear to him that it was somewhat premature to calculate the specific refractivity of that oil from the rather too scanty data to hand in our present state of knowledge.

Prof. PROCTER, in reply, said that oils might either be differentiated by index of refraction or by gravity; and by combining the two in a systematic manner, one acquired greater information than with the two separately; *e.g.*, castor oil and linseed were very similar in refractive index, but on comparing their specific refractions they were found to stand at opposite ends of the scale. In a complicated mixture like an oil, each new independent constant was of value, as affording one of the equations from which the unknowns were to be determined. The chief advantages of the butyro-refractometer over that of Amagat and Jean were, that it was cheaper and easier to use, the index of refraction might be got direct, and it had a water-jacket. He understood, however, that the latter instrument had also been furnished with one recently.

## Obituary.

### MANNING PRENTICE, F.C.S.

MEMBER OF THE SOCIETY OF CHEMICAL INDUSTRY.

MANNING PRENTICE was born at High Easter on May 4, 1846. He studied chemistry under Prof. A. W. Williamson, at University College, London, during the years 1863 and 1864, and then went to Stowmarket to assist his father in the management of the chemical works belonging to Thos. Prentice and Co.

In 1868 the chemical works became a separate concern, Manning Prentice and his uncles, Eustace Prentice and Edward Prentice, being the original partners of the new firm.

Edward Prentice died in 1871 and Eustace Prentice in 1881. From this time the main responsibility of the business lay with Manning Prentice, who, by careful management, succeeded in placing it on a sound financial basis. In 1891 he converted it into a limited company, and was appointed managing director, which position he occupied until his total breakdown in health about two years ago.

In 1890 he made a tour through the United States of America, visiting many of the largest sulphuric acid works and the Florida phosphate deposits, in order to make himself acquainted with the latest American practice; and in 1891 he visited Russia. He is probably best known as the inventor of the corrugated platinum pan for concentrating sulphuric acid (1876), and of the continuous still for the manufacture of nitric acid (this Journal, 1894, 323).

Manning Prentice took an active part for many years in the affairs of the Association of Chemical Manure Manufacturers, of which he was elected President in 1896.

In 1886 he contributed a series of articles on "Scientific Agriculture" to the "Land Agents' Record," which met with so much appreciation that they were reprinted in pamphlet form early in the following year.

Manning Prentice was elected a Member of the Council of the Society of Chemical Industry in June 1893, and a Fellow of the Chemical Society in 1864.

He acted as secretary to the British Association Committee appointed, in 1895, to investigate "The Carbohydrates of Cereal Straws," and was an active member of various agricultural societies in different parts of the kingdom.

He was a justice of the peace for the county of Suffolk, and at one time took an active part in local affairs.

In private life, his refined and sensitive nature led him to find his recreation in literature, the fine arts, and music. He leaves a widow, two sons, and two daughters.

He died on Nov. 20, quite suddenly, and was buried at Stowmarket on Nov. 23.

## Journal and Patent* Literature.

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## I.—PLANT, APPARATUS, AND MACHINERY.

*Brickwork Pillars, Testing.* Riga Indust. Zeit.; Thonind. Zeit. 22, [51], 673.

*See under* IX., *page* 1049.

### PATENTS.

*Compressing Powdered Materials into Tablets, Impts. in Machines for.* J. Y. Johnson, 47, Lincoln's Inn Fields, London. From H. K. Mulford, Philadelphia, U.S.A. Eng. Pat. 22,016, Sept. 25, 1897.

THE powdered material is fed from a hopper through a flexible tube to the feed-plate l, which delivers it into the die or mould *f*, mounted in the table $A^2$ of the machine.

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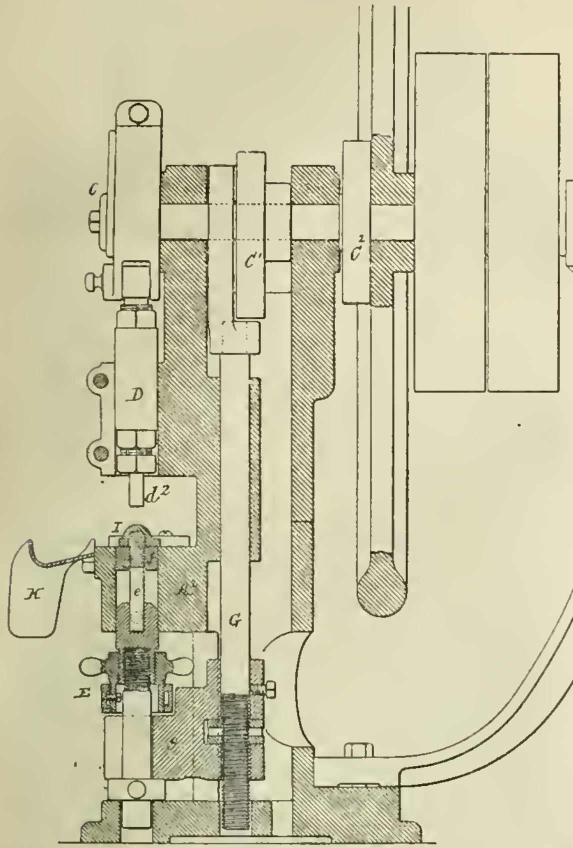
* Any of these specifications may be obtained by post by remitting 8*d.*—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.



When the mould is filled, the feed-plate is moved back, by a lever connection from the cam C² on the driving-shaft, clear of the punches $d^2$, $e$, which are then actuated by the eccentric C and cam C¹, to compress the material in the mould into a tablet. The punch $d^2$ is mounted on the plunger D, which is adjustably connected to the eccentric C, and the punch $e$ is adjustably mounted on the plunger E, which is connected by the forked arm $g$ to the rod G, actuated by the cam C¹. The various adjustments are provided to enable the pressure on the punches to be regulated, and to ensure that the finished tablet shall be forced clear of the mould by the punch $e$. The feed-plate I, on its return movement to refill the mould, discharges the finished tablet from the table A² into the chute K.—R. A.

*Kilns for Burning Limestone and other Materials, Impts. in.* V. Hughes, Tipton, and E. D. Nicholson, Llynelys. Eng. Pat. 23,403, Oct. 12, 1897.

This patent is for "a kiln, for burning limestone or other material, into which the fuel is introduced in a gaseous form from a producer, and from which air, introduced into the material through the lower portion of the kiln, and which has thereby become heated, is withdrawn in a continuous stream or streams by chimney draught, or by a mechanical arrangement for that purpose, before it reaches the part of the kiln into which the products of combustion pass from the combustion chamber into the material which is being burnt, and is utilised to heat the air which is supplied to the combustion chamber."—R. S.

*Filters, Impts. in Apparatus for Feeding.* T. W. H. Garstang, Knutsford; J. McD.McKenzie, Hale; and G. E. Ridgway, Ashley, all in Cheshire. Eng. Pat. 27,938, Nov. 27, 1897.

An apparatus for automatically feeding a series of filters in rotation with a constant amount of liquid. The liquid, as it issues from the siphoning chamber, fills successively a number of buckets (eight, for instance) attached to a water wheel. By means of levers connected with a float in the siphoning chamber, the wheel is caused to make one-eighth of a revolution at a time, and to discharge the contents of each bucket as it reaches a certain point. The number of filters corresponds with that of the buckets. The valves of each filter are opened in succession by means of arms attached to the shaft of the water wheel; one valve is thus always open, and each filter gets an influx of liquid and a period of rest.

—N. H. J. M.

*Liquefiable Gases, An Improved Method of Utilising, in the Operation of Engines or Motors.* E. Vignon-Roure, Camden City, New Jersey, and C. A. Lane and J. L. Kelly, both of Philadelphia, all in U.S.A. Eng. Pat. 16,193, July 25, 1898.

The method consists in mixing with a liquefiable gas (such as carbon dioxide) of high pressure at a given temperature, another liquefiable gas (such as methyl chloride, or a mixture of methyl chloride and toluene) of lower pressure at the same temperature, the latter gas having an absorbing affinity for the former, heating said gases, conducting them through an engine or motor to operate the same, and again condensing the gases. In the process of heating, the gases are dissociated into a mechanical mixture, so increasing their pressure, whilst, during the condensation process, the gas first condensed reasserts its affinity for, and absorbs the vapours of, the other gas, before the point of liquefaction of the latter gas is reached, so diminishing the back pressure and correspondingly increasing the working pressure. The chloride of methyl employed in the process serves as a natural lubricant.—R. A.

## II.—FUEL, GAS, AND LIGHT.

*Coal, A Contribution to the Chemistry of, with special reference to the Coals of the Clyde Basin.* W. C. Anderson. Proc. Glasgow Phil. Soc. **29**, 72–96.

The coals in the upper part of the Clyde Basin series are entirely non-coking in character, *i.e.*, they are not capable of being employed for the manufacture of metallurgical coke, and those lying between the position of the Palace Craig Ironstone and the Airdrie Blackband Ironstone are not used for coking purposes in any part of the district.

*The Ell Coal*, bright black, with cubical fracture and brownish coloured ash. Brittle, soon breaking up on exposure to the atmosphere. The large coal is sold exclusively for household purposes, whilst the small coal is a good fuel for steam raising.

*The Main Coal*, bright black, with cubical fracture and white ash. Very brittle, soon falling to pieces on exposure to the weather. It is never used for coke making, and the portion used for the manufacture of gas, when distilled in the retorts, yields a soft friable coke of little value.

*The Splint Coal*, hard and strong, and of a dull bluish-black colour. Chiefly used for iron blast furnaces and gas making but never for coking.

*The Cannel Coal* (gas coal), in the Hamilton district, found separating the Splint coal from the Virgin coal, is a hard brittle coal with smooth conchoidal fracture. There is usually a rib of bright coal between the Splint and the Cannel which does not readily part from the latter, and the change is not marked by a distinct line or bed of stratification, although it is sharp. This seems to indicate that whatever may have been the process by which these two qualities were produced, the change of conditions was sudden but the process continuous.

*Virgin Coal*, a bright black coal, coarser and stronger than Ell coal. A fair household coal and is never coked.

*Virtuewell Coal* resembles Virgin coal, and is sometimes used to make an inferior quality of coke.

*Upper Dumgray Coal* is, as a rule, a hard blue-black coal, suitable for furnace purposes. In the Slamannan district the intrusive whin has converted it into a steam coal of very good quality. In many places the small is used for coking purposes, and where the whin has not been excessive, it is a valuable coking coal.

*The Lower Dumgray* is a bright black coal, sometimes a first class house coal, but softer than the former.

The remaining seams are inferior, until we reach the *Slatyband Ironstone.* From this seam to the position of the Index Limestone the few coals which are found are inferior in quality.

Below the Index Limestone is a group of coals of a strong coking quality, well developed and largely worked on the northern outcrop of the basin, but of little value on the south. The seams vary very much in thickness and it is impossible to trace each for any distance.

*The Kilsyth Haughrigg* and *the Bannockburn Main*, are among the thickest of this group. They are soft black coals of irregular fracture, easily affected by atmospheric influences.

*Kilsyth Coking, or Banton Main Coal* takes a well defined position over a large area, but is best developed in the immediate vicinity of Kilsyth. Its characteristics are similar to the preceding coals, and from it a good coke is made.

The above coals represent the principal seams belonging to the upper, middle, and lowest divisions of the Clyde basin series. In the latter it may be generally affirmed that the coking property increases with depth, although this cannot be said to be a constant feature of all coal fields. The present investigation was undertaken by the author more particularly with a view to finding out points of difference in character and reaction between coals which might be regarded as non-coking and those which are true coking varieties. [For tables of analyses, see page 1015 *et seq.*]

For the purpose of obtaining a comparative estimate of the caking power, 1 grm. of the coal was ignited with the largest quantity of fine white sand, which it is capable of binding into a mass that just coheres. The weight of sand in grms. is the "caking index" (Campredon's test). The following results were obtained:—

| | |
|---|---|
| Ell | 4·0 + |
| Main | 4·0 |
| Splint | 3·5 |
| Gas | 3·5 − |
| Virgin | 4·0 |
| Kilsyth Haughrigg | 9·0 + |
| Bannockburn Main | 15·5 |
| Kilsyth coking | 16·0 + |

The last three are true caking coals, and are all used for the production of high-class foundry coke. In the crucible they give strongly-swollen, hard, metallic looking "cokes." The Main and Ell coals give slightly swollen residues, whilst those from Splint, Gas, and Virgin are flat or slightly concave cokes of less bulk than the original coal.

*Extraction with Solvents.*—The substances extracted with cold gasoline and carbon bisulphide were found to be similar in character to those obtained by Muck, Siepmann, and others; but as they were not present in greater quantity in coking coals than in non-coking, and their removal exercised little, if any, deteriorating effect upon the coking property, their investigation was not pursued further.

*Treatment with Caustic Potash.*—One grm. of each of the coals in fine powder was boiled for one hour with 100 c.c. of a 5 per cent. solution of caustic potash, and the residue filtered off and washed till free from alkali. Ell, Main, and Virgin coals gave a dark coffee-brown coloured filtrate, from which acid precipitated a small quantity of a brown humus-like substance. Splint and Gas coals gave a pale amber-coloured filtrate, whilst the true coking coals gave only the faintest trace of colour. On igniting these extracted coals in a closed crucible, the first five coals showed no tendency towards cohesion, the residues being loose, dull, earthy-looking powders several per cents higher in weight than the "cokes" obtained from the dry untreated coal. In the case of the three coking coals, however, caustic potash appeared to cause very little deterioration in the coke.

*Treatment with Nitric Acid.*—Two grms. of the dry powdered coal were mixed with 25 c.c. of nitric acid (sp. gr. 1·40) in a small beaker surrounded by cotton wool, and the rise in temperature noted; it ceased in about seven minutes in each case, with the exception of Main, which showed no further rise after five minutes.

| | Rise in Deg. C. |
|---|---|
| Ell coal | 17·00 |
| Main | 17·75 |
| Splint | 13·50 |
| Gas | 13·25 |
| Virgin | 15·25 |
| Kilsyth Haughrigg | 17·00 |
| Bannockburn Main | 17·00 |
| Kilsyth coking | 17·00 |

One gramme of each coal was evaporated to dryness with 20 c.c. of dilute nitric acid (sp. gr. 1·2) on a water bath, and the residue dried at 105° till constant. In each case a gain in weight was recorded, which, however, differed considerably in amount:—

| — | Increase in Weight. | = Dry Coal. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Ell coal | 10·33 | 11·47 |
| Main | 9·52 | 10·47 |
| Splint | 16·61 | 17·91 |
| Gas | 15·42 | 16·34 |
| Virgin | 12·17 | 13·19 |
| Kilsyth Haughrigg | 18·16 | 18·53 |
| Bannockburn Main | 23·31 | 23·73 |
| Kilsyth coking coal | 23·10 | 23·51 |

Bannockburn Main, after previous partial oxidation in air till coking power had been completely lost, showed an increase in weight amounting to 9·98 per cent., equal to 9·98 per cent. of dry coal.

*Oxidation of Coal.*—Atmospheric oxidation, especially at slightly elevated temperatures, causes the coal to increase in weight so long as it is carried on below a limit which in the generality of cases may be put at about 190°, beyond which point the volatilisation of certain constituents causes usually a nett loss in weight. Bannockburn Main heated in air for one hour at 160° C., showed a gain of 1·98 per cent., and gas coal 0·90 per cent., whilst the weight of the residue left on ignition was 78·02 per cent. and 59·96 per cent. respectively. Bannockburn Main, heated for 33 hours in an air bath at 115°—120° C., showed a gain in weight of 1·96. The weight of residue left on ignition was 75·66 percent. Heated for four hours at 300° C., Bannockburn Main lost 41·02 per cent. in weight and gas coal 56·98 per cent., with a further loss of 12·50 per cent. and 8·40 per cent. respectively after ignition. The ignited residues cohered below, but were in the form of loose dull powder above. In other experiments made below 300° the residues left by both coals were loose powders without any trace of cohesion, even when the previous heating was only for one hour at 160°. The gases given off on ignition in these cases were non-luminous, and the residues showed on the surface a thin layer of finely-divided carbon apparently due to the decomposition of a complex carbon compound. In order to make certain that this loss of coking property was due only to oxidation, the experiment was repeated with Bannockburn Main at 160° in an atmosphere of carbonic anhydride. Two experiments gave, instead of a gain as above, losses of 1·77 per cent. and 1·85 per cent., and the residues on ignition left a bright silvery coke similar to that obtained from the original coal and weighing 71·5 per cent. of the heated sample. It is further shown that the oxidation makes rapid progress during the first 12 to 21 hours, after which a slow increase in weight continues for a long time, the period required to complete the process (and also the increment in weight) varying greatly in the different coals.

*Products of Oxidation with Nitric Acid.*—An attempt was made to prepare in a partially purified condition, the brown acid substances obtained by acting on Ell, Splint, and Bannockburn Main coals with nitric acid. About 5 grms. were evaporated to dryness over a water bath with 200 c.c. of nitric acid (sp. gr. 1·2) the residue dissolved as far as possible by ammonia, and the filtered solution reprecipitated by hydrochloric acid. This operation was repeated and the insoluble acids were frequently washed by decantation. The acids thus obtained are insoluble in water containing mineral acids or salts in solution, but are fairly soluble in pure water. They were afterwards thrown on a filter, washed as far as possible with water and dried for some days at 105°. [For analyses, see page 1018 *et seq.*]

*Effect of Heat on the Coking Property.*—As a further proof of the assertion that the loss of the caking property which takes place when coal lies exposed to air for a lengthened period, and also when it is subjected to the action of heat under 300° during a short time, is due to oxidation and not volatilisation of matter upon which the caking property depends, samples of the above coals were subjected to a temperature of 297°—304° for three hours in a U-tube, through which a slow current of dry carbonic anhydride was passed. The results show (1) that the greatest loss at 300° takes place in Splint, Gas, Virgin, and Main coals—that is in the long-flaming coals, which have also the lowest coking index; the smallest loss is found in the strongest caking coals, Bannockburn Main and Kilsyth coking (caking indices 15·5 and 16), while Ell and Kilsyth Haughrigg, which swell up slightly on ignition in a crucible, occupy an intermediate position; (2) that the percentage of coke rises enormously after the coal has been heated to 300°; and (3) that in the coals of which the ignited residues cohered only slightly and which show the largest percentage loss at 300°, the tendency to cohere is completely destroyed; while in the case of the true caking coals it is, though impaired to some extent, still existent.

The author explains these facts, taken along with others previously mentioned, by the fact that in all the coals experimented with, a set of bodies of a resinoid character exists, which bodies are capable of extraction with dilute caustic potash solution, and which alone are accountable for the tendency to cohere in the residue from Ell, Main, Splint, Gas, and Virgin coals. These bodies are not the same in each coal, but vary alike in kind and quantity. They contribute only to a very slight extent to the weight of the fixed residue obtained on ignition, being for the most part driven off and constituting a large part of the "smoke," to which they contribute a characteristic odour. They are all more or less completely volatilised below 300° except in the case of Ell, where a part seems to remain behind, causing the partial coking of the residue on ignition. In addition to these there is in the true coking coals a constituent not so readily, if at all, acted on by dilute alkaline solutions, oxidisable, like the main constituent of the non-coking coals, in air, but not volatile at 300°. It was found that both Bannockburn Main and Kilsyth Coking coals melt in an atmosphere of carbonic anhydride at a definite point, which may be placed at 317°, and this the author takes to be the melting point of the constituent referred to. In all probability the higher percentage yield of fixed residue usually accompanying coking coals is due to the fact that this body breaks down on heating into fixed carbon and simpler compounds before being itself volatilised. Active gasification does not appear to begin below 330°, and is not particularly vigorous even at this temperature. The evolution of gas at this stage is perhaps to be traced to the decomposition of the other kind of constituent present in coal minerals, and not to the breakdown of the fusible body, which may decompose only at a considerably higher temperature.—D. B.

*Coal Mine [Miike Coal], A Japanese.* Eng. and Mining J. 1898, **66**, [12], 332.

The coal obtained from the colliery worked by the Mitsui Mining Company of Tokio, is a good bituminous variety, making very fair coke, but containing a rather high percentage of sulphur. The seam dips very gently—less than 20 degrees—and is quite regular in thickness, averaging 9 ft.; it is opened for a distance of three miles along the strike by four shafts and several slopes. A fifth shaft, now being sunk, will strike the seam at a depth of about 1,000 ft. There is no selvage, and the seam is free from thin bands of poor coal or partings of slate. There are present, however, irregularly lenticular masses of calcareous matter, often containing sulphate of lime, and these bodies necessitate, in some places, considerable hand-sorting of the coal. Owing to the heavy flow of water, the item for pumping is an important one in the mining expenses. The mine is worked on the pillar and stall system, little powder being used except for dead work, whilst the coal is easily broken down with the pick. The coal sells for about 3·5 dols. at the mine. Various improvements are in contemplation. (See page 986.)—A. S.

*Fire-damp and Air, Behaviour of, when admitted to an Incandescent Electric Lamp.* H. Couriot and J. Meunier. Comptes Rend. 1898, **127**, [16], 559—561.

On admitting a mixture of fire-damp and air of maximum explosive power (9·5 per cent. of methane) to 10 candle-power incandescent lamps run with a voltage varying from 15—110 volts, no explosion could be produced, provided the filament were not already broken. Water and carbon dioxide were formed in the interior of the lamps, but only on one occasion was there an explosion. In this case the filament was broken before the gas was admitted, and the explosion was probably caused by one of a series of sparks produced by the two ends of the filament coming together owing to vibrations. Subsequent attempts to repeat the experiment and explode another lamp have been unsuccessful.—J. S.

*By-Product Coke Ovens [Semet-Solvay and Otto-Hoffmann], Producing Illuminating Gas and Coke in.* H. O. Hofman. Eng. and Mining J. 1898, 428—430 and 458—460.

A block of 10 Semet-Solvay coke-ovens, each 30 ft. long, 5 ft. 6 ins. high, and 16 to 17 ins. wide, has, since 1897, supplied illuminating gas of 18 candle-power and heating-gas of 8½ candle-power by separate systems of mains to the town of Halifax, N.S. 32·26 per cent. of the gas made is illuminating gas, and the remainder is heating gas, from which the gas required—54·84 per cent. of the total gas production—for heating the ovens is taken. 37 short tons of coal are coked in 24 hours, and yield 310,000 cb. ft. of gas, and 75 per cent. of their weight of coke suitable for domestic use and boiler fuel.

Preliminary experiments have been made at Glassport, Pa., on one oven of a block of 30 Otto-Hoffmann ovens for the purpose of testing the coal which it is intended to coke in 400 ovens which are being erected near Boston for the supply of illuminating gas to that city. The average charge was 14,591 lb. of coal, containing 9·9 per cent. of moisture, and the time required to work off such a charge, averaged 33 hours 56 minutes. The yield of gas per long ton of dry coal averaged 10,390 cb. ft., of which 49·5 per cent., produced in the first 14 hours 46 minutes, was of 14·7 candle-power, and was available for illuminating purposes, and the remaining 50·5 per cent. subsequently produced, was of 9·0 candle-power, and sufficed to heat the oven. The average calorific value of this heating gas was 566·7 B.T.U. 69·70 per cent. of fair commercial metallurgical coke was obtained from the coal. 18·5 per cent. of the nitrogen of the coal was converted into ammonia, and 3·38 per cent. of tar of good quality was obtained. Of the original calorific value of the coal, 72·3 per cent. was found in the coke available for sale, 4·1 per cent. in the tar, 12·7 per cent. in the gas for sale, and 10·7 per cent. in the gas required for heating the ovens. The balance, amounting to only 0·2 per cent., is in the liquor or sulphur recovered, or is lost. Therefore only 10·7 + 0·2 = 10·9 per cent. of the calorific value of the coal is consumed in its destructive distillation, which is a better result than gasworks treatment of coal affords. Chemical and thermal investigations indicate that the production of illuminating gas in by-product cokeovens rests upon a very sound basis. It is also satisfactory financially.—J. A. B.

*Flame of Acetylene Gas, Effect of Carbon Dioxide on the.* J. Gas Lighting, 1898, **72**, [1850], 916.

At this year's meeting of the British Association, Prof. Emerson Reynolds performed an interesting experiment illustrating the effect on an acetylene flame of varying proportions of carbon dioxide in the gas. It was the result of a chance observation by Mr. Goodwin, who conducted a series of tests in Philadelphia on the enrichment value of acetylene when added to ordinary coal gas. He stated that expired air, when mixed with acetylene, appeared to increase the luminosity of the flame, and also to decrease the tendency to deposit carbon in the burners. More careful study, however, had shown that from 5 to 8 per cent. of carbon dioxide in the gas decreased the smokiness of the flame, and especially prevented the clogging of the burners. The increase in illuminating power was certainly not marked; but the mixture containing 5 per cent. of carbon dioxide gave as much light as the acetylene itself, and therefore there was a gain to this extent per volume of acetylene burned. The action of the carbon dioxide was regarded as probably due to its exerting some oxidising effect.

### *Acetylene Generators, After-gasification in.* P. Wolff. Metallarbeiter, **24**, [16], 122—124.

An inherent defect in acetylene generators, whether of the suffusion- or immersion-type, is the after-gasification ensuing subsequent to the interruption of direct contact between the carbide and the water, and arising from the action of the residual water present in the pores of the carbide, the deposition of condensed moisture thereon, or the absorption of aqueous vapour thereby. The author has measured the amount of gas thus liberated by 1 kilo. of carbide, by passing the gas through a meter, the carbide holder being in one series stored over water and in a check series, over oil. The results showed that whereas under the latter conditions the quantity of gas amounted to ¼ litre at the end of the first half-hour, 6 litres after 24 hours, and remained constant at 16 litres after a lapse of three days, in the other case (over water) 7½ litres were liberated in half an hour, 25 litres in 24 hours, and 50 litres in three days, the evolution proceeding, moreover, at the rate of 5—6 litres per diem up to complete exhaustion of the carbide.

These figures indicate considerable risk of explosion in small generators (*e.g.* table lamps), though the danger can be overcome in larger apparatus by employing a reservoir of sufficient size to store the excess of gas without undue compression.

Various means have been proposed for obviating the above-named defect, and among them the employment of a layer of oil as a seal on the surface of the water, this method being, however, unsuitable in practice, since a thin layer does not prevent the absorption of water vapour, whilst a thick stratum is liable to come in contact with the carbide and render the same totally impervious to water.

The most efficient appears to be that of Münsterberg, wherein a floating valve shuts off all connection between the carbide chamber and the water as soon as the former is raised, and thus restricts the after-gasification to the amount produced by the interstitial moisture in the carbide. Another defect is also remedied in this generator by the provision of a large gas reservoir, by means of which the amount of air introduced along with a fresh charge of carbide is reduced to 20 per cent.—*i.e.*, below the limits of an explosive mixture.—C. S.

### *Acetylene, Production of.* Letang and Serpollet. Eng. and Mining J. 1898, **66**, [13], 368.

The authors propose to steep the fragments of calcium carbide in a hot and concentrated solution of glucose, and then dry. If carbide which has been treated in the above manner be immersed in twice its weight of water, acetylene is given off in the usual way, but at the same time a sucrate of lime is formed. It is stated that under these conditions the generation of acetylene takes place with great uniformity, and is stopped almost immediately on cutting off the supply of water.—A. S.

### *Calcium Carbide, Composition of some Industrial Samples of.* H. Moissan. Comptes Rend. **127**, [11], 457—463.

The manufacture of calcium carbide on the large scale at first presented unforeseen difficulties, which have now been for the most part overcome. Impure lime and coke were used at first, and the carbide produced, was contaminated with calcium phosphide, aluminium sulphide, and silicides. The difficulties which ensued on the use of such carbide, directed attention to the necessity of employing purer lime and carbon, and at the present time well-fused homogeneous carbide of crystalline fracture and of the characteristic reddish-brown tint is found in commerce. Notwithstanding the distinctive appearance of good carbide, an analysis is advantageous; and Lunge and Cedercreutz's method (Zeits. angew. Chem. 1897, 651; this Journal, 1897, 1046) appears to yield good results. The sampling of carbide is a matter of difficulty, owing to its hardness. Theoretically, 1 kilo. of carbide should yield 349 litres of acetylene; and seven samples examined yielded from 292·81 to 318·77 litres per kilo. On the other hand, three samples of greyish carbide, more porous, and less crystalline and fused in appearance than the others, yielded only 228·6, 250·4, and 260·3 litres of gas per kilo. Some samples contain much ammonia; in four cases the following percentages of nitrogen were found: 0·02, 0·12, 0·15, and 0·31. Several samples yielded a small amount of phosphuretted hydrogen, but some were free from it.

The insoluble residue left on the decomposition of calcium carbide by water was specially investigated. The carbide was decomposed by a solution of sugar, which took the lime into solution. The insoluble residue was brought on to a filter, and washed with solution of sugar and then with water, both free from carbonic acid. After treatment with alcohol and ether, and drying in a vacuum at 40° C., it was examined under the microscope, and silicide of carbon, silicide of calcium and iron, particles rich in lime, and in some cases calcium sulphide and graphite, were detected. Dilute hydrochloric acid took into solution some iron, lime, alumina, and phosphorus, and left unattacked silicide of carbon and graphite. Subsequent treatment with the concentrated acid dissolved out lime, iron, and silica. By investigation with the microscope, and attacking the residue in this manner, the nature of most of the impurities was ascertained. No relation between the quantity of insoluble matter and the yield of acetylene was observed.

Silicon was found chiefly as silicide of carbon, but also occasionally as silica and silicide of calcium, and in metallic-like spheres containing also iron and carbon. Ignition of calcium carbide, due to the escape of siliciuretted hydrogen into the air, was never observed when the carbide was treated with a small quantity of water, but that gas was frequently liberated when the residue was treated with concentrated hydrochloric acid.

Sulphur was discovered as calcium sulphide or as aluminium sulphide. The calcium sulphide did not yield sulphuretted hydrogen by the action of water in the presence of hydrated lime, and sulphuretted hydrogen was not found in acetylene produced by the decomposition of carbide with excess of water. If, however, the lime used in the preparation of calcium carbide contains aluminium silicate and some sulphate, silicide of carbon and an aluminium sulphide which is decomposed by cold water with liberation of sulphuretted hydrogen, are produced.

Sulphur cannot be present as silicon sulphide, as this substance readily volatilises at a temperature below that of the electric furnace. Calcium carbide containing some calcium sulphide yielded, when decomposed by water, an organic sulphur compound, which was detected by burning the gas after it had passed through a solution of a nitrate, and bubbling the products of combustion through distilled water, in which a small amount of sulphuric acid was subsequently found. The total amounts of sulphur in three samples of calcium carbide were, 0·37, 0·43, and 0·74 per cent.

Iron was found as silicide and carbo-silicide in very variable quantity, depending on the purity of the carbon used in the production of the carbide.

Phosphorus occurred chiefly as calcium phosphide, which is decomposed by water, but also in metallic-like spheres containing also iron and silicon.

Carbon was present in some samples as graphite. A special search was made for diamond, but it was not found in any samples.—J. A. B.

### *Calcium Carbide, Electric Furnaces for the 110 - Volt Circuit, for the Production of.* N. M. Hopkins. J. Amer. Chem. Soc. **20**, [10], 769—773.

For the laboratory production of calcium carbide, the author arranges three pairs of carbons side by side, and connects them so as to form three arcs in series; the end members are then connected, through 12-ampère fuses, with the main feeders of the ordinary lighting circuit, a switch, a variable resistance, and an ammeter being interposed, and a voltmeter also connected to the terminal carbons. The carbons are arranged on two sloping blocks provided with rough screw motion, so that they can be brought into contact or separated again in the furnace. A single arc can be arranged in a crucible, for small fusions, but in this case a much larger outside resistance will be needed than with the three series arcs.—J. T. D.

*Electric Incandescent Lamps, New, of Nernst and Auer [von Welsbach].* J. für Gasbeleuchtung, 41, [15], 237—238.

*Nernst's Lamp* (see Eng. Pat. 19,424 of 1897; this Journal, 1898, 653) is based on the fact that lime, magnesia, and certain other oxides, are comparatively good conductors of electricity, when at high temperatures, and give an intense white light, without fusing or altering appreciably. At ordinary temperatures, however, they are practically non-conductors. Nernst proposes to make an incandescence body of a hollow cylinder of such substances, to heat it by some auxiliary means to such a temperature that it becomes sufficiently conducting, and then to raise it to incandescence by means of a current, preferably alternating, of moderate intensity. It is stated that on using a thin hollow cylinder of magnesia, of not quite 1 cm. in length, and a current of 0·25 ampère and 118 volts, an illuminating power of 26 Hefner units was obtained = about 1 Hefner unit per watt; whilst ordinary incandescent lamps require 3—4 watts per Hefner unit. (1 Hefner unit = 0·88 English standard candle.) At its present stage the necessity for the preliminary heating of the lamp appears to be a drawback.

*Auer's Lamp* is developed on the principle that the illuminating power of a glowing body increases rapidly with its rise of temperature—approximately as the fifth power of its absolute temperature. A filament is used, the basis of which is metallic osmium. Osmium occurs associated with the Pt metals, and is obtained from Pt residues as "osmiridium." On heating in air it burns to the volatile, pungent tetroxide. It is the heaviest known body (sp. gr. 22·477) and also the most infusible, resisting temperatures at which Pt and Ir volatilise. When heated in a vacuum, or in an atmosphere of certain reducing gases, a filament of osmium can withstand a far higher temperature than one of carbon, and is thus of greater illuminating efficiency, according to the above law. If a filament of Os in a vacuum be heated by a current to the temperature of volatilisation of Pt, it gives an agreeable white light of great intensity, and it may be heated to far above this temperature without melting. The Os must be almost chemically pure, or it may contain a small proportion of Pt, which renders the filament more elastic, without sensibly affecting its infusibility.

A further development of the lamp is an osmium filament coated with a highly refractory oxide, such as $ThO_2$. As is well known, a Pt wire conveying an intense enough current melts when it attains a white heat. Auer has observed that if the wire be coated with $ThO_2$, the intensity of the current may be raised considerably without melting the wire, as the coating radiates part of the energy as light and heat: even when the Pt core has melted, the current can be further increased, until finally the $ThO_2$ coating is ruptured by the Pt vapours. The most successful results are given with a thoria film of only a few tenths of a millimetre in thickness. The other rare earths, CaO, MgO, &c., melt or volatilise at temperatures too low for the above purpose. If the Pt be replaced by an alloy of Pt with Os, Ru, &c., or by Os itself, the filament obtained is able to sustain a temperature, and consequently a luminosity, hitherto unattained; and, as long as the core is not melted, it is practicable to break current without injury to the filament.—H. B.

*Natural Gas, Estimation of Hydrogen Sulphide in.* F. C. Phillips. J. Amer. Chem. Soc. 1898, 20, [9], 678.

*See under* XXIII., *page* 1073.

## PATENTS.

*Gas Producers, Impts. in or relating to.* J. Dymond, Crediton. Eng. Pat. 22,066, Sept. 25, 1897.

Relates to gas-producers in which air is induced to flow into the fuel within the gas producing chamber through the agency of one or more jets of steam, and the invention claimed, consists in supplying the air, in a heated state, to the jet or jets of inducing steam, whereby condensation of the latter is prevented, and gas of a more uniform quality is obtained. The gas-producer is provided with a jacket or casing arranged to form, in conjunction with the wall of the producer, a chamber or space that is in communication, through a suitable opening, with the external atmosphere, and in which is located one or more steam ejector nozzles and one or more air induction pipes for conveying a mixed current or currents of air and steam to the interior of the gas-producer. The air is drawn in from the atmosphere, and flows on in contact with the walls of the heated producer chamber before reaching the steam nozzle or nozzles.—R. S.

*Fuel, An Improved.* J. Hopwood, Wrexham, and C. Cummins, Dublin. Eng. Pat. 27,360, Nov. 22, 1897.

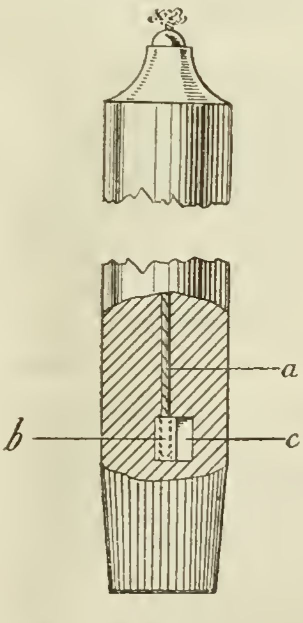
The fuel is made of a mixture of 80 per cent. of a base, such as peat, coal-dust or sawdust, 6 per cent. of astatki oil or crude petroleum, 1 per cent. of lime, and 13 per cent. of a mixture, prepared while heating, of alum and linseed oil.—R. S.

*Coke, Impts. in the Manufacture of, and in Apparatus therefor.* J. Bowing, Tilbury. Eng. Pat. 27,869 Nov. 26, 1897.

To allow an iron or steel retort to be used for coking, it is heated by radiation instead of by direct contact with the furnace gases. The construction is thus claimed. "A coke oven wherein a vertical retort of iron or steel is surrounded by a chamber or muffle, the wall forming which, is in turn surrounded by a shallow flue of uniform width at all points, and having a throat or throats through which the furnace gases enter the said flue, the area or united sectional areas of the said throat or throats measured at right angles to the direction of flow of the gases being the same or substantially the same as the sectional area of the flue around the retort measured in the same manner." The invention further consists "in the manufacture of coke in the manner described in Eng. Pat. 2071, 1897," "conveying the products of distillation which escape from the retort during the coking operation into a closed condenser, which is preferably one of the bunkers in which coal for future treatment is stored."—R. S.

*Candles, Manufacture of; Impts. in the.* N. A. Alexanderson, Stockholm. Eng. Pat. 7671, March 30, 1898.

In this invention, which has reference to self-extinguishing candles, that portion, *b*, of the wick, *a*, which has been rendered self-extinguishing, is surrounded with a cavity, *c*, in which the fluid stearin or other illuminant which cannot be absorbed by the wick, is collected, instead of running over the edge of the candle, as usually happens.—C. A. M.



*Washing and Scrubbing Gas, Impts. in Apparatus for.* Kirkham, Hulett, and Chandler, Ltd., and S. Hersey, Bridge Street, Westminster. Eng. Pat. 25,019, Oct. 28, 1897.

A washer-scrubber of the ordinary kind is used, but it is so arranged that one or more of the bays is by-passed by the washing water, and these bays contain a number of communicating tubes, through which the water for washing the gas is passed before entering the inlet to the apparatus, thus cooling the gas and becoming itself warmed. By cooling the gas, the elimination of ammonia is facilitated; also by using warm water in the bay in which the last washing of the gas takes place, the deposit of naphthalene is diminished.—R. S.

*Rendering the Sides or Walls of Gas Reservoirs, Pipe Conduits, and the like Gas-tight; Process for.* K. E. Rosenthal and J. Billwiller, Berlin. Eng. Pat. 16,043, July 22, 1898.

A SUITABLE metallic salt, such as ferric chloride, is dissolved in alcohol, and the sides of gas reservoirs, gas pipes, and the like, are impregnated with the solution. The alcohol evaporates and leaves the salt in a finely-divided state in the pores of the material to be made gas-tight, in which condition the salt is acted upon by the ammonia and hydrogen sulphide contained in the gas, so as to form products of increased bulk, namely, in this case, iron sulphide and ammonium chloride. The alcoholic solution of ferric chloride may be mixed with tar, and the mixture employed as described. "If the tightening effect is to be brought about quickly, the gas reservoirs, pipe conduits, or the like, prepared with the impregnating body, are charged, previously to being used, with a mixture of ammonia and hydrogen sulphide, and kept for some days under pressure." —E. S.

*Bunsen Burners, Impts. relating to.* H. S. Bishop and C. Salmon, Erith. Eng. Pat. 21,790, Sept. 23, 1897.

THE invention consists of a group of gas nipples, mounted on a common reservoir, and each supporting a Bunsen tube, delivering the gas into an upper combustion chamber common to all, this upper chamber being separated from the tubes by insulation material, to diminish the conduction of heat down to the tubes themselves. When required for lighting, the burner is surmounted by a mantle of the usual character, supported by a spring to reduce vibration.—C. S.

*Gas for Motive Power, Illuminating, Heating, and other Purposes; Impts. in and relating to the Generation of.* F. S. D. Scott, Clements Lane, and T. Hawkins, Barking Road. Eng. Pat. 21,833, Sept. 23, 1897.

THE generating vessel is provided with a perforated false bottom, upon which iron shavings, borings or turnings, or zinc, are placed. A horizontal plate entirely separates the vessel into two parts, the waste liquor from beneath the false bottom being forced into the upper chamber through a communicating pipe. An overhead supply tank has three compartments, or there may be three separate reservoirs, which are charged with water and glycerin, sulphuric acid and potassium bichromate, and "benzoline" or petroleum spirit respectively. These liquids are led, in the proportion desired, through transparent drip chambers and a pipe coiled in the upper part of the generator, to a distributing rose in the lower part. The generated gas passes out through a condenser, and may be employed for any suitable purpose, for example, in a gas engine. In this latter case, carbon and zinc plates may be immersed in the exhausted liquor in the upper part of the generator, to produce a current to obtain the spark for firing the gas.—R. S.

*Combustible Gases, Impts. in Compound Bodies or Substances for Producing Automatic Ignition of.* L. Pierron, Brussels. Eng. Pat. 15,411, March 9, 1898. (Internat. Convention.)

THE inventor claims the combination of compound bodies with a base of platinum, or of some metal of the same group, as the "igniting element" with a "supporting element," consisting of infusible, non-volatile, mutually inert substances unalterable in air—such as the pure oxides of "silicium," zirconium, titanium, tungsten, molybdenum, tantalum, vanadium, aluminium, iron, manganese, &c.—employed in a porous or finely divided state, with the object of preventing the igniting element from agglomeration. The finished preparation may take the form of a powder, by simple admixture and calcination; a paste, by incorporation with some agglutinant material; a liquid, by solution; or a tissue or thread, by imbibition or coating.—C. S.

*Gas from the Combination of the Products of Combustion, Exhaust Steam, and a certain amount of Atmospheric Air, and returning the same to the Firebox for the further Support and the Increase of Combustion therein; Impts. in Method of and Apparatus for Forming.* D. Clegg, Philadelphia (Pa.), U.S.A. Eng. Pat. 13,255, June 14, 1898.

THE principal features of this invention consist in the division of the smoke box of a boiler into three compartments by vertical partitions, the central one containing the waste-steam pipe, lateral branches of which, with downward nozzles, cause the current of the products of combustion ascending in the central compartment to be returned to the firebox *via* the outside compartments and a conduit into which the latter debouch underneath. At the same time air is drawn in through apertures in the front of the smoke stack, and also through louvres in the exterior compartments, and is mixed with the gases and steam. In the case of locomotives this indraught is effected by the forward motion of the engine, but in stationary engines a fan is required.

It is stated that, by the aid of a deflector plate and baffle plates in the upper portion of the smoke stack, "a compound action takes place, in which the exhaust steam and certain portions of the products of combustion, as well as the inflowing air, are united, whilst the inert and non-combustible gases are caused to pass upward, and are expelled" through the mouth of the stack.—C. S.

*Production of Combustible Gases by Means of Gas Generators, Impts. in the.* L. Mond, Regent's Park. Eng. Pat. 17,001, Aug. 6, 1898.

THE invention claimed, consists in adding "a certain quantity of gases containing carbon dioxide to the air, more or less charged with water vapour at ordinary or at a moderate temperature, as generally used for the gasification of coal in gas generators, so as to obtain from a given quantity of coal a larger yield of combustible gases, and to maintain the gas generator at a suitable temperature." —R. S.

*Incandescent Gas Lighting [by Water- and Producer-Gas], Impts. in Gas Burners suitable for Use in, and for other Purposes.* J. Dymond, Crediton. Eng. Pat. 22,065, Sept. 25, 1897.

THE first claim is: "A gas-burner so constructed that ordinary producer- or water-gas can, without enrichment by hydrocarbons, be burnt, in connection with a mantle or its equivalent, for incandescence gas lighting, as set forth." It is stated that the ordinary burners cannot be used with water-gas or producer gas, because the cross sectional area of their gas-inlet nozzles is too small, their air-inlets admit an insufficient amount of air, and the air-inlets are placed below the gas-inlet nozzles. Five forms of the improved burner are illustrated, in which relatively wide gas nozzles are used (about $\frac{3}{16}$ in. diameter), and about twice as much air is admitted as in the ordinary burners, the burners consisting essentially of "a vertical tube made in parts arranged one above the other, with an (annular) air-way between their adjacent ends, and a gas-exit nozzle or orifice arranged to admit gas to the lower part of the tube at a point below the said air-way." The air-ways may be surrounded by concentrically arranged shields. There are four claims.—H. H.

*Incandescent Mantles for Lighting, Impts. in and relating to.* A. Martini, Berlin. Eng. Pat. 26,202, Nov. 10, 1897.

THERE is claimed "the production and use of an incandescent mantle impregnated in a solution of thorium nitrate in a quantity less than $97\frac{1}{2}$ per cent., and small quantities of any one of the rare earths in combination with any one or more of compounds of potassium, sodium, lithium, rubidium, cæsium, and tin . . . ." The salts may all be dissolved together, the fabric impregnated as usual, and then burnt off, a suitable mixture for this process being $1$—$2\frac{1}{2}$ per cent. of cerium nitrate, $1\frac{1}{2}$—$3$ of potassium nitrate, and $95$—$97\frac{1}{2}$ of thorium nitrate. According to another modification, the fabric may be impregnated with a solution of $97\frac{1}{2}$ parts of thorium nitrate *plus* $2\frac{1}{2}$ parts of $KNO_3$, and then burnt off, and the mantle next dipped into cerium nitrate solution and dried. Instead of an aqueous solution of cerium nitrate, it is preferable to use a solution of caoutchouc, gutta-percha, and wax (in chloroform, ether, benzene, or suitable solvent), to which cerium nitrate, mixed with alcohol, has been added. When tin is to be used, a high percentage is to be avoided; "if the percentage amounts to more than 20 of the total weight, the mantle becomes unfit for use." Instead of the impregnation process, Chardonnet's collodion process may be used. There are six claims. —H. B.

*Incandescence Gas Burners, Impts. in.* J. Imray, London. From O. Kern, Paris. Eng. Pat. 26,064, Nov. 9, 1897.

An improvement on Eng. Pat. 294 of 1897 (this Journal, 1898, 566), in which a burner-tube was described, consisting of two truncated cones forming a tube with a constricted part about the middle point of its height. According to the present specification, the burner top which surmounts the above-mentioned tube comprises a taper casing, which has a mean diameter about twice that of the upper mouth of the tube, and a height of about three-fourths of the total height of the tube. Within the casing is a taper shell of thin metal, perforated with narrow, approximately vertical slits, and having at its top a diameter about equal to that of the upper mouth of the tube. On the top of the shell is a metal boss, carrying a horizontal disc, notched so as to resemble a toothed wheel, the teeth nearly extending across the annular space between the head of the boss and the mouth of the taper casing. The taper of the casing and shell may be varied. The gas burns above the notched disc. The burner arranged as described, is claimed. —H. B.

*Incandescent Gas Lights, Impts. in Inverted Regenerative.* H. A. Kent, Bounds Green, N. Eng. Pat. 20,823, Sept. 10, 1897.

An improvement on Eng. Pat. 3667 of 1897 (this Journal, 1898, 37), showing a form of lamp resembling the "Wenham" gas lamp. The gas-supply pipe passes down the centre of the upper part of the lamp, up which the hot products of combustion are flowing; it then bends off to the side, to a nozzle having an adjacent air inlet. The stream of gas and air passes from this point, through a short tube, to a central inverted burner, and burns within an inverted incandescence mantle. A hemispherical or other suitable globe surrounds the mantle. There are three claims, the second being "the production of an incandescent gas light or burner, having no side tubes, for giving a down light free from shadows."—H. B.

*Incandescent Oil Lamps, Impts. in and relating to.* P. J. Fitzgerald, Boston, U.S.A. Eng. Pat. 6316, March 15, 1898.

The object of the invention is to provide a lamp which, when the mantle gets destroyed, is capable of giving a luminous flame by means of an auxiliary device. The lamp comprises (1) a vaporising burner, in which the liquid hydrocarbon is vaporised, and from which the bulk of the vapours produced, can be directed into the incandescent burner whilst a small quantity is allowed to burn on the vaporiser in order to maintain the requisite heat; by means of a needle-valve the whole of the combustible vapours produced can be caused to burn on this burner with a luminous flame, in the event of the incandescent burner becoming inoperative; (2) an incandescent burner of the Bunsen type, in which the vapours mentioned, burn beneath a mantle as usual. When starting the burner, the vaporising burner receives a preliminary heating by igniting some spirit in a cup attached to the lower part of the apparatus. A vaporising burner of "the plate type" is illustrated and "is preferred by the inventor to other forms; but he does not confine himself to such type, as any character of burner which furnishes an illuminating flame and provides the heat for the proper vaporisation of the liquid hydrocarbon will answer his purpose." A burner combining such a vaporising burner and an incandescent burner is claimed.—H. B.

*Incandescent Oil Lamps, Impts. in.* J. and M. Hersey, Wood Green, London, N. Eng. Pat. 13,126, June 11, 1898.

The lamp claimed by the patentees is a circular-wick lamp, surmounted by a simple spreader carried by a central spindle, the wick-tube being surrounded by a conical collar and a metal casing; the casing and gallery, of the usual type, are perforated to admit the air supplies, of which one passes centrally up to the interior of the mantle, and two pass to the outside of the mantle.—H. B.

*Petroleum Burner, adaptable both for Blue Flame and Illuminating Flame; An Improved.* M. Graetz, Berlin. Eng. Pat. 26,683, Nov. 15, 1897.

The claim put forward in this specification is for "a petroleum-burner, characterised by the interior of the wick tube, being provided with a body to prevent an excessive inlet of air to the inner side of the wick, whereby a free burning of the flame on the inner surface of the wick can take place, in combination with a sieve-like disc arranged on the outer side of the wick" under a cap, "said disc retarding the air entering from the outside, in such a manner that the said air currents reach the wick with no excessive speed and without being especially directed to the said wick, whereby a bright flame is also produced on the outer side or edge of the wick."—C. S.

*Liquid Hydrocarbon Lamps producing the Bunsen Flame for Heating and Lighting Purposes, Impts. in.* F. E. Suddard, St. Cloud, France. Eng. Pat. 23,661, Oct. 14, 1897.

The heating flame is produced by igniting the vapour, given off from the interior surface of a hollow column of wick, at a point low down in the column, the said flame being fed by an adjustable central current of air, and prevented from ascending above the wick by a metal disc surmounting the column. A transverse partition below the flame contains a central aperture, covered by gauze and limiting the base of the flame so that "there arises between the flame and the wick an annular space which prevents the charring of the wick, the overheating of the vapour sustaining the Bunsen flame, and the wasteful consumption by the flame of much combustible." The vapour driven off from the outer surface of the wick by the heat of the flame becomes mixed with air in the enclosing cylinder, at the top of which is mounted a mantle of the usual kind for incandescent lighting.—C. S.

*Carbide of Calcium, and Apparatus therefor; Impts. in the Process for the Manufacture of.* [*Briquettes.*] G. Bower, St. Neots. From J. E. Hewes, Philadelphia, U.S.A. Eng. Pat. 1895, A. Jan. 25, 1898.

The materials required for the production of calcium carbide are mixed with suitable binding agents—such as sugar, asphalt, molasses, tar, pitch, or an equivalent carbonaceous material—in the required proportions, and are then moulded into briquettes, pastilles, or cakes, which facilitate the escape of gas, and prevent the driving off of particles of one or more of the ingredients when they are subjected to the action of the electric arc.—G. H. R.

*Electrical Furnaces specially applicable for the Production of Calcium Carbide, Impts. in and relating to.* G. W. B. Crees, W. L. Durban, and J. Gore, Trowbridge. Eng. Pat. 26,880, Nov. 17, 1897.

In the improved electrical furnace the two opposite sides or ends, which are mounted on screwed rods, contain, or themselves constitute, the two opposite electrodes, and their distance apart can be regulated by a hand-wheel or equivalent means.—G. H. R.

*Acetylene Gas, Impts. in Means for Using Calcium Carbide for the purpose of Generating.* G. Dillberg, Sydney, N.S.W. Eng. Pat. 15,214, July 11, 1898.

Calcium carbide is placed in an envelope of porous material, such as canvas, and the envelope is wholly immersed in water; or a porous division is placed above the carbide in a vessel containing water. The object of the porous material is to retain the film with which the bubbles of gas as generated are coated. The gas is said to be purified thereby, and the life of burners consuming it considerably prolonged.—J. A. B.

*Calcium Carbide, Impts. in and connected with the Manufacture of.* J. E. T. Woods, Stockwell, and W. A. Byrom, City of London. Eng. Pat. 11,453, June 30, 1898.

Gas, consisting of acetylene, marsh gas, and hydrogen, is produced by alternately reducing by petroleum vapour and oxidising by steam, hydrated oxide of iron contained in a retort heated to about 1,000° F. The gas is used to feed blowpipe burners, three of which converge on a crucible into which a mixture of lime and carbon is fed, and heat it to a temperature of from 4,000° to 5,000° F., and converting thereby the mixture into carbide of calcium. Each burner consists of a number of inclined gas tubes, each provided with an internal tube supplied with air under pressure.
—J. A. B.

*Acetylene, Impts. in Apparatus for Generating.* F. Gastaldi, Turin, Italy. Eng. Pat. 11,723, July 4, 1898.

Two generators, each consisting of a hollow cylinder, containing a cylinder the upper part of which is perforated, and within which is a spiked grid on which carbide is supported,—are fed with water from the tank of a regulating gasholder, into which the gas generated passes. One generator is brought into action automatically as soon as the other is exhausted. The feed of water is controlled through suitable mechanism by the movements of the gasholder. Cooling and condensing arrangements are provided.
—J. A. B.

*Acetylene Gas, Impts. in Generators for the Production of.* J. Billwiller and K. E. Rosenthal, both of Berlin. Eng. Pat. 14,050, June 24, 1898.

Within a water reservoir is placed a receptacle the sides of which are of porous material, such as clay. The receptacle contains carbide, to which water gains access by permeating the porous material, and the gas generated, passes away by a suitable exit pipe. The pressure of the gas within the receptacle, regulates the rate at which the water permeates its walls, which may be given any suitable form or shape.
—J. A. B.

*Acetylene Gas Generators, Impts. in.* The British Acetylene Gas Generator Co., Ld., and H. K. Spence, both of Kirkcaldy, N.B. Eng. Pat. 14,432, June 30, 1898.

Improvements are claimed on the generator described in Eng. Pat. 29,554 of 1896 (this Journal, 1897, 721), consisting of a mechanical device for diverting the supply of water from an exhausted generating chamber to a full one, and of an indicator showing which chamber is in action.
—J. A. B.

*Acetylene Gas Generators for Lamps, Impts. in and relating to.* J. Bartlett, South Tottenham. Eng. Pat. 11,910, May 25, 1898.

The generator consists of an inverted conical vessel placed above and communicating with a conical vessel, in the base of which is a screw plug, which is closed after the vessels have been filled with water. There are perforations in the walls of the vessels, by which the water percolates through a packing material to carbide which is enclosed in the space between the vessels and an enveloping cylinder, in which are perforations through which the generated gas passes to an outside case, and thence through a superposed purifying chamber to the burner.—J. A. B.

*Acetylene, Impts. in Apparatus for Generating.* C. Bolé, Manchester. From P. Dreske, Berlin. Eng. Pat. 11,964, May 26, 1898.

Carbide contained in a hopper is delivered through a rotating perforated disc, which forms the base of the hopper, to a horizontal conveyor, by which it is delivered on to a distributing cone, whence it falls into water in the generator. The disc and conveyor are rotated by a motor, and the speed of rotation is governed by the movements of the bell of the gasholder, into which the gas passes from the generator. The governing device consists either of a friction disc and pulley, or of a fast-and-loose pulley driven by a belt which is shifted through suitable mechanism operated by the gasholder. The motor runs at uniform speed.—J. A. B.

*Acetylene Gas Flames, Means or Apparatus for Purifying the Products of Combustion of.* J. S. Billwiller, Untereggen, Switzerland. Eng. Pat. 28,003, Nov. 27, 1897.

A chimney is placed over the burner, and is divided by horizontal strainers into several compartments, of which the lower ones are empty and serve as condensation chambers, on the walls of which phosphoric acid settles out from the products of combustion, which pass through the chimney. The compartments above these are packed with an absorbent impregnated with a basic material, such as potassium carbonate, which neutralises any acids in the products of combustion. A narrow-meshed sieve is placed at the top to effect further condensation and retain particles of carbon.
—J. A. B.

*Acetylene Gas, Impts. in or relating to the Production of.* W. W. Strode and G. H. White, both of London. Eng. Pat. 11,161, May 16, 1898.

Water in a closed vessel on the top of which is a hopper for carbide, is maintained at a constant level. The carbide is admitted through a valve, which may be closed when the external hopper is being re-charged to a second hopper within the vessel. The lower end of this hopper forms a shoot which embouches within a rotary conical chamber, which revolves on a spindle driven by a water-wheel within the vessel. The carbide drops from the conical chamber into the water. The water used for driving the wheel replenishes that in the vessel, which is provided with an overflow pipe to prevent the level rising above the normal.
—J. A. B.

*Acetylene Gas, Impts. in Apparatus employed in the Manufacture of.* O. V. Sigurdsson, Hammersmith. Eng. Pat. 23,351, Oct. 11, 1897.

A hopper containing carbide in powder is placed on the bell of a generating chamber, with which it communicates by a feeding chamber provided with a valve of such construction that when it is slightly open to the generating chamber it is also open to the carbide hopper. The valve is operated through a lever and stud according to the movements of the bell of the generating chamber. The generating chamber contains water, and has a conical bottom, and a device for flushing out the residue which collects thereupon.—J. A. B.

*Acetylene Lamps, Impts. in.* T. Holliday, Huddersfield. Eng. Pat. 24,360, Oct. 21, 1897.

A reticulate cage which retains undecomposed carbide, but allows lime to pass through it, is placed below a water-holder, from which water can pass through a valve which is actuated from the exterior to the carbide in the cage. The feed of water is further controlled by a cotton conductor, which conducts the water from the valve to the carbide in smaller portions than a drop at a time. Between the cage and the water-holder is a gas-container, into which the gas passes as generated, and from whence it proceeds to the adjacent burner. This chamber is provided with a safety-valve for relieving excessive pressure. The lamp is intended for use on vehicles, the jolting of which will detach the lime from the undecomposed carbide.
—J. A. B.

*Acetylene Gas, Impts. in and connected with Apparatus for the Generation and Combustion of.* J. A. Ageron and L. P. Wirth, both of Paris. Eng. Pat. 22,618, Oct. 2, 1897.

The generating apparatus consists of a water-tank with conical base and a cock for the removal of sludge, also a small bell gasholder and a carbide distributer placed side by side on the top of the tank. The carbide distributer comprises a closed vessel which is filled with carbide through an opening, which is then closed by a screw plug, and a feeding cone, the mouth of which is opened or closed according to the movements of the gasholder through the medium of suitable levers and a rod attached to the bell of the gasholder. The carbide falls from the cone when the mouth of the latter is open into the water in the tank, and the gas evolved passes into the adjacent gasholder, and thence to the burners. Modifications of the carbide-distributing device are described. Burners in which a supply of oxygen or ozone is delivered a little distance below the acetylene flame and mixes with the air round the flame, are described in several forms. They are stated to afford more complete combustion of the gas and to give a more brilliant flame than occurs in air. A pressure-regulator, consisting of a chamber into which the gas enters, the base of which is thereby pushed downwards to an extent depending on the pressure of the gas, and, by means of a rod, controls the tap on the inlet pipe, is also described.—J. A. B.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

*Petroleum Industry, The Russian.* P. Stevens. Bd. of Trade J., Sept. 1898, 271—280.

In the *Ilsky* oilfield 25 wells are in operation, 10 are being drilled, and 8 are under repair. About 250 men are employed, and the total output is between 625,000 and 750,000 barrels per annum. Two qualities of crude oil are produced, the specific gravity being 0·910 and 0·960 respectively; and the refining is carried on at Novorossisk, the products being "kerosene, benzine, and mazut." Owing to the high density of the crude oil and the difficulty of freeing it from the accompanying water, this field is not likely to become very remunerative, unless petroleum rises very considerably in price.

The *Grosni* field is only imperfectly developed, on account of the high royalties demanded for the right of exploitation, and only 12 wells were in work, more or less irregularly, in 1897, the aggregate output being about 4,380,000 barrels during the year. Twenty-four new wells were being drilled. At present there are four pipe-lines (two 5-in. and two 8-in. pipes) leading to the railway (eight miles away), and capable of forwarding 60,000 barrels per diem.

The most important consideration, however, affecting the utilisation of Grosni crude oil is the high proportion of petroleum spirit to burning oil, and the extreme viscosity of the residuum, so that when distilled for burning oil the remainder is too thick to run through a pipe-line, and is also ill adapted for fuel. To combat this defect only 10 to 15 per cent. of the petroleum spirit and burning oil are removed, the residuum then having a density of 0·915 and a flashing point of 60°—80° C., and being nearly equal to Baku residuum as fuel. This treatment increases the cost, and since the burning oil, though of good quality when fresh, quickly deteriorated on storage, the output of the Grosni field will be limited to supplying fuel for home consumption.

At the *Baku* oil region there were, on Jan. 1, 1898, the following wells of all descriptions:—517 at Balakhany; 631 at Sabounchi; 171 at Romany; and 73 at Bibi-Eibat.

Sixty-three firms were engaged in 1897, in the manufacture of burning oil, the aggregate output being 455,000,000 galls., of which about 320,000,000 galls. were produced in 11 refineries. Owing to the unprofitable rates for refined as compared with crude oil, the number of refiners has now decreased.

The total production of crude oil in 1897 was 55,375,000 barrels, the great bulk of which was obtained from the 670 pumping wells in operation. The shipments amounted to, burning oil ("kerosene"), 11,287,500 barrels; lubricating oil, 1,137,500 barrels; residuum, 27,712,500 barrels; crude oil, 2,962,500 barrels; total, 43,100,000 barrels. The large increase in production is mainly due to the "increasing demand and better prices paid for the raw material," but the heavy stocks now held, rather foreshadow a decline in the value of crude oil.

Storage receptacles in the Baku oilfields number 861, with a total capacity of 1,086,087 barrels; and the refineries possess 1,588 reservoirs, capable of accommodating 3,171,027 barrels. The carrying capacity of the 23 pipe lines is about 60,000,000 barrels per annum.—C. S.

*Petroleum, Refining with Silicate of Soda.* J. R. Michler. Eng. and Mining J. 1898, **66**, [12], 338.

The use of the so-called chloro-chromic acid in place of 66 per cent. sulphuric acid, as is recommended by A. M. Villon for refining the distillates of the heavy oils (spindle- to cylinder oils), is stated to give indifferent results. On neutralising the oils, after treatment with acid, by caustic soda or carbonate of soda, it has been frequently noticed that they will either clarify badly or else not at all. The author recommends the use of silicate of soda (water glass), either alone or in conjunction with sodium hydrate or carbonate, and states that the advantages of this method are as follows:—A smaller percentage of sulphuric acid may be used for heavy oils; although dark oils are obtained thereby, the "decolorising" property of the separated silicic acid soon changes them into pale oils. The emulsion deposits quickly, this being effected by the silicic acid in the act of settling down.—A. S.

*Petroleum, Inclusion in Quartz Crystals.* C. L. Reese. J. Amer. Chem. Soc. **20**, [10], 795—797.

Photographs are given of two quartz crystals from Guntersville, Marshall Co., Alabama, with cavities containing globules which show the yellow-green fluorescence of petroleum. The largest globule measures 2·3 mm. (longest diameter) by 1·8 mm. (shortest diameter), and is about 1 mm. thick. The cavities are for the most part triangular, with sides and edges parallel to the rhombohedral faces of the crystal. Some smaller crystals from the same source were crushed in filter-paper, which then showed grease spots, smelt of petroleum, and burned with a smoky flame.—J. T. D.

*Gas Oil, Phenol and Cresol, Pyrogenetic Decomposition of.* E. Müller. J. Prakt. Chem. 1898, **58**, [13, 14, and 15], 1—50.

After reviewing in detail the researches of Watson Smith, Lewes, Kramers, Hofmann, and others, on the chemical changes taking place in subjecting gas oil, phenol and cresol, to a prolonged red heat, the author describes his own experiments, which were carried out with the object of ascertaining whether corresponding results are obtained when phenol is gasified *per se* without the simultaneous presence of higher aliphatic hydrocarbons. The gas oil used was a sample of lignite tar oil from which the greater part of the phenol had been extracted in the works from which it was procured. The remainder was removed by the author, after which the oil had a density of 0·881 at 17°·5 C., and a viscosity of 1·68 at 20° C. (Engler). It had a dark reddish-brown colour, and gave on distillation the following results:—

| | ° C. |
|---|---|
| Began to distil at.................. | 220 |
| 38 per cent. at ..................... | 300 |
| 50 ,, ..................... | 300—360 |
| 8 ,, ..................... | 360—380 |
| 4 per cent. residue and loss. | |

Five chief experiments were made and the results are embodied in the table on next page.

In attempting to explain the results of these experiments, the author states that one or more hydrogen atoms of the benzene molecule are replaced by molecules of a more complex type, in consequence of which an enfeeblement of the aromatic character of the benzene molecule is brought about, for which the phenols are in a high degree responsible, the divalent oxygen contained therein contributing to the formation of carbonic oxide. The benzene nucleus in phenol is the only source of carbon. When this is split up with simultaneous abstraction of a carbon atom, the complex $C_5H_5$ remains, and this must necessarily undergo essential changes before it can be made to assume more stable forms. The chief products in this connection are carbon and hydrogen, whilst the formation of olefines, methane, and the like, takes place only to a limited extent.

Cresol behaves like phenol, the methyl group being split up into methane with assumption of hydrogen, whilst the

| All Weights and Measures are calculated on 100 Grms. of Original Substance. | Gas Oil. | | Phenol. | | Cresol. | | Mixtures of Gas Oil and Phenol (equal parts). | | Mixtures of Gas Oil and Cresol (equal parts). | | Oil of Turpentine, Fraction Boiling at 155°—160° C. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Material gasified per minute. | 0·5 grm. | | 0·44 grm. | | 0·20 grm. | | 0·34 grm. | | 0·52 grm. | | 0·23 grm. | |
| Evolution of gas per minute. | 240 c.c. | | 282 c.c. | | 132 c.c. | | 132 c.c. | | 240 c.c. | | 126 c.c. | |
| Volume of gas obtained at 0° and 760 mm. | 44,680 litres. | | 64,808 litres. | | 60,300 litres. | | 39,750 litres. | | 46,001 litres. | | 60,010 litres. | |
| Weight of dry gas per litre. | 1·012 grm. | | 0·5618 grm. | | 0·5989 grm. | | 0·7361 grm. | | 0·8151 grm. | | 0·6291 grm. | |
| Composition of the gas calculated on gas free from air: | Per Cent. by Volume. | Weight in Grms. | Per Cent. by Volume. | Weight in Grms. | Per Cent. by Volume. | Weight in Grms. | Per Cent. by Volume | Weight in Grms. | Per Cent. by Volume. | Weight in Grms. | Per Cent. by Volume. | Weight in Grms. |
| Ethylene............ | 39·6 | 27·6 | 1·3 | 1·05 | 1·5 | 1·1 | 14·9 | 9·7 | 16·9 | 10·8 | 8·9 | 6·73 |
| Carbonic oxide ..... | 1·7 | 0·9 | 30·8 | 24·9 | 29·2 | 21·9 | 14·7 | 7·3 | 16·9 | 9·6 | 3·2 | 2·46 |
| Hydrogen............ | 9·6 | 0·3 | 58·9 | 3·4 | 50·2 | 2·7 | 35·3 | 1·2 | 25·9 | 1·0 | 32·4 | 1·74 |
| Methane............. | 49·1 | 15·2 | 9·0 | 4·1 | 19·1 | 8·2 | 35·1 | 9·6 | 40·3 | 12·9 | 55·5 | 23·77 |
| | 100·0 | 44·0 | 100·0 | 33·45 | 100·0 | 33·9 | 100·0 | 27·8 | 100·0 | 34·3 | 100·0 | 34·70 |
| Total distillate at 120° C. | 12·78 grms. | | 7·8 grms. | | 16·6 grms. | | 8·0 grms. | | 17·0 grms. | | 15·0 grms. | |
| Specific gravity of the same. | 0·891 | | 0·910 | | 0·899 | | 0·910 | | 0·903 | | 0·888 | |
| Total benzene............ | 5·84 grms. | | 4·37 grms. | | 10·51 grms. | | 5·17 grms. | | 8·5 grms. | | 6·02 grms. | |
| Yield of products: | | | | | | | | | | | | |
| Gas.................. | 44·0 per cent. | | 33·4 per cent. | | 33·90 per cent. | | 27·80 per cent. | | 34·30 per cent. | | 34·70 per cent. | |
| Separated carbon ..... | .. | | 29·0 " | | 28·30 " | | 1·90 " | | 0·40 " | | 1·40 " | |
| Tar and paraffin oil absorption. | 51·5 per cent. | | 27·9 " | | 31·70 " | | 63·70 " | | 58·80 " | | 51·90 " | |
| Loss.................. | 4·5 .. | | 9·7 " | | 6·10 " | | 6·00 " | | 6·50 " | | 12·00 " | |
| | 100·0 per cent. | | 100·0 per cent. | | 100·00 per cent. | | 100·00 per cent. | | 100·00 per cent. | | 100·00 per cent. | |

oxygen combines with one of the carbon atoms of the ring, and is eliminated as carbonic oxide. These data refer to the decomposition of pure substances, the separation of much carbon (as much as 50 per cent.) being a characteristic feature. With mixtures containing gas-oil no carbon is separated, although carbonic oxide and hydrogen are found in considerable quantity.

Generally, from the results obtained, it is inferred that the equations—

$$C_6H_5OH = CO + 6H + 5C$$
$$C_6H_4(OH)CH_3 = CO + CH_4 + 4H + 5C$$

are applicable to phenol or cresol, either alone or when diluted with gas oil, with the exception that in the latter case the residue of the phenol or cresol molecule ($C_5H_n$) and ($C_6H_n$) is not split up into carbon and hydrogen, but unites with the products of the decomposition of gas oil, forming more complex hydrocarbons. It is known that gas oil alone yields 27 per cent. of olefines, and only 20 per cent. when mixed with phenol or cresol.

The theory attributing the presence of carbonic oxide and hydrogen to an intermediate reaction, in which water is formed, and this then decomposed by red hot carbon, thus—

$$C_6H_6O = 6C + 4H + H_2O$$
$$H_2O + C = CO + H_2$$

is untenable, for the reason that the quantity of water found in the tar and paraffin oil was exceedingly small, and that the change represented by the second equation cannot be effected under 1,100° C.; whereas the author's experiments were conducted at about 750°, and at this temperature the proportion of carbonic oxide to carbonic anhydride should have been 1 : 4.

The formation of benzene from phenol and gas oil is said to be due to a secondary reaction, owing to the circumstance that the yield was found to vary between 3 and 6 per cent. Cresol, however, gave a minimum yield of 10 per cent. The author explains this difference by supposing that one of the cresols is capable of reuniting (by virtue of its methyl group) the aromatic ring, split up by the severance of the carbonic oxide group, for which purpose orthocresol would be the most suitable, and next to it the para-compound.

The presence of methane is of some interest, as its yield is closely connected with the methyl groups contained in the gaseous products. Haber has shown that in pyrogenetic resolutions, the side chains (methyl groups) are split off—a circumstance which explains the small quantity of methane obtained from phenol (3 to 4 per cent. of the total weight originally used) and the comparatively large amount yielded by cresol (8 per cent.). By carrying this view further and applying it to oil of turpentine, which the author subjected to destructive distillation incidentally with the above experiments, results were obtained which clearly prove that the more methyl groups a liquid contains in its molecule in an easily separable position, the greater is the amount of methane found in the gaseous products.

The yield of hydrocarbons of high molecular weight isolated from the tar by precipitation with petroleum spirit, although small, affords proof of the fact that the formation depends upon the amount of phenol or cresol contained in the original material; thus with pure phenol or cresol 2 per cent. was obtained, with phenol and gas oil 4 per cent., and with gas oil alone 6 per cent.

With regard to the composition of the gases, ethane was found in all experiments made with gas oil. Its formation appears to be due to a process of "cracking" rather than "building up," a circumstance which is in full accord with Haber's view regarding the course pursued by aliphatic substances when destructively distilled.

In ascertaining the value of gas oil, the percentage of cresol as will now be seen, forms an important factor, as it dilutes the gas with non-luminous products.—D. B.

## PATENTS.

*Distillation, Refining, Chemical Manufacturing Processes, and the like; Impts. in or relating to the Maintenance and Regulation of Heat in.* H. Higgins, 4, Trinity Street, Cambridge. Eng. Pat. 23,054, Oct. 7, 1897.

In the extraction of coal-tar products, distilling, or refining processes, or the like, the vapours of toluene or xylene, or other liquids, semi-liquids, or solids, which vaporise at a temperature above the boiling point of water, are employed as the heating media. The medium is vaporised in a suitable generator, which may also comprise a superheater, and the vapours are led into an air-jacketed chamber containing the retort, or through a coil in a chamber containing the substances to be treated. The vapours may be directly condensed and returned to the feed-tank of the generator, or they may be further used, at a lower temperature, in subsidiary apparatus, before condensation.—R. A.

*Petroleum, Process for Purifying Caucasian.* L. v. J. Jarocki and T. Kiezorowski, Warsaw. Eng. Pat. 13,881, June 22, 1898.

To remove the impurities (about 1½ per cent.) in commercial Caucasian petroleum, which has been refined with sulphuric acid and water, the inventors claim a process of treating the oil in an agitator with 3 per cent. of a solution of 1—2 parts of caustic soda and 10—20 parts of "soda" in 100 parts of water, followed by 6 per cent. of water, the whole being left for the resulting emulsion to dissipate, whereupon the purified oil is drawn off.—C. S.

## IV.—COLOURING MATTERS AND DYES.

*Indigo, Natural and Artificial.* Dr. Fürth. Färber-Zeit. 1898, **9**, [19], 297—299.

As substitutes for Indigo, Indoine Blue (B.A.S.F.), Naphthindone (Cassella), Diaminogen Blue (Cassella), Diaminazo Blue (Cassella), Diazo Blue (Bayer), Oxamine Blue (B.A.S.F.), Diazo Indigo Blue (Bayer), and Zambesi Blue (Berlin), have all been used, and have the advantage over Indigo of not requiring special apparatus, or the use of the expensive hydrosulphite vat. Comparatively little success, however, has attended their introduction.

According to the author, large scale experiments show that when everything is taken into consideration, Indigo pure (B.A.S.F.) is quite as good as natural Indigo. In dyeing with the natural product, considerable loss results during the subsequent treatment with acids—some say 30—40 per cent. If this indigo be recovered from the acid washings and again used, as it contains most of the impurities of the natural Indigo (Indigo Red, &c.), bright shades cannot be obtained therefrom. Since artificial Indigo is free from admixture with these substances, the recovered Indigo is pure, and gives when again used, results equal to the original colouring matter. Taking this into account, it is thought that artificial Indigo may be more cheaply employed than the natural product.—H. I.

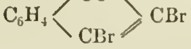
*Gambierfluorescein and Gambier-Catechu-Red.* Karl Dieterich. Chem. Centr. 1897, **2**, [1], 50—51; from Ber. Pharm. Ges. **7**, 153—161.

*Gambierfluorescein* is prepared by moistening 5 grms. of gambier-catechu with water, decomposing with 20 per cent. caustic soda solution, and shaking with benzene or ether, which dissolves, not only the fluorescein, but also two other substances, the one of an oily and the other of a waxy nature. Gambierfluorescein is purified by treating with sulphuric acid, decomposing with caustic soda, and extracting with ether. Its solution becomes red in contact with the air, and on evaporation yields a red tar similar to a phlobaphen; this the author names gambier-catechu-red. The fresh alcoholic solution of gambierfluorescein is green by reflected and yellow by transmitted light. Gambierfluorescein, which contains no nitrogen, is insoluble in water and alkalis, but dissolves in acids; the solutions are not fluorescent. It crystallises in white needles from light petroleum spirit, but on drying, the crystals turn red and partly lose their crystalline structure. It is contained in the catechu, either in the form of a catechin or of a catechutannic acid compound, or in both forms, the compound, which must be analogous in composition to a salt, becoming saponified in the process of extraction. Pegu-catechu does not contain this substance, and the main difference between these catechus is that whilst gambier-catechu-red is not contained as such in gambier-catechu, pegu-catechu-red is actually contained in pegu-catechu.

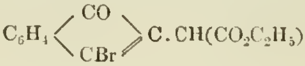
*Gambier-catechu-red* is a reddish-brown resinous powder which floats on water, becomes electrified by friction, melts at 130°—131°, is insoluble in ether, forms a blood red solution in sulphuric acid, but neither yields fluorescent solutions nor possesses the basic character of gambierfluorescein. If a faintly acid solution in alcohol be neutralised with alcoholic potash, an intensely dark blue fluorescent solution is produced, similar to that of tincture of turmeric, but the colour disappears on adding more alkali or on warming. Gambier-catechu-red does not give a precipitate with tannic acid. The acetyl number shows that it still contains hydroxyl groups. The fatty oil obtained from the catechu by means of benzene has an acid number = 14·89, ether number = 43·33, and a saponification number = 58·22; the iodine number is very low.—A. S.

*Cochineal Dyestuffs.—III.* C. Liebermann. Ber. **31**, [13], 2079—2084.

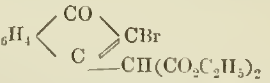
In continuing his previous work (Ber. **30**, 1731; this Journal, 1897, 794), the author comes to the conclusion, in view also of v. Miller and Rohde's work on α-bromocarmine in which they identify it as methyloxytetrabromodiketohydrindene, that cochineal is an indene derivative. A number of derivatives of carminic acid have been prepared, from which it appears probable that the formula of the acid is $C_{22}H_{22}O_{13}$. A characteristic property of the acid is the ease with which it condenses to insoluble dyestuffs, behaving thus analogously to ketoindene derivatives. In connection herewith the author also finds that dibromindone—



easily condenses with malonic ether, acetoacetic ether, &c., in presence of alcoholic sodium alcoholate, to coloured products, which, in an alkaline solution, possess great similarity with alkaline solutions of carminic acid. Similar colour reactions have been observed by Roser and Haseloff in the case of dichlorindone and methylindene carboxylic acid. On condensing dibromindone and malonic ethyl ether by means of alcoholic sodium alcoholate, the resulting product forms light yellow plates melting at 130° C., and having probably one of the following formulæ:—



or



It gives with aqueous or alcoholic potash or with baryta water a purple coloration, but does not dye wool or mordants. The mother-liquor contains another substance which appears to be formed by the condensation of two molecular proportions of dibromindone and one of malonic ether. Bromindone acetoacetic ethyl ether, $C_{15}H_{13}BrO_4$, prepared similarly to the above compound, forms small yellow granular crystalline aggregates melting at 82° C., and gives purple-red alkaline solutions. The benzoyl acetic ether compound was not obtained sufficiently pure for analysis, whilst the methyl and benzylacetoacetic ethers give with alcoholic dibromindone and sodium alcoholate colour reactions which pass very quickly through blue and green, and the products could not be isolated. Dyestuffs similar to those above described were not obtainable from α- and β-bromocarmine, or from Roser's oxybromindone.

—T. A. L.

*Azo Compounds, Electrical Synthesis of Mixed.* W. Löb. Ber. **31**, [13], 2201—2205.

According to Kauffmann and Hof, the electrolytic reduction of *m*-nitrobenzaldehyde yields *m*-azobenzoic acid, together with a small quantity of *m*-azobenzyl alcohol. On repeating this work the author found that the azo acid formed, consisted to a very small extent of *m*-azobenzoic acid, and mainly of a mixed azo compound having the formula $HO_2C.C_6H_4.N:N.C_6H_4.CH_2OH$. The present investigation was an outcome of this observation. The process described, consists in the simultaneous reduction of the components of the mixed azo compound in the form of their nitro compounds, in a suitable alkaline medium. The conditions require to be varied for each particular case, but no details as to strength of current or solution, &c. are given in the paper. It has not been found possible to reduce nitrophenols to azophenols, so that mixed oxyazo compounds have not been obtained by this method. The use of lead as the electrode in an alkaline medium, easily leads to the formation of hydrazo compounds, and it is therefore preferable to employ platinum or mercury. The following mixed azo compounds have been prepared:—*m.m*-azobenzoic acid benzyl alcohol, $HO_2C.C_6H_4.N:N.C_6H_4.CH_2.OH$, obtained by reducing *m*-nitrobenzaldehyde, forms yellow plates from alcohol melting at 183° C., easily soluble in ether, insoluble in petroleum ether and water. The simultaneous reduction of *p*-nitrotoluene and *m*-nitrobenzoic acid yields azo-*p*-toluene-*m*-benzoic acid—

$$H_3C.C_6H_4.N:N.C_6H_4.CO_2H,$$

yellow plates from alcohol melting at 192° C., insoluble in water. The *m.m*-sulpho-azobenzoic acid—

$$HO_3S.C_6H_4.N:N.C_6H_4.CO_2H$$

from nitrobenzene sulphonic acid, and nitrobenzoic acid, crystallises from alcohol in yellow plates. Its properties are intermediate between those of *m*-azobenzoic acid and *m*-azobenzene sulphonic acid, being very easily soluble in water and alcohol (difference from *m*-azobenzoic acid), whilst it is insoluble in ether, in which it differs from *m*-azobenzene sulphonic acid. It dissolves in sulphuric acid. All the salts are yellow, and, with the exception of the lead salt, are easily soluble in water. The reduction of *p*-nitraniline and *m*-nitrobenzoic acid yields azo-*p*-amidobenzene-*m*-benzoic acid—

$$H_2N.C_6H_4.N:N.C_6H_4.CO_2H,$$

which forms reddish-yellow needles; the salts are of an intense yellow colour. A sulphonic acid of this substance is obtained by reducing *p*-nitraniline sulphonic acid and *m*-nitrobenzoic acid. By electrolysing *m*-nitrobenzoic acid and *p*-nitrodimethylaniline (obtained by dissolving dimethylaniline in 10 times its weight of acetic acid, and adding the calculated amount of concentrated nitric acid), an azo-*p*-dimethylamidobenzene-*m*-benzoic acid—

$$(CH_3)_2N.C_6H_4.N:N.C_6H_4.CO_2H$$

was produced, which Griess had already obtained (Ber. **10**, 52) by combining diazotised *m*-amidobenzoic acid with dimethylaniline. Finally, the author has also prepared a hydrocarbon mixed azo compound from nitrobenzene and *o*-nitrotoluene. The product is identical with that obtained by P. Jacobsen (Ber. **28**, 2541) by the action of solid caustic soda on a mixture of nitrobenzene and *o*-toluidine.

—T. A. L.

*Aromatic Azomethine Compounds.* R. Mohlau. Ber. **31**, [13], 2250—2261.

The well crystallised products obtained by the action of aromatic aldehydes on primary aromatic amines possess well marked coloured properties, due to the presence of the chromophor, the azomethine group $-N:CH-$ connecting two aromatic nuclei. Theoretically they may be considered to be derived from the corresponding azo compounds by substituting the methine group $-CH:$ for $.N:$

| | |
|---|---|
| $C_6H_5.N:N.C_6H_5$ | $C_6H_5.CH:N.C_6H_5$ |
| Azobenzene (orange-yellow). | Benzylidene aniline (light yellow). |

Whilst, however, the azo compounds are highly coloured bodies and their derivatives are well-defined dyestuffs, the corresponding azomethine derivatives of the benzylidene aniline type are much less highly coloured and their derivatives, capable of forming salts although undoubtedly dyestuffs, are not of practical utility, one notable drawback being that they are very easily decomposed into their components, aldehyde and amido base, by the action of mineral acids, or in some cases even by water. Attention was first called to the dyestuff character of these purely aromatic azomethine compounds by Weil in 1894 (Ber. **27**, 3317; this Journal, 1895, 475), who found that dimethyl-*p*-amidobenzaldehyde reacted to form intense colorations or precipitates of benzylidene compounds, with primary aromatic amines, not only in neutral, but also in mineral acid solutions. Azo and azomethine compounds being analogously constituted, they can be classified in the same way, and thus the azo and disazo dyestuffs, the amido-azo and oxyazo compounds, and the azo dyestuffs from carboxylic acids have corresponding representatives in the azomethine series. Those already prepared are derivable from two chromogens, both light yellow, the one being anhydrobenzaldehyde aniline (benzylidene aniline)—

$$C_6H_5.CH:N.C_6H_5,$$

and the other disanhydrobenzaldeyde-*p*-phenylenediamine—

$$C_6H_5.CH:N.C_6H_4.N:CH.C_6H_5.$$

by the replacement of nuclear hydrogen by auxochromic groups. The author has prepared a number of the compounds representative of the basic phenolic and carboxylic azomethine derivatives, in order to compare their similarity physically with those of the azo series. Corresponding to *p*-amido-azobenzene is *p*-amidobenzylidene aniline, $H_2N.C_6H_4.CH:N.C_6H_5$, obtained by the action of *p*-amidobenzaldehyde on aniline. It crystallises in yellow plates or needles, and its hydrochloride forms reddish yellow needles, with a bluish-violet reflex, dissolving to a yellow solution, whilst amido-azobenzene hydrochloride dissolves to a salmon-red. The compound—

$$[4.1](CH_3)_2N.C_6H_4.CH:N.C_6H_4.N(CH_3)_2[1.4]$$

was obtained by condensing the tin double salt of *p*-amidodimethylaniline with dimethyl-*p*-amidobenzaldehyde, or else by mixing together nitrosodimethylaniline hydrochloride (100 grms.), dimethylaniline (50 grms.), concentrated hydrochloric acid (300 grms.), and paraformaldehyde (50 grms.). According to the first method, the mixture immediately turns orange-red, and on adding sodium acetate the tin double salt of the condensation product separates as a red precipitate. The base liberated by ammonia crystallises from alcohol in yellow plates melting at 230° C. The hydrochloride, $C_{17}H_{21}N_3 2HCl$, forms shining rhombic plates or prisms. Its solution dyes tannined cotton brownish-red. On boiling the aqueous solution, it decomposes into dimethylamidobenzaldehyde and *p*-amidodimethylaniline, whilst reduction of the hydrochloride solution with zinc dust yields dimethyl-*p*-toluidine and *p*-amidodimethylaniline. For the preparation of disanhydro-dimethyl-*p*-amidobenzaldehyde-*p*-phenylene diamine, $C_6H_4[N:CH.C_6H_4.N(CH_3)_2]_2$, 10 grms. of paraformaldehyde are dissolved in 150 grms. of hydrochloric acid (sp. gr. 1·17), 50 grms. of dimethylaniline are added, and then, quickly, 100 grms. of powdered nitrosodimethylaniline hydrochloride. At 70° C. a vigorous reaction sets in, and on cooling the pure hydrochloride of the colour base separates out. It can be washed with cold water, and recrystallised from water containing hydrochloric acid. The salt has the formula $C_{24}H_{26}N_4.2HCl, 5H_2O$, and forms brownish-red needles with a steel-blue reflex. It dyes tannined cotton reddish-brown, and when reduced with zinc dust in a hydrochloric acid solution, it yields dimethyl-*p*-toluidine and *p*-phenylene diamine. The same product can also be obtained by mixing aqueous solutions of 6 grms. of dimethyl-*p*-amidobenzaldehyde with the necessary quantity of hydrochloric acid and 3·6 grms. of *p*-phenylene diamine hydrochloride. The hydrochloride separates as a scarlet precipitate, from which the base is obtained by means of ammonia. After recrystallisation from dimethylaniline and washing with alcohol, this melts at 278° C. The homologous diethyl compound melts at 207°·5 C. In the preparation of phenolic azomethine compounds, the author condensed *p*-nitrobenzaldehyde with 1.4-, 1.2-, and 2.1-amidonaphthol, and obtained in each case well-crystallised products. The anhydro-*p*-nitrobenzaldehyde 1-amido-4-naphthol is obtained by dissolving 2 grms. of *p*-nitrobenzaldehyde and 3 grms. of sodium acetate in 50 c.c. of alcohol (95 per cent.), and adding a hot solution of 5 grms. of 1.4-amidonaphthol hydrochloride in 50 c.c. of water. The liquid turns deep red, and the dyestuff separates in ponceau-coloured crystals, which separate from alcohol in shining golden-scarlet plates melting at 171° C. The product dissolves easily, with a cherry-red colour in cold dilute soda lye, whilst the alcoholic solution is coloured deep violet by caustic soda. *p*-Nitrobenzene-azo-α-naphthol, on the other hand, dissolves in soda lye with a dark violet colour, and its alcoholic solution is coloured dark green by caustic soda. The azomethine derivative dissolves in concentrated sulphuric acid with a brownish-yellow colour, the liquid on dilution becoming colourless as the compound is decomposed into its components. The derivative analogous to Paranitraniline Red, obtained by condensing *p*-nitrobenzaldehyde with 1.2-amidonaphthol, forms shining prisms from alcohol, melting at 174° C. It is somewhat less soluble than the previous isomeride in cold dilute soda lye, and its alcoholic solution is coloured violet by caustic soda. Paranitraniline Red, insoluble in aqueous soda lye, also gives a violet sodium salt in alcohol, but whilst the solution of the anhydro compound in concentrated sulphuric acid is dark orange, that of the azo compound is reddish-violet. The condensation product of *p*-nitrobenzaldehyde and 2.1-amidonaphthol hydrochloride crystallises from alcohol in reddish-orange matted needles melting at 187° C. This substance is almost insoluble in cold dilute caustic soda lye, the alcoholic solution being coloured violet by caustic soda. The colour in concentrated sulphuric acid is brownish-red, becoming colourless on dilution. It would thus appear that the well-marked difference between the *o*- and *p*-oxyazo compounds in regard to their behaviour to alkalis is also shown to a certain extent by analogously constituted members of the azomethine series. With regard to azomethine derivatives of carboxylic acids, the author combined *o*-, *m*-, and *p*-nitrobenzaldehyde with 1.4.3-amidosalicylic acid, obtaining products melting at 221°, 252°, and 218° C., and gives their reactions with concentrated sulphuric acid, caustic soda, and carbonate, compared with the corresponding azo compounds, of which two are technical products, being strong and fast mordant dyestuffs, *viz.*, *p*-nitrobenzene-azo-salicylic acid (Alizarin Yellow R) and *m*-nitrobenzene-azo-salicylic acid (Alizarin Yellow G G). The comparison shows that the colours with which the neutral sodium salts of the azo compounds dissolve, are the same as those of the basic salts of the corresponding azomethine compounds, confirming the view that the azo group is a stronger chromophor than the azomethine group.—T. A. L.

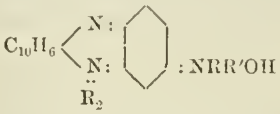
## PATENTS.

*Aromatic Aldehydes and of Colouring Matters therefrom, Manufacture or Production of.* I. Levinstein and Levinstein Ltd., Manchester. Eng. Pat. 21,968, Sept. 25, 1897.

In Eng. Pat. 19,904 of 1897 (this Journal, 1898, 914) a process is described for the preparation of *p*-nitrobenzaldehyde *o*-sulphonic acid by oxidising dinitrostilbene disulphonic acid with potassium permanganate. It is now found that this process is generally applicable to stilbene derivatives. Thus, *o*-sulphobenzaldehyde may be obtained by carefully oxidising stilbene *o*-disulphonic acid with potassium permanganate in the cold, as in the following example. A solution of 28·4 kilos. of sodium stilbene *o*-disulphonate (obtained by removing the amido groups from diamidostilbene disulphonic acid) in 800 litres of water, is mixed with vigorous agitation with 21 kilos. of potassium permanganate in 500 litres of water, the temperature being kept at about 5° C. and a current of carbonic acid being passed through the liquid. When the oxidation is complete, the manganese dioxide is filtered off, and the filtrate is used directly in the production of dyestuffs, or is evaporated to dryness, and the sodium *o*-benzaldehyde sulphonate is extracted with alcohol, from which solution it separates as a white crystalline powder readily soluble in water. When condensed with ethyl benzylaniline sulphonic acid in a dilute sulphuric acid solution, and oxidised with lead peroxide, a dyestuff is produced which gives a pure blue shade on animal fibres from an acid bath, and is said to be bright and fast to alkalis. (See also Eng. Pat. 21,825 of 1897; this Journal, 1898, 915.)—T. A. L.

*Colouring Matters of the Safranine Series, The Manufacture of New.* C. D. Abel, London. From The Actiengesellschaft für Anilinfabrikation, Berlin, Germany. Eng. Pat. 24,410, October 21, 1897.

According to Fischer and Hepp (Ber. **29**, 361 and 2,752; this Journal, 1896, 536, and 1897, 132), by heating aposafranine and isorosindulines with aniline and aniline hydrochloride, an aniline radicle enters the benzene nucleus ortho to the imido radicle. If, in place of isorosinduline, a compound derived from a tertiary amine, such as Neutral Blue, be similarly treated, no substitution takes place. When, however, primary amines are brought into reaction with Neutral Blue, &c., in absence of mineral acids and in presence of oxygen, substitution takes place in the naphthalene nucleus, symmetrical substitution products of isorosindulines being formed. The isorosindulines and their substitution products suitable for this process have the general formula—



whilst the amide compounds employed may be ammonia' amines, or diamines, their nitro, hydroxy, sulphonic, or carboxylic derivatives, amidoazo dyestuffs, amido dyestuffs of the triphenylmethane, acridine, or induline series, &c. For example, (1) 20 kilos. of isorosinduline carbonate, from nitrosodimethylaniline, and ethyl β-naphthylamine, 100 kilos. of alcohol, and 50 kilos. of 10 per cent. ammonia, are heated in an autoclave for six hours to 150°—160° C. The alcohol is distilled from the bluish-red solution, and the dyestuff is separated as the hydrochloride or zinc chloride double salt. It dyes tannin-mordanted cotton bluish-red shades, and dissolves with a bluish-green colour in concentrated sulphuric acid.

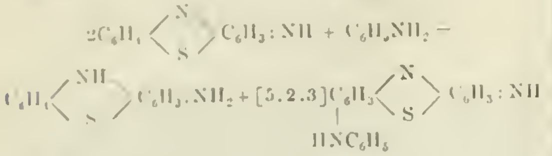
(2) A solution of 4 kilos. of Neutral Blue and 3·5 kilos. of Magenta in 80 kilos. of alcohol is mixed with 2·5 kilos. of soda lye, and a current of air free from carbonic acid is passed through the mixture at 25°—30° C. The dyestuff so formed is dissolved in water made acid with hydrochloric acid and precipitated with salt. It dyes tannined cotton a bluish-violet. If Phosphine be substituted for the Magenta, a greyish-black dyestuff is formed. In cases wherein an insoluble product is obtained, this may be rendered soluble by sulphonation.—T. A. L.

*Para- and Meta-nitroanisidine, and of Colouring Matters [Red] therefrom, Manufacture of.* O. Imray, London. From The Fabriques de Produits Chimiques de Thann et Mulhouse, Thann, Alsace. Eng. Pat. 25,756, Nov. 5, 1897.

If *o*-acetanisidine be nitrated, a mixture of *p*- and *m*-nitroacetanisidines is obtained in varying proportions, according to the manner in which the nitration is performed. On hydrolysis these acetyl derivatives yield a mixture of nitroanisidines. By a choice of suitable conditions, it is possible to obtain *m*-nitroanisidine only, but not the para derivative. The latter is separated from the mixture by taking advantage of its weak basic properties. 20 kilos of *o*-acetanisidine are gradually added to a mixture of 120 kilos. of nitric acid (38° B.) and 100 kilos. of glacial acetic acid in the cold. The nitration is complete in a few minutes, when the melt is poured into water. The product consists of about 66 per cent. of *p*- and 34 per cent. of *m*-nitroacetanisidine. By gradually adding 12 kilos. of acetanisidine to 200 kilos. of nitric acid (41° B.) at 25—40° C., and pouring into water, a mixture of 75 per cent. of para and 25 per cent. of meta is obtained. Pure *m*-nitroacetanisidine is formed by dissolving 10 kilos. of acetanisidine in a mixture of 40 kilos. of sulphuric acid (66° B.) and 20 kilos. of glacial acetic acid, cooling to 0° C. and running in 8·4 kilos. of nitric acid (40° B.), mixed with 16·8 kilos. of sulphuric acid (66° B.), and pouring into water. The nitroacetanisidines obtained according to either of these processes are hydrolysed with acid or alkali. In order to separate the two isomers, one part of the mixture is dissolved by the aid of heat in 8 parts of sulphuric acid (25 per cent.). Subsequently 10 parts of water are added, and the whole is allowed to stand for a short time, when the para derivative separates out, the meta remaining in the liquor being precipitated by an alkali. Both products form yellow needles insoluble in water, soluble in alcohol, ether, and other organic solvents. The *p*-derivative melts at 140° C., the meta at 118° C Both yield diazo derivatives, and give azo compounds with the usual components. Combined with β-naphthol on cloth goods, reds and pinks are obtained.—T. A. L.

*Colouring Matters [Blue], Manufacture of New.* C. D. Abel, London. From The Actiengesellschaft für Anilinfabrikation, Berlin, Germany. Eng. Pat. 27,051, Nov. 18, 1897.

By reacting with primary, secondary, and tertiary amines on imidothiodiphenylimide, violet-blue to blue dyestuffs are produced, suitable for silk, wool, and mordanted cotton, fast to light, acids, and soap. The reaction in the case of aniline takes place according to the following equation—



The leuco derivative of imidothiodiphenylimide, which is formed as an intermediate product, is immediately oxidised by the air to the imide, which then again reacts. In place of imidothiodiphenylimide, its leuco derivative, *p*-amidothiodiphenylamine, may be used. As an example for carrying out this invention, 1 kilo. of the zinc chloride double salt of imidothiodiphenylimide is stirred into 10 kilos. of aniline. When no further thickening takes place, the dyestuff is filter-pressed, washed with dilute hydrochloric acid, and dried. It gives violet-blue shades on silk, wool, and mordanted cotton, fast to light, acids, and soap, from a bath acid with acetic acid. In the case of primary and secondary amines, the reaction takes place readily in the cold, whilst with tertiary amines it proceeds a little more slowly. The dyestuffs form dark blue or brown crystalline powders, soluble in hot water, alcohol, and acetic acid, with a blue colour. The free bases liberated by caustic alkali or ammonia are all more or less soluble in water, with a violet colour. On shaking these solutions with ether, the latter turns magenta-red. The solutions in concentrated sulphuric acid are dull green, which turns bluish-green or blue on dilution. Colourless leuco derivatives are obtained by adding stannous chloride.
—T. A. L.

*Mordant Dyestuffs [Red, Violet] from Anthraflavic and Isoanthraflavic Acids, Manufacture of.* O. Imray, London. From The Farbwerke vorm. Meister, Lucius und Brüning, Hoechst a M., Germany. Eng. Pat. 6533, March 17, 1898.

The specification relates to the preparation of two new isomeric tetrahydroxyanthraquinone disulphonic acids from anthraflavic and iso-anthraflavic acids. These acids, on sulphonation and nitration, yield dinitro-anthraflavin disulphonic acid and dinitro-iso-anthraflavin disulphonic acid, and, on reduction, the corresponding amido-sulphonic acids. The amidohydroxy-anthraquinone disulphonic acids, when gently oxidised in a neutral alkaline or acid solution, are converted into two isomeric tetrahydroxyanthraquinone disulphonic acids. With regard to the quantities employed, 10 kilos. of anthraflavic acid are stirred into 80—100 kilos. of fuming sulphuric acid (10—20 per cent. $SO_3$) and heated to 110—120° C. until soluble in water. The melt is then cooled and 2 molecules of nitric acid, preferably as nitration acid, are run in whilst cooling, the whole being finally heated to 40—60° C. to complete the nitration, when it is poured into water and can be separated as the well crystallised potassium salt. In order to reduce it, the sulphuric acid melt as above obtained is diluted with water, and the calculated amount of stannous chloride or other suitable reducing agent is added. The solution turns bluish-red, and the diamido-anthraflavine disulphonic acid is best precipitated with potassium chloride. As an example of the oxidation of this substance, it is dissolved in just the requisite amount of hot water, acidified, and mixed with 2 to 3 molecular proportions of lead or manganese peroxide paste. The intense red colour of the solution changes to orange yellow, and from the filtered solution, salt or potassium chloride precipitates the acid salt of tetrahydroxyanthraquinone disulphonic acid as a yellowish-brown crystalline precipitate. The substance is a valuable mordant and acid dyestuff, and its acid colour can be subsequently shaded with metallic salts. Alum produces bluish-red shades, whilst chromium fluoride gives pure violet shades quite fast to milling and light. The product from isoanthraflavic acid differs tinctorially in giving dye lakes of a somewhat redder shade.—T. A. L.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Silk, Double Refraction of.* R. Panebianco. Rivista di Mineralogia e Crystallografia ital. di Panebianco, **15**, 57; Chem. Centr. 1898, **2**, [13], 736.

The author, as opposed to Rossinski and Vignon, has arrived at the following results:—

(1.) Silk fibres are doubly refractive. One line of extinction is parallel to the central axis.

(2.) The colours between crossed Nicols are very brilliant.

(3.) The fibroin fibres have two axes, with small optical axial inclination.

(4.) Sericin is doubly refractive, but further investigation is not possible. A thin section of "seripterium" shows, like the fibroin, two axes, and the plane of the axes parallel to the striæ of the section.

Sericin shows brilliant colours in concentric rings; it has two axes, and the plane of the axes is parallel to that of the fibroin.—A. S.

*Cellulose, The Technical Application of.* M. Kitschelt. Färber-Zeit. **9**, [17], 261—263.

This is a sketch of the history of cellulose and materials made from substances containing cellulose, as, for example, paper; their various applications and modifications, up to the present time.—J. E. H.

### PATENTS.

*Woollen Fabrics intended for keeping the Human Body at an Equal Temperature, An Improved Process for Impregnating.* M. Ravizza and R. Schonlin, London. From J. R. Bautz, Munich. Eng. Pat. 21,719, Sept. 22, 1897.

Woollen fabrics or flannel are "boiled" in the following solution for half an hour, namely:—"For each 15 square metres of fabric a solution of 50 grms. of alum, 50 grms. of potash and 50 grms. of salt, in about 40 litres of water."

The fabric is then steeped in cold water for a quarter of an hour, wrung out, dried, and ironed. In this way it is claimed that a fabric is obtained which is unchanged, either by perspiration, or cold or hot water. "It does not shrink, and can dry of itself on the body" without influencing the warmth of the latter.—C. M.

*Fibrous Materials, in the Production of Material to be used for Insulating and other Purposes; Method and Apparatus for Saturating, Vulcanising, and Oxidising.* F. Lamplough, London. Eng. Pat. 25,120, Nov. 2, 1897.

The fibrous material (hemp, cotton, wood fibre, &c.) is placed on trays in a closed tank capable of withstanding heat and pressure. The tank is then filled with the saturating medium, which consists of a mixture of resins or resin oil and fish oil, and the temperature is gradually raised by means of a steam coil in the tank, to slightly above the boiling-point of water. Air and moisture are thus expelled, and escape by a safety tube. The temperature is then further raised until the vulcanising point is reached. This latter must be determined by experiment as the various fibres become vulcanised at different temperatures. The heat is kept constant for some time by the aid of a thermo-regulator on the steam supply. Afterwards the oily fluid is run out into a second tank, and air is pumped into the first tank containing the fibre, under a pressure of a few atmospheres, being constantly changed until the material is oxidised. It is then pressed and dried.
—W. P. S.

*Mercerising Cotton Yarns and Fabrics, An Improved Process of.* L. S. van Westrum, Magdeburg, Germany. Eng. Pat. 12,669, June 6, 1898.

Cotton yarn, in a slightly stretched state, is immersed "in a strong alkaline bath (not less than 32° B.)" for 10 minutes. The lye ("potash or soda") is run off and the frame and yarn placed in a gaseous acid chamber, preferably carbonic acid. The gas impregnates the fibre, and the alkaline salt is formed on the yarn in crusts. This process takes from 10 to 20 minutes. When neutralised, the yarn is thoroughly washed with water, and prepared for the market in the usual way. (See also this Journal, 1898, 149, 240, 345, 452, 573, 756, and 840.)—C. M.

*Vegetable Material, Cables of; Impt. in the Manufacture of.* F. J. Hounay, Marcinelle, and A. Warnant, Namur, Belgium. Eng. Pat. 16,186, July 25, 1898.

"A process for the manufacture of cables of vegetable material, consisting in impregnating the vegetable fibre with a solution of rosin in a suitable solvent."—C. M.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Tannin Compounds in Aqueous Solution on Standing, The Changes in Similar.* P. Sislen. Leipziger Färber- und Zeugdrucker-Zeit. 1898, **47**, [10], 434—435.

It is well known that tannin solutions, on standing, become dark brown. The brown substance is an oxidation product which, up to the present, has not been isolated in a pure condition. The author has studied the formation of this compound under various conditions, and the following are some of his results:—

It is not due to fermentation, because sterilised solutions also become brown on exposure to sterilised air. Dilute solutions oxidise more quickly than do concentrated ones. Heat assists, and also alkalis, whilst acids hinder the formation. Experiments were made in the presence of reducing agents, such as sulphurous acid; but although the tannin solutions became colourless, they reoxidised after a short time. Phosphorous and arsenious acids were the only substances which prevented the formation of the brown compound for any length of time. Tannin extracts contain nitrogenous substances, glucosides, &c., which, if the extract be left, soon set up fermentation. Experiments made with various antiseptics proved that boric acid (1 grm. per litre) is the best; this acid has no detrimental action on most colours, and is recommended for these reasons.—C. M.

*Wool, Mordanting of.* G. Eberle and Fr. Ulffers. Färber-Zeit. 1898, **9**, [19], 295—297.

According to the chemical theory of dyeing, compounds of the wool substance with basic and acid dyestuffs exist (E. Knecht), and a similar explanation has been given to the chemical changes taking place during the mordanting of the wool fibre. The basic character of wool is rendered evident by its behaviour towards such a dyestuff as Ponceau, while it plays the part of an acid towards magenta. Wool can behave both as an acid and basic substance at once; thus, after dyeing with Ponceau, it will take up from a magenta bath the same quantity of the magenta base as the undyed material. In both cases the colour acid and the colour base are chemically united with the substance of the wool.

When mordanting with metallic salts, the same chemical changes take place, *viz.*, union of the wool substance with both the acid and the base of the mordant. On account of the greater simplicity of the chemical changes involved, the authors undertook the examination of the behaviour of wool towards an aluminium, and not a chromium (bichrome), mordant. Aluminium is taken up in the same state of oxidation as it is present in the mordant solution, whereas chromic acid has first of all to be reduced to chromic oxide before it can act as a mordant for acid colours.

When heated in a solution of alum, wool takes up acid from the salt, as is proved by the bath becoming cloudy through the separation of alumina, but alumina is also taken up, proved by the power which the wool has, after treatment, of uniting with adjective dyestuffs.

Experiments were undertaken with a view to studying the influence of the nature of the acid of the aluminium salt used in mordanting.

To this end it was attempted to prepare the aluminium salts by dissolving freshly-precipitated alumina in solutions containing equivalent quantities of a number of different acids, but it was found that constant results could not be obtained. Further, many aluminium salts, especially those of the weaker acids, are unstable and often insoluble; the authors therefore took alum, and added either the free acid, or the acid or normal potassium salt, in equivalent quantity to the solution. Ten grms. of woollen yarn were mordanted for $1\frac{1}{2}$ hours in a bath containing such a solution, and afterwards dyed in a separate bath containing 12 per cent. of logwood extract. In the following table the results obtained with the potassium salts of certain acids and alum as a mordant are shown:—

| The Mordanting Bath contained 1 Equivalent of Alum and 1 Equivalent of the Normal Potassium Salt of— | Shade produced with Logwood Extract. | Relative Strength of Acids as determined by | | 1/100 Gram-Equivalent of Acid in 250 c.c. of Water, dissolved, of Alumina— |
|---|---|---|---|---|
| | | Electric Conductivity. | Inversion of Cane Sugar. | Grms. |
| Hydroxyisobutyric acid .......... | Deepest violet ...................... | 1·2 | 0·010 | 2·004 |
| Lactic " .......... | Deep violet ......................... | 1·0 | 0·013 | 1·884 |
| Glycollic " .......... | " " weaker than above ..... | 1·3 | 0·010 | 1·904 |
| Oxalic " .......... | Dull violet.......................... | 19·7 | 0·186 | 3·455 |
| Malonic " .......... | Clear red-violet .................... | 3·1 | 0·031 | 1·954 |
| Malic " .......... | Medium deep reddish-violet ....... | 1·3 | 0·013 | 0·752 |
| Tartaric " .......... | " " bluish-violet......... | 2·3 | .. | 1·328 |
| Sulphuric " .......... | Bright reddish-violet as obtained with alum alone ............... | 65·0 | 0·536 | .. |
| Ethylsulphuric " .......... | | .. | 1·000 | .. |
| Hydrobromic " .......... | | 100·1 | 0·980 | .. |
| Hydrochloric " .......... | | 100·0 | 1·000 | .. |

From the foregoing table it will be seen that the intensity of the colour produced is inversely proportional to the strength of the acid present in the assistant salt; consequently, the weaker the acid the more the bath is exhausted.

By separate experiments it was further shown that the presence of free acids or of acid salts in the mordanting bath unfavourably influenced the subsequent dyeing, in proportion to their quantities and the relative strengths of the acids contained therein. If weak alkalis or free alumina be added to such a bath this can be corrected.

The conclusion drawn by the authors is, that the intensity of the shade produced when dyeing with logwood on an aluminium mordant, is a function of the acid-strength of the acid of the salt used as an assistant.

Apparent exceptions, such as the use of oxalic acid and cream of tartar when mordanting with potassium bichromate, will be treated of in a subsequent paper.—H. I.

F

*Felt Hats, Dyeing Black.* C. A. Otto. Färber-Zeit. 1898, 9, [19], 293—295. (See also this Journal, 1898, 24[illegible], 453, and 843.)

Previous to the application of the coal-tar colours, hats were dyed black by means of logwood on an iron or chromium mordant.

The author gives a number of recipes, of which the following are a selection:—3 per cent. of bichrome, 2 per cent. of copper sulphate, 2 per cent. of sulphuric acid of 66° B., [illegible] per cent. Cloth Red G (Oehler). Boil 1 hour; next day dye with 75 per cent. Logwood chips and 5 per cent. Old Fustic chips. Boil half an hour, and sadden with 1 per cent. copper sulphate (boiling ½—¾ hour). A good brilliant black is obtained. The Cloth Red prevents its "greening" too quickly.

Or, the hat-bodies may be dyed in a single bath as follows:—75 per cent. Logwood, 5 Old Fustic, 10 copperas, 5 copper sulphate, 3—4 oxalic acid. The dye-woods are first boiled for 1 hour, then the mordants added, the bath cooled to 62° C., when the wetted-out hat-bodies are introduced and the boiling continued for 1½ hours longer. After dyeing, in both cases, the material is well washed with water or with urine. If the hats have been stiffened, only [illegible]—¼ of the dye-woods are used, and the washing is conducted with lukewarm water.

At present artificial dyestuffs are more commonly employed. In dyeing with these it is important not to have the bath too acid, to boil fairly long, and when fixing, to see that sufficient acid is present to decompose all the dyestuff, otherwise when shaping and pressing the colour is apt to rub off. In general, when using acid dyestuffs, great care must be taken when adding the acid to the dye bath, otherwise greenish-white flecks form on the material, which are almost impossible to remove even with ammonia or soda.

The following artificial dyestuffs have been employed, and their method of application is briefly stated:—

*Brilliant Blacks B and E (B.A.S.F.).*—See this Journal, 1898, 213.

For 100 parts of the material in each case take—

*Naphthylamine Black (Cassella).*—5 parts of dyestuff, 4 of oxalic acid, 6 of acetic acid, and 3 of copper sulphate. Enter the wetted-out hat bodies at 38° C., heat slowly to the boil, and maintain at that temperature 1—1½ hours.

*Wool Black (Bayer).*—5 parts of dyestuff, 10 of Glauber's salts, ¼ of Fast Yellow extra, and ¼ of Fast Green (blue shade). Boil ¾ hour; add dilute sulphuric acid carefully until the bath is exhausted.

*Diamond Black F (Bayer).*—5 parts of dyestuff, ¼ of Fast Yellow extra, 10 of Glauber's salts, and 5 of ammonium acetate. Enter at 38° C., boil ½ hour, then add 5 parts of acetic acid, boil 1 hour, take out, turn, and fix with 1½ per cent. of Bichrome.

Or, two-bath method.—Mordant with 3 parts of Bichrome, 2 of cream of tartar, and 1 of sulphuric acid for 1 hour; dye in a bath containing 1,000 litres of water, to which 3 litres of ammonium acetate solution, 3 per cent. of the dyestuff, ¼ per cent. of Alizarin Cyanine 3 G, have been added at 38° C., for ¼ hour. 3—4 per cent. of acetic acid are then added, and the bath is heated to boiling for 1—1½ hours.

*Chromotrope S (M. L. and B.).*—Also gives good results.

*A[illegible] Black G (M. L. and B.).*—5 parts of dyestuff, 10 per cent. of Glauber's salts, 3 per cent. of acetic acid, and [illegible] per cent. of Victoria Yellow. Boil ¼ hour, then add 3—4 per cent. of sulphuric acid.

*[illegible] Patent Black 4 B N (Kalle).*—5 per cent. of dyestuff, 10 of Glauber's salt, 3 of acetic acid, and [illegible] of Fast Yellow. Enter the wetted-out hat bodies, heat to the boil, and then add [illegible] of sulphuric acid and [illegible] of copper sulphate, and boil for ½ hour longer. This black is fast even to dilute soda.

*Palatine Black (B.A.S.F.).*—As the above.

*Wool Deep Black 3 B (Berlin).*—As the above.

*Alizarin Black W R, paste (B.A.S.F.).*—Dyed with the addition of acetic acid, saddened with 1½ per cent. of bichrome, and topped with Alizarin Blue [illegible]. This black is very good, but is little used, as it requires very careful washing; otherwise it is apt to rub off.—H. [illegible]

*Lanacyl Dyestuffs, Dyeing of Wool with.* G. Robrecht. Färber-Zeit. 9, [17], 263—265.

The Lanacyl dyestuffs of Cassella consist of the following five brands:—Lanacyl Blue B B and R, Lanacyl Marine Blue B and 3 B, and Lanacyl Violet B. They can be used for the production of blues on woollen piece goods in a single bath. They dye with difficulty in a neutral bath, slowly in a weak acetic acid bath, and rapidly in a bath containing much acetic acid or sodium bisulphate. The goods to be dyed must be very clean, and it is recommended that they be passed (especially carbonised goods) through a lukewarm bath of ammonia and be well washed, previous to dyeing. The ammonia must be well rinsed out, or the dyeing will proceed slowly, owing to the neutralising of the acetic acid in the dyebath. The general method of dyeing is to fill the vat one-third full with water, add the dyestuff, boil up so as to dissolve it, then to add 15—20 per cent. of Glauber's salt, 5 per cent. of acetic acid, and enough water to fill the vat. Enter the goods, run a few minutes without steam, and turn on the steam so that the liquor boils in ½ hour. Continue boiling for ¾—1 hour, when most of the dyestuff will be absorbed; shut off steam, add a further 5 per cent. of acetic acid which has been well diluted with water, and run 20 to 30 minutes longer; the dyebath will then be completely exhausted.

By this method the goods—even those containing dead fibres—are well covered. For goods which are difficult to penetrate, the author employs 6 per cent. of ammonium acetate (2 parts of concentrated ammonia and 5 parts of acetic acid (30 per cent.)), and proceeds exactly as above, adding Glauber's salt, &c.

The Lanacyl dyestuffs are well adapted for shading in the boiling bath, as also are such level-dyeing acid colours as Cyanol, Fast Acid Green, Patent Blue, Formyl Violet, Fast Acid Violet, Orange, Tartrazin, Tropaeolin, Lanafuchsin, &c.

If the colour of the goods be substantially lighter than is required, the author prefers to use the Lanacyl dyestuffs, on account of their fastness to light. In this case the dyebath must be vigorously cooled. The dyebath can be employed for new lots, since it is completely exhausted. A portion of the liquor (½—⅔) is run off, the dyestuff, previously dissolved, is added, along with 5 per cent. of Glauber's salts, the goods are entered, and the process carried out exactly as for the first lot.

Lanacyl Blue B B and R are very fast to light, being followed in this respect by Lanacyl Violet and Lanacyl Marine Blues.

Lanacyl Marine Blues and Lanacyl Violet are very fast to steaming, whilst Lanacyl Blue B B and R satisfy the usual requirements; the latter marks withstand strong steaming, if, in dyeing, 1—2 per cent. of copper sulphate be added to the dyebath and the goods be allowed to run 15 minutes without boiling.

These dyestuffs are of satisfactory fastness towards rubbing, ironing, perspiration, and alkali (street-mud). They stand only a slight milling, and therefore are generally unsuitable for loose wool. They are not fast to acids, and consequently cannot be used when blue yarns are interwoven with stoved yarns, unless the latter have been previously desulphurised.

A valuable property of these colours is that they leave the cotton borders or the white cotton threads interwoven in a fabric, perfectly white.

For marine and dark blue shades, the author employs Lanacyl Marine Blue B, since it dyes more level than the mark 3 B. Lanacyl Blue B B, in consequence of its clear green tone, is preferred for Saxony Blues; it mixes well with Lanacyl Blue R or Lanacyl Marine Blue B.

Lanacyl Marine Blue is completely fixed, if, after moderate exhaustion of the dyebath, 3—5 per cent. of sodium bisulphate be added (instead of acetic acid) and the goods are allowed to boil strongly for ½ hour. This addition is advisable when using a mixture of this dyestuff and Lanacyl Blue B B.

Olive, brown, and dark green shades are dyed with Lanacyl Blue R and B B or Lanacyl Violet, and a yellow and red of the acid dyestuffs previously mentioned; or Anthracene Yellow C and Wool Red B may be employed, since they only stain very slightly any cotton threads present, which cannot be said of the above acid dyestuffs. Of course, any shading required is done with the acid dyestuffs. The method of dyeing is similar to that employed for blues, except that more sodium bisulphate (up to 10 per cent.) is added.—J. E. H.

*Hydrosulphite Vat, Dyeing with Artificial Indigo in the.* Leipziger Färber- u. Zeugdr.-Zeit. 1898, **47**, [9], 400—402.

According to direct evidence, artificial indigo is applied to wool in a manner similar to that employed in the case of the natural product. Remarkably beautiful and full shades are said to be obtainable both in the fermentation and the hydrosulphite vat.

*The Hydrosulphite Solution* is prepared as usual; the quantities given are, for 100 litres: 135 kilos. of commercial bisulphite solution (38°—40° B.), 70 litres of cold water, 13·5 kilos. of zinc dust, and 50 litres of milk of lime (20 per cent.).

*The Stock Solution of Reduced Indigo.*—Fifty kilos. of indigo paste (20 per cent.), or equivalent amount of the powder, are mixed with 80—100 litres of water at 60°—80° C. Sixty litres of caustic soda solution (25° B.) and 200 litres of the hydrosulphite solution are stirred in, the temperature is maintained at 40°—50° C. for 1—2 hours, when the reduction is complete and the solution has acquired a brownish-yellow colour.

*Wool Dyeing.*—This solution is added to 2,800—3,000 litres of soft water, to which 20 litres of hydrosulphite have been previously added. After standing over night, the vat is ready for use, and when thus prepared contains from 0·3 to 0·333 per cent. of indigo. The dyeing is conducted in the same manner as when ordinary indigo is used with the hydrosulphite vat.

Dark shades are matched by several passages (25—40 minutes), while for light shades only one dip in the vat is given.

After each dyeing operation, additions of hydrosulphite are made, and the stock solution is added only when necessary to maintain the dyeing power of the vat.

Caustic soda gives better results than lime with cotton. Such a vat will run for years if due care be taken. When the dissolved salts greatly accumulate, the vat is used for dyeing light shades, and as the liquor is removed by the goods, fresh water is added in its place.—H. I.

*Waste Liquor from Sulphite Wood-Pulp Manufacture. [Mordanting Wool.]* H. Seidel. Rev. Gén. des Mat. Col. **2**, [22], 370—373.

For the manufacture of 200 quintals of cellulose, 600 quintals of wood shavings, containing 25 per cent. of water, are heated under pressure with a solution of bisulphite of lime containing 36 quintals of sulphur. The residual lye contains the same quantity of sulphur, and 250 quintals of organic matter. It is a brown or yellow liquid of specific gravity about 1·05 and of slightly aromatic odour, and contains 10—12 per cent. of dry substance, which itself yields 12 per cent. of ash and 6—8 per cent. of sulphur. The liquor contains small quantities of sulphuretted hydrogen, sulphurous acid, dextrose, mannose, pentose or pentosan, furfurol, and vanillin, but its most important constituent is a body of the formula $C_{26}H_{30}SO_{12}$, which may be considered probably as a lime salt of a sulpholignic acid. At any rate, the greater part of the sulphur in solution in the lye is organic, and not separable by ordinary precipitants. The substance also contains methoxyl groups, and at least one carboxyl, as shown by its behaviour towards phenylhydrazine. It is soluble in water, very glutinous, and a strong reducing agent. The Waldhof factory, near Mannheim, with a daily output of 1,200 quintals (120,000 kilos.) of cellulose, sends into the Rhine daily about 160,000 litres of this lye, containing 160 quintals of dry substance, 12 quintals of which is organic sulphur. Works not situated near rivers are obliged to evaporate the lye and burn the residue. The author's invention for using these liquors under the name of lignorosin as an assistant to take the place of tartar or lactic acid in mordanting wool with bichromate, has already been noticed (this Journal, 1898, 845 and 863), and has been patented (Eng. Pat. 19,005 of 1897). Some details of laboratory experiments are given in the paper. Experiments in alizarin dyeing on the large scale have shown that 1·5 per cent. of bichromate, in conjunction with lignorosin and sulphuric acid, gives the same shades as 2·5 per cent. of bichromate with tartar, so that the lignorosin effects a saving of 40 per cent. of bichromate. In comparison with lactic acid, which utilises the bichromate quite as well as lignorosin, the latter has the advantage of reducing it more slowly, and is, besides, hardly one-third the price. The shades obtained are full and very bright, and answer all requirements as regards fastness to soap, acids, and light. Some loose wool dyed with alizarin on lignorosin and bichromate mordant, and then milled three hours in acid of 4° B. with white wool, did not bleed in the least on to the white. As a formula especially applicable to loose and spun wool, the author recommends 3 per cent. of lignorosin, 1·5 per cent. of $K_2Cr_2O_7$, and 0·8 per cent. of $H_2SO_4$. Corresponding quantities of the other mordants would be 3 per cent. of tartar and 2·5 per cent. of $K_2Cr_2O_7$, or 3 per cent. of lactic acid with 1·5 per cent. of $K_2Cr_2O_7$ and 0·6 per cent. of $H_2SO_4$. The wool must first be well wetted, entered cold, the bath gradually raised to the boil in half an hour, and the boiling continued for 1—1¼ hours. The wool is drained for half an hour, rinsed, and then dyed in the usual way. Dyed patterns of blacks on cotton accompany the paper. Most of these have been mordanted with 3 per cent. of lignorosin, 3 per cent. of bichromate, and 1·5 per cent. of sulphuric acid. The quantities of the various dyestuffs employed are also given. (See also this Journal, 1898, 923.)—T. A. L.

*Cellulose, Technical Applications of, for Textiles, Paper, &c.* M. Kitschelt. Färber-Zeit. 1898, **12**, [18], 282—284.

The most suitable materials for coating with pegamoid—a body prepared from nitrocellulose, camphor, and a solvent—are moleskin and cretons. They are first washed, scoured, and singed, and then the pegamoid is applied by means of a trowelling arrangement. The goods, in the form of an endless band, are passed through a trowelling machine over rollers into a heated chamber containing horizontal racks and provided with a suitable ventilator and hot-air supply, to remove the alcohol used as a solvent. In this way usually five coloured and two colourless coats of pegamoid are applied, the first two being liberally diluted with alcohol. The pegamoid material only penetrates a little distance into the fabric, the chief deposit being on the surface.

The materials which have been coated with pegamoid lose very little of their softness and flexibility, are waterproof, resistant to dilute acids and alkalis, and are consequently easily cleaned with soap or even with soda. Perhaps the most remarkable property of pegamoid is its resistance to mechanical abrasion—as, for example, a leather imitation cannot be scratched with the finger-nail.

Pegamoid has been applied to the manufacture of army maps, which have proved themselves most resistant to the weather and various ill-treatment in the French army. Bed cloths, aprons, and other articles used in surgery have also been made from pegamoid, and are most serviceable.

The manufacture of imitation leather is of especial importance. When made into purses, cigar cases, bookbinders' leather, army saddles, &c., pegamoid can hardly be distinguished from the real leather goods, is lighter, and costs much less. Allusion is also made in the paper to viscose in its application to the manufacture of horn, ivory, and leather substitutes, and to its more extended use in the printing of textile fabrics for the production of opaline effects.

The writer also briefly notices the attempts which have been made in Sweden to manufacture alcohol from cellulose. —H. I.

*Lactic Acid, Preparation of, for the Dyer and Printer.* C. Dreher. Färber-Zeit. 1898, **12**, [18], 277.

*See under* XX., *page* 1070.

## PATENTS.

*Colouring and Ornamenting Paper and like Materials An Improved Method of and Process for.* S. H., and J. T. Musgrave, London. Eng. Pat. 24,506, Oct. 22, 1897.

A solution of tannic acid and an emulsion of sea moss (preferably Irish moss) are mixed together. The paper is coated with this mixture and then passed through a chamber containing ammonia vapour and quickly dried. The paper thus treated exhibits iridescent effects. If a coloured ground be desired, a suitable colouring material is added to the mixture of tannin and sea moss.—R. B. B.

*Printing Paper-hangings, Calicoes, and the like; Impts. in, and in Apparatus therefor.* T. W. Statter, J. Y. Shorrock, and J. C. Statter, Heywood. Eng. Pat. 25,679, Nov. 5, 1897.

Instead of the blanket roller usually employed, a hard "bed" roller without any covering is used, and, in combination with it, a surface printing roller with a raised pattern composed of india-rubber or other pliable material. This relief roller is furnished with colour by a metal furnishing roller engraved all over with a "pin point" or other similar pattern.—R. B. B.

*Printing or Ornamenting Fabrics, Impts. in.* J. F. Hill and Co., Ltd., and S. Wrigley, Manchester. Eng. Pat. 25,918, Nov. 8, 1897.

The fabric is first mercerised under tension, in order to render it lustrous, and then a design is printed with a dull or opaque substance, such as zinc oxide, china clay, cellulose, &c., either alone or mixed with a carrying agent—*e.g.*, albumin, starch, or oil. The lustrous appearance disappears in the printed portions, and the lustre of the other parts is heightened by contrast.—R. B. B.

*Irisated Prints, Improved Process and Apparatus for the Production of.* J. M. Heppler, Leer, Ost Friesland, Germany. Eng. Pat. 26,058, Nov. 9, 1897.

This refers to a process for enabling irisated prints of any pattern to be produced at will, in tints or shadings of limited width, and gradually blending into one another. The desired result is attained (1) by giving such forms to the ground-colour patterns that the colours do not lie side by side, but fit together with contiguous edges, which are toothed or zigzagged; and (2) by rubbing together the tongues or zigzags of the ground colours transversely to the direction of the gradation or shading. By giving a tapering shape to the projecting parts of the ground colours, a soft and gradual blending of one tint into the other is obtained.—R. B. B.

*Dyeing, An Impt. in.* T. Ingham, Manchester. Eng. Pat. 27,937, Nov. 27, 1897.

This relates to an improvement on Eng. Pat. 1629 of 1897 (this Journal, 1898, 151), and consists in adding to the second or developing bath a small quantity of a coal-tar dyestuff, in order to avoid deterioration of the "bloom," in goods requiring subsequent treatment—*e.g.*, the treatment to which cotton velvets are subjected after dyeing.—R. B. B.

*Colouring or Printing Yarns, Impts. in Methods of and Apparatus for.* C. L. Horack, New York, U.S.A. Eng. Pat. 11,641, May 23, 1898. Date under Internat. Convention, Oct. 29, 1897.

An apparatus for applying bands or stripes of colour to yarn, consisting of yarn drum, colour carriage, and distributing wheel, together with a support for carrying a suitable rubbing surface, and mechanism for moving the support and locking it in various positions, corresponding with the varying operative positions of the rubbing surface. The rubbing surface may be adapted to cover single bands of colour or adjoining portions of two colour bands.—R. B. B.

*Fabric Surfaces, Printing Representations of [on Paper, &c.].* A. J. Boult, London. From A. B. Sherwood, Chicago, U.S.A. Eng. Pat. 18,134, Aug. 23, 1898.

The fabric to be reproduced is fastened on the cylinder or bed of a printing press, and the press is operated to ink the fabric and feed the paper, so that the latter shall receive an impression directly from the inked surface of the fabric. When the fabric is sufficiently coarse, and the paper sufficiently thin, the paper is embossed in addition to being printed with the design. Patterns or designs on, *e.g.*, watered silks, &c., can thus be reproduced on paper surfaces.—R. B. B.

# VII.—ACIDS, ALKALIS, AND SALTS.

*Bauxite in Austria.* R. Helmhacker. Eng. and Mining J. 1898, **66**, [16], 457.

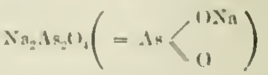
A description is given of two of the deposits of bauxite in Austria. In Carniola, the bauxite occurs near the bank of a small tributary of the river Save, which flows through a small valley in the north-eastern portion of the province. The mineral forms a layer of about 4 m. in thickness at the junction of the Triassic and Jurassic limestones, the outcrop extending for some distance. The specific gravity is 2·55. Analyses of a number of samples taken in 1895, show: $Al_2O_3$, 67·6—51·4; $Fe_2O_3$, 0·7—19·3; $SiO_2$, 5·9—14·4; manganese, lime, magnesia, and titanic acid, traces; and water, 23·1—12·2 per cent.

The deposit of clayey bauxite in Styria, occurs on the northern slope of the Dobrol mountain, and is a superficial one, forming patches thickly interspersed through the clays and sandy clays which rest on a base of Mesozoic limestone. The mineral has a specific gravity of 3·06, and contains: 58·6 per cent. of $Al_2O_3$; 18·7 per cent. of $Fe_2O_3$; and 11 per cent. of $SiO_2$; the balance being water.

No considerable quantities have been mined from either of these deposits.—A. S.

*Sodium Metarsenite, Action of, on Metallic Salts.* C. Reichard. Ber. 1898, **31**, [13], 2163—2171.

All attempts to convert ortho-, pyro-, or metarsenites into the corresponding hydroxides have been unsuccessful, arsenious anhydride and water being invariably produced. With the object of throwing more light on the constitution of these and similar salts, the author has made a number of experiments in different directions. In addition to the varieties of arsenites mentioned above, there is a class of salts corresponding to another hypothetical hydroxide, of which the potassium compound $K_2As_4O_7$ ($=K_2O.2As_2O_3$), discovered by Pasteur (Journ. Pharm. **3**, [13], 395), is an example. As this has marked crystalline properties, the author examined its behaviour with solutions of various metallic salts, in the expectation that the corresponding metallic arsenites would be formed. But in most cases the reaction which occurred was of quite a different character, and was often accompanied by side-reactions. The arsenites which were formed showed considerable variations among themselves when subjected to the same chemical and physical influences (Ber. **27**, 1019; this Journal, 1894, 732). Experiments were then made to determine the reactions which occurred on mixing solutions of the salts of various heavy metals with a representative of another class of alkali arsenites, *viz.*:—



$$Na_2As_2O_4\left(=As\begin{matrix}\diagup ONa\\ \diagdown O\end{matrix}\right)$$

So many kinds of arsenites were formed that the author considered it not improbable that the reactions took place in several stages. In addition to the production of the definite arsenite, free sodium hydroxide was probably formed, which reacted with the metallic salts, forming a metallic hydroxide and a sodium salt, of which the former was often precipitated simultaneously with the arsenite. Of the 12 arsenites prepared, only two (those of cobalt and cadmium) were pyro-arsenites; whilst four (those of zinc, tin (stannous), lead, and nickel) were ortho-arsenites.

Singularly enough, none of the compounds were metarsenites. The action of the sodium metarsenite on the salts of the following different metals is described, *viz.*, cobalt, nickel, manganese, copper, lead, silver, zinc, cadmium, tin, iron, and mercury. Of these, the mercuric salts gave a white precipitate, which settled down as a heavy powder and could readily be separated by decantation $5HgCl_2 + Na_2As_2O_4 + 4H_2O = Hg_5As_2O_8 + 2NaCl + 8HCl$. This salt underwent partial decomposition on exposure to light, turning yellow. When acted upon by alkalis, ammonia, or alkaline carbonates, there was an instantaneous blackening of the mass, with the liberation of mercury and mercurous oxide. On boiling with water, there was a similar partial decomposition, the compound becoming dark grey. On ignition, part of the dry salt sublimed unchanged, whilst the remainder was decomposed, with the formation of arsenious anhydride, mercurous oxide, and metallic mercury.—C. A. M.

### *Alum and Iron Sulphate [Copperas], New Method of Treating Pyritic Lignites, or Shales, for the Production of.* — Geschwind. Rev. Prod. Chim. **1**, [9], 129, 130.

The author finds that the ordinary process of treating pyritic shales or lignites, by leaving them to oxidise spontaneously in heaps, can be accelerated, perfected, and rendered more productive by sprinkling the heaps with sulphuric acid (35°—60° B.), or the waste acid tars from petroleum refineries, the temperature of the mass facilitating the action of the acid on the excess of clay. At the same time, the insoluble basic sulphates formed during oxidation are rendered soluble, so that the final amount of soluble sulphuric acid is greater than the sum of the initial and added quantities.

The acid is preferably distributed over the mass after the second turning, and when the mineral has been piled in large heaps (20 by 20 by 10—12 metres); under these conditions the reaction is completed within about a month.

In extracting the soluble constituents by lixiviation, the author prefers to work on the counter-current principle, applying heat as the solution becomes more and more concentrated, so that finally the water is in a state of saturation. This saves the expense of concentrating by evaporation, the economy thus effected being estimated at 6,350 kilos. of coal per cubic metre of liquor (final volume). The iron sulphate (copperas) is prepared by adding scrap iron, and leaving the liquor to crystallise in the usual manner.

To recover alum from the residual mother liquor, the latter is heated to 90°—100° C., and left to crystallise, after an addition of ammonium or potassium sulphate, the crystals being purified by re-solution to 41°—42° B. strength and re-crystallisation after filtration, care being taken to keep the solution warm, to prevent crystallising in the filter. This process is simplified by the author, by heating the mother liquor to only 60° C., so as to produce small crystals, which are then drained, washed with alum water, and dried in the hydro-extractor, the latter operations being repeated. The product, which is free from iron, is re-melted and cast into blocks.—C. S.

### *Alums, Formation of, by Electrolysis.* Howe and O'Neal. J. Amer. Chem. Soc. **20**, [10], 759—765.

The electrolytic apparatus consisted of a platinum basin or crucible as anode, and a platinum wire as cathode, with a porous pot to serve as diaphragm. The porous pot contained a strongly acid solution of the sulphate (iron, cobalt, manganese) of the metal, and the outer vessel the same solution mixed with the alkali sulphate. Iron ammonium alum was readily obtained in large crystals; iron potassium alum not so well; and no iron sodium alum could be obtained. The iron alums with rubidium and cæsium were both prepared in octohedra of a delicate violet colour; they are very slightly soluble in water. Cobalt-rubidium and cobalt-cæsium alums were also obtained, as minute deep-blue octohedra, stable in dry air but not in moist, and rapidly decomposed by water with evolution of oxygen. Chromium ammonium alum was obtained in small quantity at the cathode when a strongly acid solution of potassium chromate was electrolysed, but the potassium alum was not formed. Attempts to prepare the alums of manganese and the cæsium alum of ruthenium failed.—J. T. D.

### *Copper Sulphate, Direct Preparation of, from Metallic Copper.* E. Sorel. Rev. Prod. Chim. **1**, [10], 146—147.

In place of roasting the copper, or treating it with nitrous or sulphurous compounds before exposure to the action of sulphuric acid, the author finds that the addition of a small quantity of such organic bodies as oxalic acid, alkali oxalates, lactic acid, alkali lactates, tartaric, malic, or citric acid, the corresponding salts, or crude glycerin, facilitates the action of the reagent, without the added substance being itself decomposed or consumed. He proposes to apply this method in practice by charging a vertical vessel with copper turnings, which are then exposed to the action of a continual stream of dilute (1 : 3) sulphuric acid, containing a few grms. per litre of one of the organic substances cited, and kept at 75°—80° C. by steam heat. Provision is made for the passage of a current of air through the apparatus.

This arrangement results in the copper being rapidly attacked, and the solution, which drains off through the false bottom of the apparatus into a settling tank, is returned for use over and over again until saturated, whereupon it is cooled and the copper sulphate crystallised out. The copper and acid are renewed as required, and the washing water is utilised to dilute the latter.

The crystals of copper sulphate obtained are very small, and of a high degree of purity.—C. S.

### *Potassium Ferrocyanide, Action of Carbon Dioxide on.* Guido Gigli. Chem. Zeit. **22**, 775—776.

A stream of carbon dioxide passed through a cold solution of potassium ferrocyanide, produces no apparent effect; but, if the liquid be distilled, a turbidity is produced as soon as the boiling-point is reached, whilst hydrocyanic acid can be detected in the filtrate. Further passage of carbon dioxide through the turbid liquid produces no effect; but, if the liquid be filtered, the filtrate behaves like the original solution, and the series of operations—passage of gas, boiling, filtration, and cooling—can be repeated a great many times. The amount of ferrocyanide decomposed, or of hydrocyanic acid produced at each operation, is very small, but quite sufficient to prevent the action of carbon dioxide being used, as Fresenius proposed, as a test for the presence of potassium cyanide in ferrocyanide. The greenish precipitate formed seems to be "Everett's white salt"—

$$K_2Fe.FeCy_6$$

(the ratio K : Fe in it was determined), and the carbonic acid appears thus to act similarly to, though of course not so powerfully as, weak sulphuric acid. Sulphur dioxide behaves in the same way, but is more energetic than carbon dioxide, so that by repetitions of the series of operations the ferrocyanide can be completely decomposed.—J. T. D.

### *Sulphides of Calcium and Strontium. Crystallisation of.* A. Mourlot. Comptes Rend. **127**, [11], 408—410.

The sulphides of calcium and strontium have been obtained in the crystalline form (cubical system) by subjecting in each case a mixture of the sulphate and charcoal to the action of the electric furnace. In order to obtain a product free from carbide, a sufficiently large quantity of the mixture was heated, for not more than four minutes, in a furnace supplied with a current of 1,000 ampères and 60 volts. The fused sulphides crystallised on cooling, and had the composition CaS and SrS respectively. Similar products were obtained by simply fusing, with a similar current, the amorphous sulphides prepared by Sabatier's process. The crystallised sulphides had no action on polarised light. Their densities, determined by weighing in turpentine at 15° C., were — CaS, 2·8; SrS, 3·7. Oxygen, at a red heat, converted them completely into the sulphates. Fluorine attacked them in the cold, giving the metallic fluoride and fluoride of sulphur. When mixed with carbon, and heated for 10 minutes, in a furnace supplied with a current of 1,000 ampères and 50 volts, not a trace of S remained, and the products had the composition $CaC_2$ and $SrC_2$.—H. B.

## PATENTS.

*Lead Carbonate, Ammonium Nitrate, Silicates of Sodium, and Caustic Soda; Impts. in the Manufacture of.* W. Garroway, Netherfield, N.B. Eng. Pat. 22,395, Sept. 30, 1897.

A CONSTANT stream of nitric acid is produced by heating a mixture of silica and sodium nitrate in a suitable furnace in a current of superheated steam, as described in Eng. Pat. 2489, 1896 (this Journal, 1897, 239), and in Eng. Pat. 7066, 1897 (this Journal, 1898, 157). The residue of sodium silicate may be utilised as such, or it may be treated with lime water, to obtain caustic soda in solution, and a residue of silicate of lime, available as a cement. The nitric acid is led, as produced, into earthenware pipes or stone towers, in which it is brought into contact with scrap lead or litharge. The lead nitrate solution formed, after neutralisation with ammonia, is treated under pressure with ammonia and carbonic acid, or with ammonium nitrate, to obtain lead carbonate and ammonium carbonate. The ammonium carbonate for effecting the precipitation may be obtained by decomposing ammonium sulphate with barium carbonate, which process is included in the claims.—E. S.

*Evaporating or Concentrating Liquids (Brine), Impts. in or relating to Apparatus for.* P. R. Allen and J. B. Chadwick, both of Runcorn, Cheshire. Eng. Pat. 23,339, Oct. 11, 1897.

THE evaporating pan or vessel is provided with a mechanical skimming apparatus, which can be moved to one side, when necessary, to permit of the convenient removal of the salt. For rectangular pans, the skimming apparatus consists of endless chains or their equivalent, arranged one at each side of and above the pan: means for supporting, guiding, and driving the chains, and skimming arrangements, which may be made of flexible tubing sufficiently buoyant to float on the liquid, carried by the chains, and caused thereby to travel first over and in contact with the surface of the liquid, and then back, clear of the pan and liquid. For circular pans, the skimmers are attached to one or more arms, carried by a vertical shaft, which is mounted to revolve above the centre of the pan.—R. A.

*Salt and other Substances, Impts. in Apparatus for Pulverising and Purifying.* A. MacNab, London. Eng. Pat. 23,173, Oct. 12, 1897.

SALT with concentrated brine is fed into a vertical cylinder, enlarged at its lower part, in which a central revolving shaft carries a disc, which is slightly inclined below the shoulder formed by the enlargement of the cylinder, in such manner that the salt carried by the brine through the aperture is crushed between the surfaces of the disc and the shoulder. The mixture of pulverised salt and brine passes a valve into an adjoining lower cylinder, in which a shaft revolves carrying a number of inclined blades, and having diaphragms which cause the mixture to flow in a zig-zag course to the outlet. The mixture of brine and salt is then caused to descend a column in which inclined blades revolve so as to check the downflow; the brine, carrying the impurities with it, overflows from the top of the column into a valved channel, while the salt falls to the lower part of the column.—E. S.

*Potash Salts, Impts. in the Manufacture of.* O. Imray, London. From W. T. Gibbs, Buckingham, Canada. Eng. Pat. 21,525, Oct. 22, 1897.

FELSPAR, or any other siliceous potash-containing mineral, is mixed with lime or an alkaline carbonate in stated proportions, and the mixture is fused in an electric furnace. The molten mass may be run into water, and then treated for recovery of the potash and alumina by known methods. Aluminium, iron, or calcium phosphate may be substituted for lime in the process, in which case the product, after grinding, is directly applicable as a fertiliser. If it be desired to obtain phosphorus as one of the products, carbon is added to the mixture described, and suitable arrangements are made for the conduct of the processes of distillation and condensation.—E. S.

*Fluoro-Compounds, A Process for the Manufacture of.* W. Schmeisser, Siptenfelde, Germany. Eng. Pat. 14,038, June 24, 1898.

"A PROCESS" is claimed "for the manufacture of hydrofluoric acid and fluosilicic acid respectively, as main-products, and of an alkaline sulphate as a by-product, consisting in heating a mixture of fluorspar and an acid hydrogen alkali sulphate to dull redness."

Also, "the production of alkali fluorides and silico-alkali fluorides" by the reactions above indicated.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Seger Pyrometric Scale, Extending the Limits of the.* —Techt.Thonind. Zeit.; Sprechsaal, **31**, [24], 738—739.

THE highest member of the Seger series of cones, *viz.*, No. 36, exhibits the disadvantage of departing from the regular gradation adopted in the remainder of the series, in that it contains the same molecular proportion of $SiO_2$ as No. 35, the increased fusibility being secured by the use of Rakonitz clay instead of Grünstadt kaolin.

To overcome this objection, as well as that due to the fact that the difference between the fusing points of cones Nos. 35 and 36 is greater than between the next succeeding members, *e.g.*, 35 and 34, 34—33, &c., the author proposes to modify all cones higher than No. 35 by reverting to Grünstadt kaolin for a basis, and qualifying this with additions of calcined alumina, in such a manner that either 1, 1·1, 1·2 mols. of $Al_2O_3$ are present per 2 mols. of $SiO_2$; or else the latter is modified, and 2, 1·9, 1·8, &c., mols. of $SiO_2$ are present per 1 mol. of $Al_2O_3$; the final term of the series being, in either case, pure alumina.

The following proportions are given as suitable for refractory cones, above No. 35 (pure Grünstadt kaolin):—

No. 36:—$Al_2O_3$, 1·66 $SiO_2$ = 259 parts by weight of Grünstadt kaolin, and 20·6 parts of calcined alumina.

No. 37:—$Al_2O_3$, 1·33 $SiO_2$ = 259 parts kaolin and 51·5 parts of alumina.

No. 38:—$Al_2O_3$, 1 $SiO_2$ = 259 parts of kaolin and 103 of alumina.

The fusing point of Seger's No. 36 cone is between those of the above Nos. 36 and 37.

The author considers that the higher members of the series will be useful in future developments in the manufacture of fireproof materials containing magnesite or chromite, which for some processes are more suitable than substances containing silica.—C. S.

*Clays of Missouri.* H. A. Wheeler. Eng. and Mining J. 1898, **66**, [15], 126—127.

*White-Ware Clays.*—The white-ware clays, (1) kaolin and (2) "ball" clay, the plastic clay used for furnishing the body and strength of white ware, are found in the south-eastern portion of the State, as surface deposits, resting on or associating with the lower silurian limestones. The kaolin sheets, which are always found resting on the limestone in association with heavy beds of flint *débris*, are very extensive, but most of the clay is too impure from sand, flint, "chert-boulders," &c., to be saleable without careful washing. The ball clays usually occur as pockets or small local basins in a pure limestone; these pockets are scarce, but contain amounts varying from 500 to 10,000 tons of first-class clay. The white-ware clays have the following composition:—

| — | Silica. | Alumina. | Combined Water. | Iron Sesquioxide. |
|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Ball clays....... | 45—49 | 34—37 | 12—13·5 | 0·4—1·5 |
| Kaolins.......... | 57—72 | 19—27 | 6—11 | 0·4—2·0 |

| — | Lime. | Magnesia. | Alkalis. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Ball clays ...... | 0·7—1·1 | 0·4—1·1 | 0·7—1·8 |
| Kaolins......... | 0·2—0·7 | 0·1—0·4 | 0·2—0·8 |

*Fireclays.*—There are two varieties of fireclay, the flint or rock clays, which occur in isolated pockets or basins in silurian limestones, and the plastic clays, which occur in continuous beds or sheets in the coal measures and tertiary formations. The flint clays are hard, rock-like, non-plastic, white to gray clays that are found filling former sink-holes or pot-like depressions in limestone. They contain at the most only 1—2 per cent. of impurities, and are infusible at any furnace-temperature; but owing to their non-plasticity, they are only used for "grog" or "grit," after calcining, in refractory goods. They contain no free silica or sand, but a larger proportion of alumina than normal kaolinite.

The plastic fireclays occur in two distinct portions of the State, (1) a very extensive, persistent sheet, known as the "St. Louis bed," at the base of the lower coal measures in the eastern-central part of the State, and (2) a bed of less refractory clay in the belt of tertiary measures in the south eastern corner. There are also numerous so-called beds of fireclay in the coal measures that overlie the northern half of Missouri, but the clay from these will not withstand a temperature of 2,500° F., and is thus only suitable for stoneware and terra-cotta. The fireclay from the St. Louis bed is stated to give a strong, highly resistant, superior grade of firebrick, especially when the flint clay is used for "grog." The fireclays have the following composition :—

| — | Silica. | Alumina. | Combined Water. | Iron Sesquioxide. |
|---|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| Flint clays...... | 43—50 | 38—42 | 12—14 | 0·2—0·8 |
| Plastic fireclay.. | 48—70 | 20—38 | 7—13 | 0·2—5·0 |

| — | Lime. | Magnesia. | Alkalis. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Flint clays...... | 0·2—1·9 | 0·1—0·6 | 0·1—2·0 |
| Plastic fireclay.. | 0·1—2·0 | 0·1—1·0 | 0·1—1·4 |

*Potter's Clay.*—This variety of clay, which is suitable for stoneware, terra-cotta, &c., occurs abundantly in beds in the western and northern parts of Missouri, in the coal measures, as occasional pockets in the drift in the northern portion of the State, and as extensive beds in the tertiary measures in the south-eastern part of the State. The clay only differs from the plastic fireclay in containing a larger proportion of fluxing impurities, *i.e.*, 6—10 per cent. as against 4—6 per cent.

*Shales.*—The shales occur as sheet-like formations, from 1 ft. to over 100 ft. in thickness, in the coal measures in the western, northern, and part of the eastern portions of the State. They vary greatly in composition :—Silica, 46—75 ; alumina, 10—25 ; combined water, 4—9 ; iron sesquioxide, 2—8 ; lime, 0·5—11 ; magnesia, 0·1—8 ; and alkalis, 1·5—4 per cent.

*Brick Clays.*—The "loess" variety of surface clay occurs abundantly in Missouri ; it is stated to give a strong brick of excellent red colour, that is rather free from the very common trouble of efflorescence. The clays are quite free from stones or gravel, and are generally quite uniform over local areas, though liable to be rather sandy in places.

The glacial clays that overlie the northern half of the State, and the reassorted residual clays derived from the chemical disintegration of local limestones, are also used in the southern portion of the State, but both are liable to be very variable in character. The loess clays have the following composition :—Silica, 71—74 ; alumina, 11·5—13 ; combined water, 3—7 ; iron sesquioxide, 3·4—4·8 ; lime, 0·8—2·4 ; magnesia, 0·6—1·5 ; and alkalis, 3—3·3 per cent.

*Gumbo Clays.*—These are soft, black, extremely tenacious or plastic clays, that are found along all the low river bottoms ; and they are said to be especially satisfactory for burning into railroad ballast or burnt-clay gravel, on account of their great toughness, easy fusibility, and peculiar property of decrepitating or breaking up into coarse granules of gravel when burnt. The burnt "gumbo" makes a durable, elastic, clean ballast that is easier to handle than broken stone. The clays vary in composition as follows :—Silica, 55—65 ; alumina, 15—20 ; combined water, 6—10 ; iron sesquioxide, 5—7 ; lime, 1—3 ; magnesia, 0·5—2 ; and alkalis, 2·5—4 per cent.—A. S.

*Glass, Experimental Researches on the Dilatation of.* A. Granger. Monit. Scient., Oct. 1898, **12**, 681—690.

The conclusions drawn from the experiments of Grenet, detailed in the paper, are to the effect that—

Whilst the addition of such compounds as $B_2O_3$, PbO, CaO, MnO, $Al_2O_3$, in small quantity, reduces the coefficient of dilatation of glass, the employment of larger amounts has an opposite effect.

The circumstance that the dilatation of the double silicates of potassium and sodium is less than the mean of that of the constituent simple silicates indicates that glasses of simple composition will exhibit a high degree of dilatation, and *vice versâ*.

Although alumina, in the proportions in which it can be added, reduces dilatation, yet, owing to the stability it imparts to the glass, this oxide enables very alkaline silicates of high dilatation and power of resisting water to be produced.

Cryolite, when used in sufficient amount to crystallise in glass, largely increases the dilatation.

Though the dilatation of fluorine is high, the effect produced by calcium fluoride is merely slight. The addition of moderately large proportions results in devitrification, probably the result of complex reactions between the fluoride and the silicate. The quantity can, however, be increased in presence of calcium phosphate.

To summarise the results :—Potash, soda, lithia, lime, calcium phosphate, calcium fluoride, and cryolite, all increase dilatation, whilst the converse effect is produced by boric anhydride, silica, alumina, lead oxide, zinc oxide, iron oxide, pigmentary oxides, and cryolite in small amount.
—C. S.

*Glass, Coloured.* W. M. Sprechsaal, **31**, [22], 677—678.

The following recipes are given for glasses suitable for working together in pairs by flashing, on account of their approximately equal coefficients of expansion :—

I. *Spar Glass.*—Sand, 100 ; potassium carb., 5 ; "ammonia soda" (soda ash), 19 ; fluorspar, 10 ; felspar, 20 ; zinc white, 6 ; minium, 6 parts ; and

*White Glass.*—Sand, 100 ; potassium carb., 6 ; "ammonia soda" (soda ash), 24 ; nitre, 2 ; lime, 16 parts.

II. *Bone Ash Glass.*—Sand, 100 ; potassium carb., 44 ; bone ash, 28 ; minium, 2 ; nitre, 2 parts ; and

*White Glass.*—Sand, 100 ; potassium carb., 42 ; lime, 16 ; nitre, 2 parts.

III. *Cryolite Glass.*—Sand, 100 ; soda ash, 20 ; potassium carb., ·6 ; cryolite, 14 ; lime, 5 ; minium, 2 ; nitre, 3 parts ; and

*Dark Green Glass.*—Sand, 100 ; soda ash, 24 ; potassium carb., 12 ; lime, 10 ; minium, 2 ; nitre, 3 ; chromium oxide, 1 ; copper oxide, 3 ; iron scale, 1 ; cobalt oxide, 0·2 part.

IV. *Cryolite Glass.*—Sand, 100 ; potassium carb., 18 ; lime, 12 ; cryolite, 15 ; zinc white, 3 ; arsenic, 1 part ; and

*Black Cryolite Glass.*—Sand, 100 ; potassium carb., 33 ; lime, 16 ; manganese oxide, 15 parts.

V. *Alabaster Glass.*—Sand, 100 ; potassium carb., 40 ; nitre, 6 ; bone ash, 6 ; magnesium silicate, 4 parts ; and

*White Glass.*—Sand, 100 ; potassium carb., 40 ; lime, 16 parts.

VI. *Alabaster Glass*—V., but with 42 parts of potassium carb. in place of 40 ; and

*Golden-Yellow Glass.*—Sand, 100 ; potassium carb., 24 ; soda ash, 12 ; lime, 18 ; nitre, 3 ; arsenic, 0·5 ; flowers of sulphur, 5 (or wood charcoal, 3, and manganese oxide, 3) parts.

VII. *Alabaster Glass.*—Sand, 100 ; potassium carb., 42 ; magnesium silicate, 4 ; bone ash, 6 ; nitre, 4 ; arsenic, 1 part ; and

*Chrysopras Glass.*—The same glass *plus* sodium uranate, 1 part ; and copper oxide, 0·3 part ; or

*Turquoise Glass.*—The same alabaster glass, *plus* cobalt oxide, 0·04 ; and copper oxide, 0·7 part.

VIII. *Enamel Glass.*—Sand, 100; potassium carb., 18; nitre, 6; minium, 24; arsenic, 10; zinc white, 5; cryolite 6 parts; and

*Blue Flash Glass.*—Sand, 100; potassium carb., 21; soda ash, 10; nitre, 2; borax, 2; lime, 16; copper oxide, 1; cobalt oxide, 0·2 part.—C. S.

*Ceramic Objects, Underglaze Printing on.* F. Ohnemüller. Thonwaaren-Indust. **12**, [26], 493.

The new process consists in coating the biscuit-ware with glycerin, after which the design is applied by means of transfers, and the goods are fired subsequent to dipping or otherwise covering the surface with glaze.

The ink of the transfers adheres firmly to the glycerin-coated ware, and the paper is removed in the ordinary way. The design can then be shaded by colours laid on with the brush, and the whole is left to dry before dipping. The glaze is also allowed to dry for an hour, and is then smoothed by rubbing with the fingers, this operation also distributing the glaze over any of the closely covered surfaces of the design to which it did not properly adhere during the immersion.—C. S.

*Porcelain Enamels, On the Colorations of.* A. Le Chatelier and P. Chapuy. Comptes Rend. **127**, [12], 433—436.

The authors have studied the range of coloration produced by different metals on glazes in the porcelain kiln. The temperature of the kiln was kept at 1,360°—1,390° C., the percentage of metal (added as oxide or other compound), and the composition of the atmosphere and of the glaze, being varied. The following are the principal colours obtained in the works furnace, independently of the more extended laboratory trials.

Cr.—*Violet* (O), *blues* (O), green, yellow, *orange* (O), red (O).

Co.—Blue, *green* (R), *rose* (R).

Cu.—Blue, green, yellow, red.

Di.—*Violet* (R), *blue* (R), *green* (R).

Er.—*Violet* (R), *blue* (R), *green* (O), *red* (O).

Fe.—Blue (R), green (R), yellow, red (R).

La.—*Violet* (R), *blue* (R).

Mn.—*Violet* (O), *blues* (O), greens, yellow, *reds*.

Mo.—*Violet* (O), *blues* (O).

Nd. (Neodymium).—*Violet* (R), *blue* (R), *green* (R).

Ni.—*Violet* (O), blue (O), green, yellow, *red* (O).

Ti.—Violets (R), blue (R), greens (R), yellow, red (R).

W.—*Violet* (R), blues (R), yellow, *reds* (R).

Tl.—*Blue* (R), *green* (R).

Th.—Blue (R), green (R).

V.—*Blue* (O), *green* (O), yellow (O).

In this list, *italics* indicate new, or hitherto little known colorations. "O" and "R" indicate oxidising and reducing atmospheres respectively, but only the types of atmosphere, which may vary in composition—for example, Cl, S, metallic vapours, &c. The same colours are often obtainable in different atmospheres, if one varies suitably the composition of the glaze and metallic compound employed. The chromium *blues* are obtained in an atmosphere of air, using a felspar glaze in presence of zinc, on adding 0·5 to 1·2 per cent. of $Cr_2O_3$; with 1·2 to 1·5 per cent., *rose* develops; and at 1·6 per cent. the blue disappears. $Di_2O_3$ gives tints different from those given by pure $Nd_2O_3$, the latter being not so bright; both give their colorations in the oxidising atmosphere also, but the tints are duller. The violets, greens, and reds with $TiO_2$ were obtained without the addition of other colour oxides, simply by varying the atmosphere. The blues of W were obtained by adding $WO_3$ to a glaze free from Ba.

Analyses of some of the glazes are given at the top of the adjoining column.

Among the colorations the authors obtained in the porcelain kiln, are the following, as presenting most interest:—

*Violet tints* from Cr., Mn., and Di.

*Blue tints* from Y., V., Mo., W., tints similar to that from Ti., Cr., Fe., Mn., Th., Di., which approach the copper blues.

*Green tints* from Co., excellent. Many greens from Fe., Mn., Ni., different from those already known.

*Red tints* from Fe. Tungsten red is quite comparable with copper red. Erbium red.

| — | Erbium. | | | Neodymium. | | |
|---|---|---|---|---|---|---|
| | Blue. | Green. | Red. | Violet. | Blue. | Green. |
| $SiO_2$ ...... | 42·5 | 31·5 | 60·8 | 59·9 | 63·5 | 47·3 |
| $Al_2O_3$ ..... | 8·8 | 6·2 | 11·6 | 10·8 | 11·8 | 9·3 |
| CaO ...... | 1·5 | 4·9 | 1·1 | 2·0 | 1·4 | 1·3 |
| SrO....... | .. | .. | .. | 11·8 | 17·3 | .. |
| BaO ...... | 26·0 | .. | 18·5 | . | .. | 27·9 |
| $K_2O$ ...... | 0·5 | 0·4 | 0·7 | 0·7 | 0·6 | 0·5 |
| $Na_2O$ ..... | 1·7 | 0·9 | 1·9 | 1·8 | 1·3 | 1·8 |
| $B_2O_3$ ...... | 5·4 | .. | .. | .. | .. | 6·0 |
| ZnO ...... | .. | 46·8 | .. | .. | .. | .. |
| NdO ...... | .. | .. | .. | 1·3 | 4·2 | 5·8 |
| ErO ...... | 13·7 | 9·4 | 5·3 | .. | .. | .. |
| | 100·1 | 100·1 | 99·9 | *100·0(*sic*) | 100·1 | 99·9 |

* The actual total here should be 88·3.

—H. B.

## PATENTS.

*Hardening, Enamel, and similar Kilns, Impts. in.* H. Capewell, Hanley, Staffordshire. Eng. Pat. 23,175, Oct. 9, 1897.

The kiln is conveniently rectangular in shape, with an arched roof having an opening at the top for the escape of the products of combustion. An inner wall, built 3 in. from the outer one, provides a space forming a vertical flue, completely surrounding the sides and top of the oven. This flue is divided vertically into sections by partition walls, and each compartment is provided with an independent damper to regulate the course of the products of combustion. The oven is also divided by walls, and these are pierced by holes so as to give free communication between the various compartments. The kiln has two fire holes, one at the side and the other at the end, with branched flues, the branches communicating with the compartments of the side flue. The bottom flues are built upon an incline, rising from the fire holes inwards. It is to be observed that the vertical side flues are of only half the width that is customary in kilns of this character.

—H. H. B. S.

*Sheet and Plate Glass, Impts. in or relating to the Manufacture of.* P. T. Sievert, Dresden. Eng. Pat. 14,873, July 6, 1898.

In the manufacture or finishing of glass plates, the plastic glass is rolled or pressed between soft, damp, elastic surfaces of fibrous substances, such as wood, paper, or asbestos pulp, instead of, as usual, between metallic surfaces. The plates or rollers used, are either prepared entirely from these substances, or they are faced with them, and provision is made for maintaining them in a damp condition during the pressing or rolling. It is said that it is possible in this way to produce sheet or plate glass as thin as desired, and with a faultless polished appearance.

—H. H. B. S.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*Sandstone, Artificial, in Belgium.* Eng. and Mining J. 1898 **66**, [11], 307.

A description is given of the method of manufacturing artificial sandstone at Uccle-Calevoet, Belgium, the product obtained being a silicate of lime, whilst the working conditions are probably similar to those under which natural sandstone is formed, *viz.*, the presence of hot water and great pressure. The process is as follows:—80 parts of very clean well-dried, course sand are intimately mixed with 20 parts of hydraulic quicklime, and the mixture charged into a box of sheet iron, which is then introduced into a closed boiler filled with hot water that is kept for 72 hours under a pressure of 6 atmospheres and at a temperature of about 165° C. The block of sandstone produced possesses the advantage over natural sandstone of being absolutely homogeneous. When taken out of the box, it is so soft that it may be cut with a knife, but on exposure to the air, it gives up its moisture, rapidly hardens, and acquires considerable strength. It is not affected by frost, and does not absorb more than 6 or 7 per cent. of moisture, even after prolonged desiccation. Its resistance to crushing strain is greater than 400 kilos. per sq. cm., being four times that of French white freestone, and almost as great as that of the famous Gobertange stone, which it resembles in colour and appearance. The artificial sandstone, which resists climatic influences better than the natural variety, may be produced in large blocks of irregular shape, for about 10 cents per cubic toot, which includes cost of boxes for blocks of regular dimensions very near to the finished sizes, and also saws for supplying sawn stone.—A. S.

*Slag Brick in Russia.* Eng. and Mining J. 1898, **66**, [14], 394.

The manufacture of slag brick in Russia is carried out in the following manner:—The granulated blast-furnace slag, after sifting and drying, is dry-mixed with from 4 to 10 per cent. of very finely ground lime (in a particular example, 4 per cent. was added, bringing the total lime content up to 55 per cent.), and the mixture formed into bricks in a revolving dry press. The bricks must be handled very carefully at first, but after being dried by exposure for six days, they become perfectly hard. Burning is not necessary. The slag brick is stated to possess strong hydraulic properties, to withstand high and low temperatures, to weigh less than stone, and to require little mortar when laid up. Its tensile strength is about 312 lb.; crushing strength, from1,250—5,600 lb. per square inch, according to the time of hardening. The composition of the granulated slag is: Silica, 22·5—35·0 per cent.; alumina, 14—15 per cent.; iron, 3·3—1·1 per cent.; manganese, 0·0—0·3 per cent.; lime, 51—45 per cent.; magnesia, 1·4 per cent. to traces; sulphur, 0·3—0·4 per cent. Loss on heating, 2·3—7·5 per cent.—A. S.

*Brickwork Pillars, Testing.* Riga Indust. Zeit.; Thonind. Zeit. **22**, [51], 673—675.

In order to ascertain the amount of superficial area of brickwork—*i.e.* number of bricks—that can be set by means of 1 cb. ft. of mortar, a number of pillars were built, the mortars employed being compounded of sand and lime, or Roman or Portland cement (or a mixture of both). The average contraction of the wet mortar, as compared with the original bulk of the loose materials, was equal to a reduction of about 30 per cent. by volume.

On demolishing the pillars it was found that 1 cb. ft. of wet mortar sufficed to set about 4 cb. ft. of brickwork, *i.e.*, 37 bricks (size 24·75 × 11·75 × 7·3 cm.).

Compression tests were performed on special cubes of the mortar, that taken from the pillars being too irregular in surface to afford satisfactory test-samples. In general the best results were yielded by the samples containing one part of sand per three parts of cement, the highest figures being furnished by Portland cement mortar, and the next best by that containing Roman and Portland cements in equal proportions.

It was also observed that the bricks in all the pillars set with Portland cement were very difficult to dislodge, and, for the most part, split, leaving fragments adhering to the mortar, even when the ratio of cement to sand was as low as 1:7. On this account it is considered highly advisable to increase the tenacity of Roman cement mortars by an addition of Portland cement.—C. S.

*Portland Cement, Comparative Action of Various Additions to.* L. Erdmenger. Thonind. Zeit. **22**, [81], 929—930.

The subjoined tables give the tensile strength furnished in parallel experiments conducted by Lundteigen and the author, with the cement mixtures specified:—

*Tensile Strength in Kilos. per Square Inch.*

| | — | Pure Cement. | 2 Cement. 6 Normal Sand. | 1 Cement. 1 Trass. 6 Normal Sand. | 1 Cement. 1 American Volcanic Ash. 6 Normat Sand. | 1 Cement. 1 Swedish Factor Waste.* 6 Normal Sand. |
|---|---|---|---|---|---|---|
| | | Water, 25 per Cent. | Water, 8½ per Cent. | Water, 10 per Cent. | Water, 10½ per Cent. | Water, 11½ per Cent. |
| Erdmenger. | 7 days | 283·0 | 123·0 | 75·0 | 78·9 | 159·9 |
| | 1 month | 358·6 | 152·6 | 119·1 | 143·4 | 453·4 |
| | 3 months | 447·4 | 206·6 | 163·2 | 202·0 | 294·1 |
| | 6 " | .. | 235·5 | 196·7 | 236·9 | 329·8 |
| | 12 " | .. | 265·2 | 221·7 | 453·4 | 331·0 |
| | — | Water, 23½ per Cent. | Water, 12 per Cent. | Water, 12½ per Cent. | Water, 12½ per Cent. | Water, 13½ per Cent. |
| Lundteigen. | 7 days F. | 415·0 | 124·7 | 57·7 | 46·7 | 260·0 |
| | 7 days S. | 485·1 | 124·3 | 66·7 | 73·7 | 255·3 |
| | 1 month F. | 496·0 | 176·7 | 150·0 | 139·0 | 357·3 |
| | 1 month S. | 611·3 | 206·3 | 185·3 | 217·3 | 414·3 |
| | 3 months F. | 634·7 | 259·3 | 194·3 | 218·0 | 428·3 |
| | 3 months S. | 720·0 | 286·0 | 338·7 | 363·3 | 469·0 |
| | 6 " F. | 638·3 | 304·0 | 232·0 | 347·3 | 459·0 |
| | 6 " S. | 824·3 | 310·3 | 407·3 | 419·3 | 490·0 |
| | 12 " F. | 637·7 | 346·7 | 294·7 | 399·0 | 536·0 |
| | 12 " S. | 783·3 | 365·7 | 421·0 | 428·0 | 524·3 |

N.B.—F = fresh water; S = salt water, containing $CaSO_4$, 1·0214; $MgSO_4$, 0·238; NaCl, 0·1515 grm. per litre.

* $SiO_2$, 59·04; $Al_2O_3 + Fe_2O_3$, 14·66; CaO, 1·38; $SO_3$, 2·66; loss on calcination, 21·76 per cent.

—C. S.

*Clays of Missouri.* H. A. Wheeler. Eng. and Mining J. 1898, **66**, [15], 426.

*See under* VIII., *page* 1046.

*Portland Cement Mortar, The Calcium Hydroxide (Free Lime) in Hardened.* Schuliatschenko. Thonind. Zeit. **22**, 400.

*See under* XXIII., *page* 1073.

## PATENTS.

*Cement, Artificial Stone, and the like; An Improved Composition applicable for.* C. G. J. Frosell, Stockholm, Sweden. Eng. Pat. 23,993, Oct. 18, 1897.

Burned magnesite or dolomite is mixed with an aqueous solution of zinc or magnesium chloride. After the mass has become somewhat stiff, it is further mixed with aqueous solutions of phosphates, carbonates, or stannates, or with stannic acid, sulphates of the alkalis, or sulphate of magnesia. This mixture forms the cementitious material, and it is used to bind sand, broken marble or other stone, metallic powder, coal, vegetable or animal fibre, &c.—H. H. B. S.

*Portland Cement, Impts. in the Manufacture of.* O. Imray, London. From La Société Anonyme des Ciments et Platres de Vilvorde, Belgium. Eng. Pat. 15,173, July 25, 1898.

Common salt is thrown into the kilns with the charges, in the proportion of 50 kilos. of salt to 10 tons of cement material. The addition of the salt is said to effect a large economy in the consumption of fuel, and to produce better manufacturing results. No sodium chloride is found in the finished cement.—H. H. B. S.

## X.—METALLURGY.

*Goldfields, Mining Practice of the Witwatersrand, S. Africa.* G. A. Denny. Trans. Australasian Inst. of Mining Engineers, **5**, 8—70.

This paper is a detailed review of mining generally, and its cost, on the Witwatersrand. In the course of his remarks, the author works out a comparison between the air-compression drilling plant and the new Bladray electric drill plant in respect to the original outlay, cost of working, and labour. The effects of the Bladray drill upon the economics of the mining industry, judged from its experimental performances, are summed up as follows:—The capital outlay and the cost of the upkeep of a rock-drilling plant would be considerably reduced; the effective performance of the Bladray machines, proportionately to the power generated, is 85 per cent. as against 10 per cent. with air-drills; the simplicity in design and the few working parts commend it for long and rough usage; the cost of running, as against air-drills, on equal footage bored, is in the proportion of 10 to 31, or practically one-third; it is the most successful mechanical stoping-drill in hand formation, and its use in the stopes will effect a saving over hand-labour of about 78 per cent.; only one-tenth of the present native labour would be required, with a consequent reduction in labour transit, and the wages would be 60 per cent. of the existing rate; it would enhance the profit-earning power, and would advance the exploitation of low-grade ores in outlying districts.—A. W.

*Ores [Lead, Silver], Reverberatory Furnace Smelting of.* T. J. Dyson. Trans. Australasian Inst. of Mining Engineers, **5**, 71—75.

The author gives the results obtained by smelting lead sulphide ores (500 to 600 lb.) with added carbonate or oxide of lead (400 lb.) on the surface of a bath of blast-furnace lead in a reverberatory furnace. The action takes place for two hours, after which the slag is skimmed off the metal on to a sloping bed between the fire bridge and the lead bath, where it is drained or liquated for two hours previous to removal and subsequent treatment in the cupola. After about twenty charges, the lead, containing about 1 per cent. of silver, is tapped for cupellation treatment. The richer and cleaner the ore, the greater is the proportion of lead recovered; thus, about 56 per cent. of the lead present was obtained with an ore mixture containing 50 per cent. of lead, over 36 per cent. being retained in the slag, and 7 per cent. unaccounted for; whereas with a pure ore, containing 75 per cent., the yield amounted to from 75 to 80 per cent. of the metal present. These results compare advantageously with other furnaces where the lead bath and separate draining bed for slags are not used. The high cost of hand-labour in Australia renders this process less advantageous with poor ores, slimes, concentrates, &c., which produce heavy slags, especially the sticky zinc slags; and slimes containing 15 to 25 per cent. of zinc and only 25 to 30 per cent. of lead should not be treated by the roast-reaction process.—A. W.

*Surfusion in Metals and Alloys.* W. C. Roberts-Austen. Proc. Roy. Soc. **63**, 447—454.

The author records some further curves obtained by his recording pyrometer, showing the arrest of the fall of temperature, and the temporary rise due to the release of the latent heat of fusion, during the solidification of gradually cooling molten metals and alloys. Surfusion has been now detected in copper, bismuth, antimony, lead, and tin, and in the eutectic, or lowest fusing-point alloy of bismuth and copper. In pure alloys, the angle of arrest is sharply defined, but the angle of resumption is rounded off; whereas in isomorphous mixtures neither angle is sharp, the arrest being gradual, as the alloy assumes the pasty state previous to actual solidification.

It is observed that with gold the temperature during surfusion falls about 2° below its true freezing point. The curves of alloys sometimes show more than the simple release of the latent heat of fusion, such as smaller breaks at the moment of surfusion, due probably to some molecular adjustment of the constituents. This is noticeable in an alloy of 64 tin and 36 lead, where a point in the downward surfusion curve indicates probable crystallisation of a small quantity of lead, which is confirmed by a corresponding point in the upward curve of increased temperature when the lead would be remelted. In the series of lead tin alloys it is found that there are two, and sometimes three, freezing points,—first, the initial freezing point, which is higher as the percentage of tin is increased; second, the eutectic alloy, which is a constant at 183°; and the third, below this temperature, which is sometimes occasioned by the falling out of lead from the surfused alloy. As it is necessary to stir the alloy to obtain the first point, and to allow it quietly to surfuse to obtain the third, it is not possible to record the three points in one experiment. If tin be in excess of the amount required for the eutectic alloy, it crystallises first, and its presence in the bath prevents the surfusion of the eutectic alloy. By these results, alloys are, for the first time, experimentally shown to be similar in behaviour to saline solutions as regards selective surfusion.

In speaking of the vaporisation of metals at low temperatures *in vacuo*, the author mentions an experiment in which he heated polished discs of silver and cadmium, separated by a ring of glass, in a vacuous tube in a water-bath at 50° C. for eight days, when an appreciable deposit of a tinted cadmium-silver alloy was found on the surface of the silver, proving that cadmium must have passed across the intervening vacuous space.—A. W.

*Cast Iron, Heat of Solution of.* E. D. Campbell and W. E. Hartman. J. Amer. Chem. Soc. **20**, [9], 690—695.

It is well known that, in the blast furnace, cast iron changes colour with increase of temperature; this change continues until silvery or glazed iron is formed, when the appearance and properties of the iron undergo an abrupt change. It seemed probable that this abrupt change might be due to a change of form in which silicon exists in cast iron, and, to obtain definite evidence on this point, thermo-chemical investigations were made.

Each sample of cast iron was first carefully analysed, and then the heat of solution of 1 grm. in ammonium copper chloride (+ 0·84 per cent. free hydrochloric acid) determined, corrections being made for loss by radiation and gain by oxidation of cuprous chloride. The number thus found was divided by the percentage of iron actually obtained in solution, to give the number of calories evolved per grm. of iron dissolved. From this number was subtracted the heat due to oxidation of manganese and phosphorus, and also the heat of solution of 1 grm. of pure iron. The remainder was taken to represent the heat evolved by the oxidation of the silicon accompanying each grm. of iron dissolved. The conclusions drawn were: (1) When the carbon is nearly all in the graphitic form, the heat evolved by the oxidation of the silicon is proportional to the amount of that element present. (2) When the carbon is chiefly combined, the heat evolution is greatly diminished, owing to the large amount of heat absorbed in the decomposition of the various compounds of iron, carbon, and silicon. (3) The heat of formation of the compound of silicon and iron is about 27,700 calories. (4) When the percentage of silicon present corresponds with the formula $SiFe_3$, the compound is insoluble in ammonium copper chloride.—E. H. T.

*Steel, A New Method of Case-hardening.* A. Gay. Rev. Gén. des Sciences; through Eng. and Mining J. 1898, **66**, [12], 334.

The method used at the Pamiers Steelworks for case-hardening ingots, consists in directly carburising one of the ingot faces, at the time of casting, by lining one of the vertical sides of the mould with carburising matters, and preventing this carburisation from penetrating too deeply into the inside of the ingot, by cooling the vertical side of the mould opposite to the carburising side, so that, on contact with it, the steel will soon become pasty and incapable of absorbing the carbon, which begins to dissolve. The mould into which the extra-mild steel is run has one of its sides lined with a prism, made of carburising matters; and, on this side, the thickness of the layer of substances that are bad conductors of heat behind the carburising matters may vary according to the amount of cementation or case-hardening required. The opposite side is, on the contrary, formed of a substance which is a good conductor of heat, *viz.*, a block of cast iron, cooling down by its conductibility the back portion of the ingot. The linings of the two vertical sides at right angles to the two above mentioned, affording transition between the bad and the good conducting sides, consist of prisms of refractory materials, the edges of which abut against the metal side, while the form of the mould, in which account is taken of the differences of cooling at the centre and at the sides of the ingot, affords a progressive cooling, so that the layer of cemented steel is almost uniform over the whole of the hard surface of the ingot. The case-hardened surface is rather rough; but all irregularity disappears in forging, which may be effected without special precaution and at a comparatively low temperature, by the press rather than the steam hammer. Ingots of half a ton to three tons have been cast in this manner; and a three-ton ingot, 40 cm., reduced by forging and rolling to one-fourth that thickness, was found to contain from 1·8 to 0·20 per cent. of carbon between its hard surface and a depth of 5 mm.; from 0·60 to 0·40 per cent. between 1 in. and 2 ins. from the surface; and from 0·35 to 0·15 per cent. between 90 and 100 mm., or $3\frac{7}{16}$ ins. and 4 ins.—A. S.

*Gold Amalgamation, Influence of Temperature on.* E. R. Woakes. Eng. and Mining J. 1898, **66**, [13], 370.

The author has met with the same difficulties in gold amalgamation as described by Sharples (this Journal, 1898, 928), but has obtained satisfactory results with the following method:—Cast-steel liners are used in the mortars in place of the back coppers, whilst a silvered copper plate (6 ins. wide) extends along the outside lip of the mortar, and is held in place by the pressure of the chuck block, under which it passes. A splash board is placed in front of the screens at such an angle as to lead the pulp well on to the top of the plate. Two inches below this top lip plate is a second removable lip plate, 50 by 10 ins., then an amalgam trough, 4 ins. deep and 4 ins. wide, then the apron plates, 9ft. by 48 ins., whilst below these are two more mercury traps or "catch-alls," from whence the pulp passes over Fruevanuers and blanket tables, the tailings going to waste, and the concentrates to cyanide treatment. Every eight hours the plates are softened with mercury and rubbed with rubber to remove loose amalgam, which is accounted for in the table as daily clean-ups. At the end of the month, the eight lip plates are taken out and surrounded in a box with hot sand, the latter being first heated nearly to dull redness, and then shovelled on to the plates. After half an hour, the plates are taken out and scraped, the amalgam recovered in this operation being of the heaviest and finest quality. Hot sand is spread over the upper 4 ft. of the apron plates, and, as it cools, is replaced by fresh hot sand until a trial shows the plates to be soft, when the amalgam can easily be removed by scraping, leaving the plates in good condition.

The yield of amalgam from the various sources during a month's run was as follows. The amalgam contains 36 per cent. of gold, the melted gold being 932·71 fine:—

| — | Amalgam. Oz. Troy. | Per Cent. of Total. |
|---|---|---|
| Daily clean-ups | 606·5 | 24·00 |
| Blanket washing (by barrel) | 23·7 | 0·94 |
| Lip plates | 815·7 | 32·28 |
| Apron plates | 461·8 | 18·28 |
| Battery sands and upper catch-alls | 590·3 | 23·36 |
| Lower catch-alls | 28·6 | 1·13 |
| | 2,526·6 | 99·99 |

—A. S.

*Cyanide Process, Application of Oxygen in the.* H. T. Durant. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 59—62.

The author advocates the use of the centrifugal pump for supplying the necessary oxygen to slimes, &c., undergoing extraction with cyanide, and describes a pump in which one or more air-valves, which open inwards and close with inside pressure, are fitted into the suction pipe between the Peet valve, regulating the flow of the slimes, and the centrifugal pump. By this means the quantity of air drawn in can be regulated in a manner far more satisfactory than by trusting to the air being drawn through loose glands and defective packing.—A. W.

*Gold [Cyanide], Precipitating, by Means of Copper Salts.* P. de Wilde. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 65—66.

In meeting the objections to the process of precipitating gold, as cuprous aurous cyanide, from cyanide solutions by means of sulphurous acid and copper sulphate, the author admits that a considerable amount of water is required. He ventures to overcome this by destroying the reducing properties of the liquid after the gold precipitate has been separated, and thus rendering it fit to be used again with fresh cyanide. For this purpose he adds barium peroxide, sodium dioxide, solution of bleaching powder, or an alkali or other chlorate to the liquid until the bisulphites present are destroyed.

It is possible, although not practicable, to recover part of the cyanogen from the dried precipitate of cuprous aurous cyanide by passing over it gaseous sulphuretted hydrogen, by which is produced hydrocyanic acid gas, cuprous sulphide, and aurous cyanide. The first of these is absorbed, after passing over lead sulphate to remove the sulphuretted hydrogen, by alkali to produce fresh cyanide, whilst the mixed residue is roasted to yield gold, copper sulphate, and copper oxide. This mixture is then treated with sulphuric acid and the remaining spongy gold melted.—A. W.

*Slimes, Coagulation and Rapid Settling of.* A. Prister. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 82.

The coagulating action of various salts and acids in solution on suspended slimes may be explained as a simple solution problem. The slime pulp may be considered as a chemical and physical solution which is somewhat comparable with the result of the action of water on lime, wherein there are two combinations of the water and lime—one solid and the other liquid. An equilibrium is established between them, and continues while the conditions remain unchanged, but it is disturbed by a change in temperature. The author maintains that water containing suspended slimes is homogeneous, and therefore may be considered as a solution, the equilibrium of which is disturbed, with the resulting physical effect of coagulation, by (1) raising the temperature of the liquid; (2) the addition of some acid, alkali, or salt; or (3) changing the proportions of water and solid matter. This disturbance may or may not be accompanied by a chemical reaction.—A. W.

*Slimes, Coagulation and Rapid Settling of.* W. A. Caldecott. J. T. Carrick. J. Chem. and Metall. Soc. of S. Africa, 1898, **6**, 96—97.

These authors separately write in reply to Prister's explanation of the coagulating effect of lime upon slimes. The former states that perfectly clear lime water will

instantly cause flocculation of slime pulp, and that in the case of weathered ores yielding ferric hydrate with the lime, the settlement is less rapid than in slimes from new ores. The latter disputes the statement that ordinary slimes settle according to Rittinger's formula, although they may do so in the presence of lime; and he maintains that agglomeration must take place before precipitation. Consequently he advises the addition of the lime to the mortar box, to give more time and chance for the particles to agglomerate.
—A. W.

*Slime Treatment, Discrepancies in.* W. A. Caldecott. J. Chem. and Metall. Soc. of South Africa, 1898, **6**, 90—92.

A WET slime residue contained 10 grains of undissolved gold per ton in the solid portion, and sufficient dissolved gold in the moisture to bring the total to 25·5 grains per ton in the whole residue when dried. On assaying this dried material, the result yielded only 15, 17, and 20 grains, according to the fineness and quantity of litharge used in the assay. If previously digested with water, filtered, and the solution evaporated with litharge to secure the soluble gold, and this assayed with the washed residue, the results were 21 and 23 grains, as the quantity of water used was increased. The probable explanation of these discrepancies is that the soluble gold deposited in the dried slime is in such a minute state of division that even the lead globules from the litharge fail to collect it.

The author next points out that the two formulæ for calculating the specific gravity of dry slimes, though both mathematically correct, give results as widely different as the difference between 2·7 and 2·5. In commercial application, care should be taken to use that formula which starts with moist slimes, as being more in accordance with the treatment and results by practice. To obtain the specific gravity of any slime that has once been dried, it is necessary to boil with water, apparently to expel entangled air.
—A. W.

*Metallic Calcium.* H. Moissan. Eng. and Mining J. 1898, **66**, [15], 424.

THE author has obtained metallic calcium by reducing calcium iodide with excess of sodium in an iron crucible, which is kept at red heat for an hour. The resulting regulus still contains sodium, and the latter is extracted either by means of alcohol, which does not attack the calcium as long as there is enough sodium present, or in an electrolytic crucible cell of nickel, which forms the cathode, with a carbon anode. By the first method the calcium is obtained as a silver-white powder; with the latter process, in small globules or fused and crystallised masses. The metal can be forged, moulded, and drawn to wire. When heated in hydrogen, it forms, at a red heat, a hydride, $CaH_2$, in the form of a white powder; heated in nitrogen, a nitride, $Ca_3N_2$, is formed. This nitride reacts with hydrogen, forming ammonia and the hydride already mentioned; it also yields ammonia with water.—A. S.

*Tin, Electrolytic Determination of.* E. D. Campbell and E. C. Champion. J. Amer. Chem. Soc. **20**, [9], 687.

*See under* XXIII., *page* 1073.

*Zinc in Ores, Electrolytic Determination of.* Paweck. Eng. and Mining J. 1898, **66**, [15], 421.

*See under* XXIII., *page* 1073.

*Slag Brick in Russia.* Eng. and Mining J. 1898, **66**, [11], 391.

*See under* IX., *page* 1014.

*Cyanide Solution, Estimation of Oxygen in Working [Gold].* A. F. Crosse. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 107.

*See under* XXIII., *page* 1074.

## PATENTS.

*Steel, Impts. in the Manufacture of.* T. J. Heskett and H. Jones, both of Adelaide, S. Australia. Eng. Pat. 21,123, Sept. 14, 1897.

THE cupola, for melting the pig iron, scrap, &c., or the usual ore charge, and the converter, are connected together by a short firebrick-lined flue or channel, so that the metal from the cupola can run direct into the converter without loss of heat. Furthermore, the converter is closed, and the hot gases and particles of metal from the Bessemerising process therein, are conducted back through the same flue into the cupola, where the heat of the gases is utilised and the particles of metal caught, melted, and returned to the converter with the down-flowing metal. Consequently the melting down and converting may be carried on simultaneously and continuously in practically one furnace.
—A. W.

*Gold Ores, An Improved Process of Treating.* C. Wetherwax, New York, U.S.A. Eng. Pat. 21,483, Sept. 18, 1897.

IN this patent the author uses sodium or potassium silicate solution alone (this Journal, 1898, 768) with which to digest the gold ore at a steam heat, and at a steam pressure of 80 lb. per sq. in., so as to render the gold more susceptible to the action of solvents.—A. W.

*Slag Wool, Impts. in or connected with the Manufacture of.* W. P. Ingham, Middlesbrough. Eng. Pat. 23,670, Oct. 14, 1897.

SUITABLE slag from a series of blast furnaces is tapped into a ladle or kettle placed upon a truck running on lines connecting each furnace with a small annealing oven with a door. The ladle contains a tapping hole, plug, and spout at its base. The truck with the ladle is run into the annealing oven, which is suitably heated to sustain the fluidity of the slag. The tapping spout protrudes through a hole in the wall immediately over a steam jet, which blows the running slag, when tapped, in the form of wool, through an opening into a convenient chamber or wool-house situated in front of the jet.—A. W.

*Slag Wool, Impts. in or connected with the Manufacture of.* W. P. Ingham, Middlesbrough. Eng. Pat. 25,468, Nov. 3, 1897.

IN place of the annealing oven in the foregoing specification (Eng. Pat. 23,670), a cover, lined with non-conducting material, is lifted on to the truck and encases the whole of the slag ladle with the exception of the tapping spout. In the top of this cover is an aperture with a door, so that the ladle may be refilled with molten slag without the removal of the cover, thereby saving loss of heat. In other respects the operation is carried out as before.—A. W.

*Aluminium, A New or Improved Alloy of, and Process for making same.* P. E. Secretan, Paris. Eng. Pat. 24,267, Oct. 20, 1897.

THE alloy consists of 94—95 parts of absolutely pure copper, 4 to 5 parts of pure aluminium (*i.e.*, above 99·5 per cent., and free from sodium), not more than 0·0005 part of phosphorus, and not more than 0·01 part of nickel. The phosphorus is added to the molten copper in the form of a phosphide of copper or aluminium, and the nickel as an alloy of nickel and copper or aluminium. The resulting alloy is soft, resistant, and with a high limit of elasticity, both when cold and hot, and is recommended for locomotive furnaces, high-pressure and superheated steam tubes, torpedo boats, gun cartridges, &c.—A. W.

*Metal [Aluminium, &c.], Impts. in the Mode and Means for Annealing.* R. Wootton, Edgbaston, and G. B. Martin, Smethwick. Eng. Pat. 25,080, Oct. 29, 1897.

IN annealing metal, such as aluminium tubes or other articles, a fusible plug is placed in the pipe or tube conveying the heat or superheated steam from the furnace to the annealing chamber, so as to prevent the articles to be annealed from being melted, or heated to a temperature higher than the melting point of the plug.—A. W.

*Zinc, Impts. in the Treatment of Zinc-bearing Ores for Obtaining, and other Metals therefrom.* S. O. Cowper-Coles, London. Eng. Pat. 5943, March 10, 1898.

The zinc-bearing complex ore is finely crushed, and, after preferably mixing with 25 to 50 per cent. of zinc blende or other material to prevent binding, is dead-roasted to convert the zinc into sulphate and oxide. It is then lixiviated with water and the solution evaporated for sulphate. The residual ore is placed in a revolving screen, to separate it into fine and coarse portions, which are then deposited in different leaching vats and treated separately to the same process.

This consists of leaching with water containing 1 oz. of sulphuric acid to the gallon, whilst a current of electricity is passed through the mass, whereby oxides of zinc and copper are dissolved. The liquor is passed over a bed consisting of fragments of zinc and carbon, or iron and carbon, or both, to remove copper, and is used again and again with fresh acid in the leaching vat, until it contains from 20 to 30 oz. of zinc sulphate per gallon, when, to obtain the metal, it is electrolysed in cells having vertical revolving cast-iron discs for cathodes, and lead plates for anodes. The zinc sulphate solution remaining with the ore after draining, is washed out with water and added to, and evaporated by waste heat with the original water extraction.

The ore in the vats is next repeatedly leached with a solution of caustic soda (30 oz. to the gallon) to dissolve the lead, lime also being added to prevent waste of soda as carbonate or silicate. The sodium plumbate solution is then electrolysed to obtain lead, or treated with carbon dioxide to yield white lead, the carbonate of soda formed in the latter case being used to precipitate zinc carbonate from the original aqueous solution of sulphate in the production of zinc-white. Finally, the ore is treated with cyanide for any gold or silver that may be present.—A. W.

*Metals and Alloys, Process for Increasing the Density of, in Melting or Casting.* H. Schmidt, Saalfeld, Germany. Eng. Pat. 6861, March 21, 1898.

Wires are placed in the moulds or melting vessels, so that an electric current can be passed through the metal or alloy, when molten, and preferably until it has cooled or solidified. As a consequence the molecules of the metal are said to attract each other and pack themselves closer together, under the influence of the current, and thus render the casting denser.—A. W.

*Zinc Ores and other Volatile Metals in Blast Furnaces, Impts. in Method and Apparatus for Reducing.* A. M. G., Sebillot, Paris. Eng. Pat. 7904, April 2, 1898.

The blast furnace is of the water-jacket type, with the top hermetically closed, and with two rows of blast pipes, one much above the other, the outlet from the furnace being through a hole in its side situated between these two rows. To this hole is attached a small chamber containing charcoal to reduce any escaping carbon dioxide, through which the gases and vapours pass on their way to a condensing sloping flue with a tap hole, and thence to the powder-condensing chamber and shaft. The charge, which consists of zinc oxide and carbon, or zinc sulphide, iron oxide and carbon, is fed through a hopper at the top that can be closed. Air is forced downwards through the charge from the upper row of blast pipes, and upwards from the lower row, the vaporous products and gases meeting and passing through the outlet between, into and through the hot charcoal, which completes the reduction to carbon monoxide and zinc vapour, previous to the condensation of the latter.
—A. W.

# XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

## (A.)—ELECTRO-CHEMISTRY.

*Electrolytic Decomposition, Voltage necessary for.* E. Bose. Zeits. f. Elektrochem. 1898, **5**, [14], 153—177.

The author repeated and confirmed the observations of Glaser (this Journal, 1898, 462—464) on the electro-motive force necessary for the decomposition of water, and then extended his experiments in order to determine the points at which decomposition begins in different solutions. The general conclusions arrived at are as follows :—

The determination of the point of decomposition is preferably made with the help of a continuously increasing voltage, and not by a step-up arrangement. This is accomplished by winding the resistance spirally on an insulating drum which is driven at a suitable rate by a motor. The rate can also be varied by a system of change wheels, whilst the resistance itself is continuously tapped all along its length by a small loose pulley bearing on the wire and running on a shaft parallel with the axis of the drum. Deviations of the curves from the theoretical form, especially with very dilute solutions, are due to differences in the concentration of the electrolyte round the electrodes, and hence the most accurate results are obtained by using the smallest possible currents.

Extremely well marked points of decomposition are obtained on the deposition of solid substances, whilst if soluble substances, and especially if gases are liberated at the electrodes, the curves show a gradual rounding off near the point of decomposition instead of an abrupt change of curvature.

The influence of temperature and concentration on the electro-motive force required for the decomposition of silver nitrate solutions was also carefully investigated, and the results are given in tabular form. Solutions of copper salts show two characteristic cathodic points of decomposition, the lower one of which is probably due to the presence of $\overset{+}{Cu}-\overset{+}{Cu}$-ions. An oxygen electrode is strongly depolarised in solutions of mercurous salts. Silver is deposited from nitric acid solutions as a singly charged ion, $\overset{+}{Ag}$. In general, acids show just as many points of decomposition as there are ions split off on progressive dissociation. Both acids and alkalis show the anodic point of decomposition, observed by Glaser, at 1·08 volt, due to the presence of $^{-}O^{-}$ ions, and corresponding to the point of reversal of the gas battery.
—J. S.

### PATENTS.

*Storage Cells, Impts. in Electric.* [*Plates.*] C. P. Elieson, London. Eng. Pat. 23,489, Oct. 12, 1897.

The improved plates consist of several layers of perforated and corrugated sheet lead, arranged so that the corrugations of adjacent plates cross one another, and they are riveted together and united by autogenous soldering. Armatures or channel pieces of lead are laid across the layers and similarly united to them at the top and bottom. The exterior of the completed plate is given a coat of varnish or other material to protect it during "formation."—G. H. R.

*Electrodes* [*Carbon*], *Impts. in.* J. Hargreaves, Farnworth-in-Widnes. Eng. Pat. 25,519, Nov. 3, 1897.

The electrode is composed of pieces of carbon having either "trued" or rough surfaces, built up in the form of walls, floors, columns, or arches, retained in position by screws or wedges, with or without a layer of powdered plumbago inserted in the joints. These columns or arches may be supported by casting the walls of the cell about their extremities.—G. H. R.

*Negative Plates for Secondary Batteries* [*Lead-Zinc*], *Impts. in or relating to.* P. J. R. Dujardin, Paris, France. Eng. Pat. 28,242, Nov. 30, 1897.

Negative plates for batteries of the lead-zinc type are constructed of zinc or of cadmium, or of a suitable material covered with zinc or cadmium, and are divided into sections or conductors of small height in the shape of simple wires or of twisted or interwoven wires, or of combinations of strips either perforated or cellular, being protected at their ends by an enamel preventing the transfer of the mercury from one conductor to another, and being arranged horizontally for normal working. Supports not susceptible of amalgamation retain the plates at the proper distance apart and are provided with cavities to prevent undue electrodeposition at their ends, and to retain the mercury draining from the plates when they are placed vertically at the end of discharge. The porous layer of zinc or cadmium is capable of retaining so much mercury in its pores that in the normal working of the cell there is no apparent displacement of mercury.—G. H. R.

*Accumulator, An Improved Electrical.* P. Brandt, Maarssen, Holland. Eng. Pat. 29,180, Dec. 9, 1897.

The cell, which is contained in a glass or other suitable vessel, is formed of porous material, and is divided into a number of compartments by a corresponding number of sliding and removable partitions of porous clay. These compartments contain leaden combs which serve as electrodes, and are filled with a paste composed of a mixture of alkali sulphates and lead oxides with sulphuric acid and calcium sulphate, and also of organic lime-salts, the object being to ensure a higher useful effect without the employment of lead grids, and to avoid local action. The porosity of the plate may be increased by the addition of butyric or any other organic acid and glycerin.—G. H. R.

## (*B.*)—ELECTRO-METALLURGY.

*The Carlin Tube: Movable Electrolytic Connection for the Electric Current in Electrolysis.* A. von Dessauer. J. Chem. and Metall. Soc. of South Africa, 1898, **1**, 63—61.

The tube consists of an ordinary iron pipe, ½ in. in diameter, closed at its ends with screw plugs and filled with mercury. As many holes as there are electrodes are drilled in the top, from end to end as it lies horizontally, of sufficient size to allow the copper connecting wire to pass through easily and dip into the mercury. The electric main is affixed to one or both of the end plugs in a convenient manner. The pipe may also be made of copper or brass, when there will be a risk of amalgamation; or it may be replaced by a solid iron rod with one long groove or a series of small cavities to hold the mercury.

With this system of connection there is practically no loss of tension of current, experiment showing, with an 8-ft. tube with 52 cathode connections, a loss of only 0·3 per cent., as compared with a loss of 18·4 per cent. with the ordinary mercury trough connection. The intensity of current is evenly distributed, and the connection is rapid and perfect, and without the possibility of becoming dirty or oxidised. Furthermore, there is no heating of the mercury, nor loss by evaporation or spilling.—A. W.

*Nickel, Separation of, from Iron by means of Electrolysis.* H. Neumann. Chem. Zeit. 1898, **22**, 730—731.

*See under* XXIII., *page* 1073.

## PATENTS.

*Metals, A Process for the Electrolytic Separation of.* Dr. H. von der Linde, Crefeld, Germany. Eng. Pat. 25,24[illegible], Oct. [illegible], 1897.

The characteristic feature of this process for the separation of metals is the use of ammonium carbonate as the electrolyte, which dissolves only the metal to be separated. The process is particularly applicable to the stripping of platings of nickel, copper, or copper-nickel alloys from iron articles, the iron remaining unattacked.—G. H. R.

*Metallic Alloys, and Apparatus therefor; Impts. relating to the Electrolytic Production of.* [*Alkali Alloys.*] C. E. Acker, East Orange, U.S.A. Eng. Pat. 6637, March 18, 1898.

The apparatus consists of a hearth, supporting, by suitable lugs, a pan or partition and a chamber. A cover is provided, having an opening or openings for receiving an anode and feeding in the salt of the alkali or alkaline earth metal which it is desired to alloy with a heavy metal, such as lead, tin, zinc, &c., the latter in a fused state forming the cathode. A body of the salt rests on the cover and closes the openings, and the fused salt forms the electrolyte. The metal of the cathode is in fluid connection with the chamber, which is sealed against the access of the electrolyte, and in which the alloy rises until it reaches the desired level, when it is automatically drawn off, and by means of a circulating device, the flow of the molten metal is directed transversely below the electrolyte. The partition or pan is immersed in the cathode metal slightly below the true cathode surface, and has an extension towards the circulating device. Openings or channels are provided in or partly formed by the pan and its extension, whereby the molten metal may flow underneath and over the same, so that the alloy is stratified, the lighter portion flowing off from the surface, while the heavier is returned to the point where it takes up the lighter metal.—G. H. R.

## XII.—FATS, OILS, AND SOAP.

*Pumpkin-Seed Oil.* H. Poda. Zeits. f. Untersuch. Nahr.- u. Genussmittel, 1898, [9], 625—628.

This oil is largely used for culinary purposes in Austria and Hungary. Of the various edible oils it is next to olive oil in price. In Graz the price per kilo. of these oils is: Olive-oil, 80 krz. to 1 florin; pumpkin-seed oil, 70 to 80 krz.; sesamé oil, 60 krz.; cotton-seed oil, 40 krz.

In order to obtain specimens of undoubted purity, the author prepared the oil himself by cold expression from the seeds, and also from the seed-cake by cautious roasting and subsequent hot expression. The oil obtained by cold expression was of a greenish colour, with a faint red fluorescence; whilst that obtained by the second method (which is substantially the manufacturing process) was viscous, and its colour was brownish-green by transmitted light and deep red in reflected light.

The following analytical results were obtained:—

| —— | | Iodine Value (Hübl). | Saponification Value. | Butyro-refractometer at 25°. |
|---|---|---|---|---|
| Oil I.. | Cold expression | 124·49—123·91 | 189·31—189·51 | 70·2 |
| | Hot " | 123·89—122·85 | 189·24—189·37 | 71·4 |
| Oil II. | Cold " | 125·12—125·13 | 189·93—189·09 | 71·1 |
| | Hot " | 124·31—124·76 | 189·56—189·62 | 72·0 |

A number of specimens of the commercial oil were also examined, and the following were the limits of the constants of ten which the author regarded as genuine:—

| | | |
|---|---|---|
| Specific gravity | | 0·923—0·925 |
| Iodine value | | 122·76—130·68 |
| Saponification value | | 188·36—190·17 |
| Butyro-refractometer at 25° C. | | 70·0—72·5 |
| Melting point of fatty acids... | Commenced | 26·5—28·5 |
| | Ended | 28·4—29·8 |

The author considered that the variations in the iodine value were not so considerable, having regard to the fact that pumpkin-seed oil has considerable drying properties, and that during its preparation, the seeds are subjected to various degrees of drying and roasting. Moreover, the oil readily becomes rancid, and it is well known that the iodine value of an oil falls during the progress of rancidity.

The oils chiefly employed to adulterate pumpkin-seed oil are linseed, sesamé, cotton-seed, and rape oils. An addition of any of these can be detected by means of the refractometer, and by the iodine value and specific gravity, and the melting point of the fatty acids. Cotton-seed oil can also be detected by means of Becchi's test and sesamé oil by the Baudouin reaction, since pumpkin-seed oil gives a negative result with both tests.—C. A. M.

### *Olive-Kernel Oil, Contributions to our Knowledge of.* O. Klein. Zeits. angew. Chem. 1898, [37], 847—850.

According to Benedikt and other authorities, this oil differs from olive oil in having a sharp and bitter taste and a dark green colour, and in being readily soluble in alcohol and glacial acetic acid, on account of the large proportion of free acids which it contains. It is also generally accepted, among the manufacturers and dealers in olive oil, that the presence of kernel oil causes olive oil to undergo rapid decomposition. In the author's opinion, the curious difference in the character of the oil extracted from different parts of the same fruit must be attributed for the most part to the manner in which the oil is obtained. The fresh olives are crushed in mills, in which means are taken to prevent the simultaneous crushing of the stones. The crushed mass is packed in lenticular bags of esparto grass, which are stacked one upon another and subjected to a more or less complete expression. They are then thrown aside, and, after the lapse of some time, the residual mass (*bagasses*) is again ground in the mill, and pressed after the addition of hot water, or extracted with petroleum spirit or other solvent.

It is not surprising that the "kernel" oil thus extracted has a sharp and burning taste, for rapid oxidation occurs in the interval between the two pressings, and after two or three weeks the oil contained in the mass contains as much as 50 or 60 per cent. of free fatty acids. On standing, large quantities of oxy-fatty acids are deposited. The oil has a dark brown colour, through the oxidation of the chlorophyll. The analysis of an oil thus prepared gave the following results:—Specific gravity, 0·9277; iodine value, 71·57; saponification value, 190·5; free fatty acids, 71·12 per cent.

The author prepared specimens of undoubtedly pure kernel oil, free from any admixture with oil from the pulp of the fruit, both by warm and cold expressions. The analytical results obtained with these were: Specific gravity, 0·9186 to 0·9191; iodine value, 86·99 to 87·78; saponification value, 182·3 to 183·8; free acids, 1·00 to 1·78; refractive index, 1·4682 to 1·4688.

From these results it appears that the specific gravity and iodine value of pure olive-kernel oil are somewhat higher than is usually the case with olive oil. Only in exceptional cases, and with the oil from such varieties of olives as *sevilhana*, *cordovil*, and *mançanilha*, is the iodine value of olive oil so high. The saponification value is rather lower, and the quantity of free acids higher, than in olive oil, which, when obtained from sound, fresh fruit, varies in the latter particular from 0·4 to 0·8 per cent. The author attributes the difference to oxidation during the time which unavoidably elapsed between the removal of the endocarp from the kernels and the expression.

In order to determine whether the presence of kernel oil brought about alterations in olive oil, experimental mixtures of the pure oils were made in 1891 and 1892, and kept in well-stoppered bottles, excluded from light, until 1898. The samples showed scarcely any change in taste, colour, or smell; and, as is seen in the subjoined table, the amount of free acid showed hardly any increase:—

| Variety of Olives. | — | Free Acids. | |
|---|---|---|---|
| | | 1891 and 1892. | 1898. |
| | | Per Cent. | Per Cent. |
| Lentisquera .......... | Fruit pulp | 0·59 | 0·74 |
| " .......... | Pulp and kernel | 0·79 | 1·09 |
| Gallega............... | Pulp | 0·52 | 0·77 |
| " ............... | Pulp and kernel | 0·75 | 0·98 |
| " ............... | Pulp | 0·81 | 1·26 |
| " ............... | Pulp and kernel | 0·53 | 0·61 |

Hazura found olive oil to contain 28 per cent. of glycerides of solid fatty acids, 67 per cent. of olein, and 5 per cent. of linolein. According to Benedikt, the saturated fatty acids consist of stearic and palmitic acids, with traces of arachidic acid. The author found a specimen of olive-kernel oil to contain 9·7 per cent. of solid fatty acids, which, from their melting point and molecular equivalent, he concluded to consist of a mixture of 40 per cent. of stearic acid and 60 per cent. of palmitic acid. Arachidic acid was not detected. The liquid fatty acids examined by Hazura's method were found to contain oleic and linoleic (linolic) acids.

The general conclusions arrived at by the author from the results of this research were:—

1. The characteristics hitherto assigned to olive-kernel oil are really those of *bagasses* oil.
2. The physical and chemical characters of pure olive-kernel oil are very similar to those of olive oil.
3. Olive-kernel oil is as stable as olive oil, provided it is kept with proper precautions.
4. It can be mixed with olive oil without any ill effect, and there is no reason why the stone and the fruit should not be crushed together.—C. A. M.

### *Lard, American; Notes on.* F. Voigtländer. Zeits. angew. Chem. 1898, [37], 857—864.

The importation of American lard into Germany is steadily increasing, as is shown by the following figures, published by the Hamburg Chamber of Commerce:—

| — | Kilos. | Value in Marks. |
|---|---|---|
| 1895 | 757,485 | 51,055,000 |
| 1896 | 873,758 | 43,111,000 |
| 1897 | 931,813 | .. |

Comparing the characteristics of American and German lard, the author states that, generally speaking, the American product is softer and requires considerable working-up to prevent the separation of lard-oil. German lard, after melting, solidifies with a characteristic stellar formation on its surface—an appearance which is not often observed with American lards, owing to their more fluid nature.

The iodine value of American lard is high, both on account of the large proportion of lard-oil which it contains, and of the more unsaturated nature of that lard-oil. German lard contains about 50 per cent. of lard-oil, with an iodine value of from 70 to 75, whilst American lard contains about 60 per cent., with an iodine value of about 88. Many of the semi-fluid lards in the German market are of Russian or Servian origin, and contain as much as 90 per cent. of lard-oil. The author gives, in a table, the following iodine values of the lard, rendered by himself from the original unsalted tissue of different parts of Chicago pigs:—Neck, 65·0—66·6; back, 61·5—65·1; paunch, 59·0—63·0; leg, 68·8; ham, 66·6—68·4; mean results of a mixture in equal proportions, 65·5. These results are in close agreement with the similar series of figures given by v. Raumer and by Mans. This difference in the character of American and German lard is largely to be attributed to the different food given to the pigs. It was also formerly attributed to the different method of manufacture, fat from all parts of the body being employed; but the author states that during the last few years this has been discontinued. The lard is now principally made from the kidney and intestine fat, whilst the finer kinds of tissue are more profitably used in the margarine industry. Only in very high-priced lards is the back and side fat employed.

The lard exported from America is sold in three qualities, *viz.*, Choice Western Steam (rohes Schmalz), Pure Lard (raffinirtes Schmalz), and Neutral Lard (Flohmenschmalz). Steam-lard has a slightly burnt flavour, and the author attributes its superior keeping qualities to the presence of traces of burnt substances. This taste and smell are absent from lard rendered at lower temperatures, or bleached. Pure lard is steam lard refined by the Hungarian process. The melted fat is mechanically stirred in closed vessels until solidification commences, when a certain proportion of solid lard is added. An addition of from 5 to 8 per cent. of lard "stearin" is only made in the very best kinds. Neutral lard, consisting of the better kinds of fat, is used up almost exclusively in the margarine industry. Owing to the falling price of lard, the addition of beef or mutton stearine has of late years been but little practised, though it is still met with when a product with a low iodine value has been required. In the author's experience, steam-lard, refined as described above, keeps its consistency through the summer without any addition, although lard-oil soon separates from untreated steam-lard. Compound lards, containing cotton-seed oil stearine and by-products of the margarine industry, although extremely common in the last decade, are now met with relatively but seldom. The author throws doubt on the reliability of many of the tests which are often looked upon as conclusive as to the presence of foreign substances in lard. With regard to the iodine value, he has examined 100 samples of American lard, of which 88·5 per cent. had an iodine value of between 61 and 66, and 11 per cent. above 61. Dieterich, who examined 112 samples of German lard, found that 71·7 per cent. had an iodine value of between 48 and 53, whilst only 2 per cent. gave as high a figure as 64. The author deprecates the fixing of any definite limits of the iodine value, which, in his opinion, renders adulteration more easy. He states that lard with an iodine value of 67, contains foreign substances much less frequently than lard with an iodine value of 64. In his experience, nowhere is beef stearin more frequently found in lard than in Bavaria, where 62 was formerly adopted as the lower limit for the iodine value.—C. A. M.

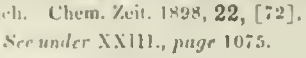
*Fats, Direct Oxidation of.* M. Hanriot. Comptes Rend. 1898, **127**, [16], 561—563.

By passing ozone through melted, purified fat, the author has shown that a considerable quantity of oxygen is absorbed. He has, however, not been able to detect any reducing body in the product. Acetic acid and probably butyric acid are formed, but not oxalic acid.—J. S.

*Oleic Acid, Alteration of, on long Keeping.* M. v. Sénkowski. Zeits. physiol. Chem. **25**, 131—139; Chem. Centr. 1898, **2**, [13], 701.

It has been previously shown by Salkowski (Festschr. zu Virchow's Jubilaum, 1891, 19) that oleic acid, on keeping for a long time, is partly changed into a crystalline modification melting at 48° C. The author has come to the conclusion, after determining the acid number, ether number, saponification number, iodine number, acetyl-acid number, acetyl-ether number, and acetyl number, that the substance consists of 32·1 per cent. of oleic acid, 8·3 per cent. of stereolactone, 20·6 per cent. of hydroxystearic acid, and 39 per cent. of an unknown substance. The author considers it probable, in view of the fact that the oleic acid absorbs water on long preservation, that it is first converted into a lactone according to the following equation:—

$$C_nH_{2n+1}.CH:CH.C_nH_{2n}.COOH =$$



$$C_nH_{2n+1}.CH_2.CH.O.C_nH_{2n}CO$$

and the lactone, by absorption of water, into the corresponding hydroxyacid.—A. S.

*Candles, Manufacture of; Impts. in the.* N. A. Alexanderson, Stockholm. Eng. Pat. 7671, March 30, 1898.

*See under* II., *page* 1031.

*Beeswax and its Adulterants, The Iodine Value of.* K. Dieterich. Chem. Zeit. 1898, **22**, [72], 729.

*See under* XXIII., *page* 1075.

### PATENTS.

*Purifying Oily Fluids, and for Separating the Water therefrom; Apparatus for.* J. Schuschl and H. Hinterberger, Pola, Austria. Eng. Pat. 25,570, Nov. 4, 1897.

The apparatus claimed consists of a closed cylindrical metal vessel divided into compartments by means of arched perforated floors or partitions. The oil to be purified enters at the bottom of the vessel and impinges upon inclined plates in the lower chamber, where the coarser particles are separated. Rising into the second compartment, which is filled with calcium chloride, the oil is dried, and then passes up into the next, this chamber being packed with asbestos. After filtering through the asbestos, the oil is drawn off from the top of the vessel. The partition above the asbestos is rendered movable by means of a screw spindle passing through the cover of the vessel, and serves for the purpose of compressing the filtering materials.—W. P. S.

*Fish Offal Digesters, Impts. in, and Means of Supplying to.* T. Watts, Vancouver, British Columbia. Eng. Pat. 16,214, July 26, 1898.

Offal is supplied to a series of hopper tanks with sloping bottoms from a conveyor chute provided with trap-doors. The tanks have contracted outlets at the bottom, fitted with vertically sliding trap-doors, which are opened and closed by a rack and pinion adjustment. The offal passes through these, and then along inclined, movable ducts, pivoted to the tanks, to the digesters. The latter have conoidal bottoms, a closable orifice at the top, a sloping bottom supported just above the cone, and a closable door above this for the removal of the residue; further, there are ducts for injecting steam, one at the top and one at the bottom of each digester. By closing the lower steam duct and increasing the pressure in the upper one, the dissolved gelatin and the oily and aqueous matter, are driven to the lower bottom, from which they are ejected through a pipe.—E. H. T.

*Refining Oils, Impts. in the Method of.* M. Ekenberg, Göteburg, Sweden. Eng. Pat. 17,087, Aug. 8, 1898.

For neutralising the free fatty acids of oils, the patentee uses a solution of an alkaline hydrate in glycerin. Sufficient of this is added, and the mixture is heated to 100° C. or more, when the soap formed readily separates from the oil. It is necessary to add so much glycerin that the soaps formed shall contain from 5 to 10 per cent. of their weight of this substance.

With the cheaper kinds of oils, the addition of glycerin may be avoided by adding the alkali in such quantity that, besides neutralising the free acid, a portion of the oil itself is saponified, glycerin being thus produced.—W. P. S.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

*Paints, Permeability of, to Water and Gases.* J. Treumann. Chem. Rev. Fett- u. Harz-Ind. **5**, [7], 135—137; [8], 153—156. (See also this Journal, 1898, 359 and 608.)

Spirit varnishes and paints are less permeable than oil paints owing to their content of resins, and as this quality varies directly with the proportion of oil-paints present, those spirit-paints that are poor in oil are the best suited for resisting the influence of water.

For his method of testing permeability with hydrogen sulphide, the author claims that a paint which will stand this test will prove more resistant towards water, oxygen, &c. than one which does not stand the test, though the proportionate resistance is not necessarily the same.

Generally, he finds that for a given varnish, the permeability of oil paints to hydrogen sulphide varies considerably according to the pigment employed, lampblacks and ochres being much more permeable than white lead or minium, the least affected of all being zinc white. In all cases the influence of the aqueous solution of the reagent was greater than that of the gas. A coating of varnish or spirit-varnish increases the resistance of the paint towards the gaseous reagent, but only the spirit-varnishes poor in oil have the same effect towards the solution; and the best results are obtained by spirit-varnish paints.

Another important factor, the absence of which may nullify the advantages derived from impermeability, is the *durability* of a paint; and it is the lack of this quality which renders oil paints unsuitable, in most cases, for use under water, whilst the spirit-varnishes and paints poor in oil are better adapted for this purpose than for use where they are exposed to the air.

All things considered, the red-lead paints have, in general, the greatest protective power as grounding paints—notwithstanding their permeability—for exposure to the air, and should only be superseded when special requirements necessitate such a course.

Referring to Lösner's steam test (this Journal, 1898, 359), the author points out that the high temperature of this test renders it unsuitable for fusible resins, and also that (12 months) old coatings of paint would appear, by their behaviour under this test, to be less permeable than such as are fresh. Moreover, Finkener's results indicate that the presence of manganese and phosphorus in iron predisposes the metal to rust in presence of steam or hot water, and thus a fresh element of uncertainty in the test is revealed. In addition, his own experiments with the Lösner test failed to distinguish the slightest difference in permeability between minium and zinc white paints, whereas a considerable divergence in this respect is known to exist.—C. S.

## (C.)—INDIA-RUBBER, &c.

*India-Rubber, Sources of Commercial.* Cantor Lectures, D. Morris. J. Soc. Arts, 1898, **46**, 745—760, 761—772, 773—782, and 785—796.

The first record of india-rubber was made soon after the discovery of the new world by Columbus, who, during his second voyage, noticed the inhabitants of Hayti playing with balls made from the gum of a tree. Torquemada mentions for the first time the name of a rubber tree—*Castilloa elastica.* La Condamine, in 1735, gave the first accurate information concerning the tree yielding the Para rubber, and Fresnau and Aublet described the rubber trees of French Guiana. Howison, in 1798, discovered india-rubber in Asia, at Prince of Wales Island, from the plant now known as *Urceola elastica*, and in 1810 Roxburgh traced the Assam rubber tree, *Ficus elastica.* The extensive use of india-rubber began when, in 1847, Goodyear in America and Hancock in this country discovered the vulcanising process by means of sulphur. The progress of the rubber industry may be inferred from the following table of imports into England:—

| Year. | Tons. | Year. | Tons. |
|---|---|---|---|
| 1830 | 23 | 1879 | 7,530 |
| 1840 | 334 | 1888 | 11.018 |
| 1850 | 381 | 1897 | 19,816 |
| 1870 | 7,606 | | |

There has been of late years a very considerable increase in the quantity of rubber imported from British possessions. The annual rubber imports of the principal European countries, and also of America, are as follows:—

| Countries. | Year. | Quantity. | Value. |
|---|---|---|---|
| | | Tons. | £ |
| Great Britain....... | 1896 | 21,558 | 4,991,122 |
| France ............ | 1896 | 5,177 | 1,111,256 |
| Germany ........... | 1897 | 8.436 | 2.320,150 |
| Belgium............ | 1897 | 2,236 | 545,835 |
| Holland ........... | 1897 | 1,072 | 141,667 |
| Austria-Hungary ... | 1897 | 2,109 | 811,415 |
| U.S. America ....... | 1897 | 18,821 | 4,514,587 |

The vessels in which the rubber yielding milk or latex is contained are generally found in the inner layers of the bark. They are most abundant in the older tissues, and gradually spreading to the leaves and flowers. There is no milk obtainable from the wood. The latex contains, besides rubber, certain salts, proteids, and resinous matters. The methods of coagulating vary in every country and every district, and will be referred to later on. Quite recently experiments have been made in Trinidad to produce the rubber from the latex according to a method invented by Messrs. Howard and Biffen. In this method the latex, after being mixed with 50 per cent. of water, and placed in a machine very similar to a cream separator, is then rotated rapidly for a few minutes. The machine is then allowed to come to rest gradually, when the rubber is obtained floating on the top of the liquid in a thick white mass, all impurities and dirt being found at the bottom.

The "fine Para" of the present day is still the best rubber in commerce. Similar rubber is also obtained from Peru, Bolivia, and Venezuela. The produce of the Central American Castilloa trees is known as Nicaragua, West India, Honduras, Mexican, Guatemala, Panama, and Peruvian "Caucho." Ceara rubber is a dry rubber, very elastic, and free from stickiness. Pernambuco or Mangabeira rubber is a "wet" rubber, and is also obtained from Bahia, Southern Matto, Grosso, and Uruguay. African rubbers are produced throughout tropical Africa. From the West Coast come: Gambia, Sierra Leone, Lagos, Gold Coast, Congo ball, Thimbles, Calabar, Cameroon, Batauga, Liberian. On the East Coast are produced: Mozambique ball, Tanraga, liver, unripe; Madagascar, good and fine, and black. The Asiatic rubbers from Assam and Rangoon are mostly scrap rubber. Java, which resembles them, is not quite so good. Borneo rubber is very spongy and porous. New Guinea rubber, when free from bark and impurities, is good. There are several hundred plants with a milky juice, but, so far, less than 50 of these contain sufficient caoutchouc to repay the cost of extraction. The plants now yielding commercial rubber may be classed under four natural orders:—

*Rubber-producing Plants.*

| | Species. |
|---|---|
| I. Spurges (Euphorbiaceæ)................. | 14 |
| II. Nettles (Urticaceæ) ..................... | 4 |
| III. Dogbanes (Apocynaceæ).................. | 27 |
| IV. Asclepiads (Asclepiadaceæ)............... | 1 |
| Total................................ | 46 |

*Para rubber* derives its name from the town and province of that name in Brazil, and is principally the product of *Hevea brasiliensis* (syn. *Siphonia brasiliensis*). The trees grow to a height of as much as 60 ft., they flower in January and February, and the seeds ripen in July or August. Trees are tapped if they have a circumference of 18 to 24 ins., and the tapping takes place from June to December. The tree does not appear to suffer from the tapping, and will yield milk in abundance for 30 or 40 years, but it dies if in the tapping, the wood of the tree is wounded in the slightest degree. The collected latex is coagulated by dipping into it round-bladed paddles and then holding the latter in the smoke ascending from burning Urucuri nuts. Coagulation takes place instantly, and these two dipping and coagulating operations are repeated until the rubber is about 2 ins. thick, when it is cut off the paddle. There are practically only two grades of Para rubber, *viz.*, Fine Para and Negrohead. The latex of *Hevea brasiliensis* is alkaline, and its coagulation through the smoke of the Urucuri nuts is, no doubt, due to the action of the acetic acid and creosote in this smoke. The latex of *Castilloa elastica* is acid, it is not acted upon by the above-named smoke, but coagulated by means of alkalis. The yield varies from 2 up to 12 lb. per tree. In 1897 the distribution of Amazonian rubber was as follows:—

| | Tons. |
|---|---|
| United Kingdom..................... | 8,843 |
| France............................. | 2,010 |
| Italy............................... | 65 |
| U.S. America ....................... | 11,626 |
| Total ..................... | 22,544 |

A table is given indicating graphically the price per lb. of fine Para rubber within the period from 1877 to 1897, in London and Liverpool.

A new form of Hevea rubber has lately appeared under the name of Mollendo rubber. This is shipped from the port of Mollendo, on the Peruvian coast, to which it is brought by rail from Lake Titicaca. Bolivia is rich in Hevea trees. Para rubber trees are now also well established in various parts of India, and in most of the eastern British possessions.

*Central American rubber* is derived from *Castilloa elastica*, allied to the Breadfruit tree. This rubber tree grows to a height of from 160 to 180 ft., and attains a circumference of stem of as much as 15 ft. It has never been observed east of the Andes, but otherwise it has an extensive geographical range, and is evidently capable of existing under considerably varied climatic conditions. The tree is fit to be tapped when about seven to ten years old. The best season for tapping is after the autumn rains, from October to the end of January. The latex is coagulated by adding to it the aqueous solution obtained from the stem of the moon plant (*Calonyction speciosum*). The yield of rubber per tree during a season amounts to about 16 lb. According to consular reports from all parts of Central America, the rubber exports from these districts are rapidly decreasing, owing to the ruthless cutting down, instead of bleeding, of the trees by the collectors.

*Ceara rubber* is derived from *Manihot glaziovii*, a tree growing from 30 to 50 ft. high, with a stem about 8 to 20 ins. in diameter. It occurs chiefly in the very dry, arid part of North-east Brazil. From 200 to 300 tons of Ceara rubber are annually imported into this country. There are three grades of it in commerce, the first of these coming next to Para in value. The collectors tap the trees and allow the exuded latex to dry spontaneously, when it is pulled off and rolled into balls, or put into bags in loose masses. Extensive experiments have been made to cultivate this very hardy tree in Ceylon, but it appears that the yield of rubber proved unsatisfactory.

*Mangabeira rubber* is obtained from *Hancornia speciosa*, a tree somewhat resembling the weeping birch. It is found nearly all over Brazil. The latex is coagulated with alum, and the coagulated lumps are then dried in the sun.

*Matto Grosso rubber* is exported from the Brazilian state of this name. It is doubtless yielded by one of the numerous species of Hevea.

*Colombian rubber* is produced by several plants, the principal being *Castilloa elastica*. A very excellent quality, second only to Para, is produced in the Colombian Andes at elevations of 6,000 to 8,000 ft. The plant yielding this rubber has been identified as one of the numerous varieties of *Sapium biglandulorum*

*Venezuelan rubber* is yielded by a species of Hevea, but there are doubtless other rubber-yielding trees to be found in that country. It has lately been shown that immense tracts of that country are covered by a species of Manihot.

*British Guiana rubber.*—There are several species of Hevea, of which *H. pauciflora* and *H. dubia* have been described, growing in British Guiana.

*Jamaica rubber.*—An excellent quality was shown to be obtainable from *Forsteronia floribunda*, a climbing plant.

*African rubber.*—The development of the rubber industry in the equatorial parts of Africa is one of the most remarkable incidents in recent years. As regards the world's commerce in rubber, Africa now occupies the second place. The plants yielding African rubber belong to the genera Landolphia and Kickxia, but recently a new and singular rubber-yielding plant of a semi-herbaceous character has come into prominence in the Congo State, where it covers enormous tracts of dry and grassy country. The chief districts of export on the West Coast are Senegambia, Sierra Leone, Gold Coast, Lagos, Niger Territory, Congo State, Gaboon, Loanda, and Benguela. On the East Coast, Mozambique, Madagascar, Zanzibar, Nyasaland, and German East Africa. The value of the imports of African rubber into the United Kingdom amounted to 1,652,812*l.* in 1896.

*Lagos silk rubber* is obtained from *Kickxia africana*, a tree widely distributed in West Africa. The latex obtained from this tree is coagulated either by allowing it to dry spontaneously in a hollowed-out trunk (cold process), or by boiling (hot process). The rubber obtained by the latter process is inferior to the product yielded by the cold process.

*New root rubber* is obtained from *Carpodinus lanceolatus* and allied species, especially *Clitandra henriqueziana*, both belonging, like the Landolphias, to the natural order Apocynaceæ. The interesting point is, that these are neither trees nor shrubby climbers, as other rubber-yielding plants in tropical Africa. They are extensively distributed in tropical Africa. Their stems contain rubber, but the larger quantity is at present obtained from the creeping underground stems (rhizomes). From these the rubber is obtained by rasping them in water and then boiling.

*Soudan.*—The French in the Soudan are developing the rubber industry of that region. The value exported in 1896 was 6,000*l.*

*Gambia.*—The rubber exports from this district are greatly fluctuating, owing to the reckless destruction of the vines.

*Sierra Leone.*—Large tracts of forest land in this colony are covered with rubber trees. The most valuable of these is *Landolphia owariensis*. The next is *L. florida.*

*Gold Coast.*—The development of the rubber industry in this colony is almost entirely due to the initiation of Sir Alfred Moloney. The value exported in 1896 was 313,816*l.*

*Lagos.*—The rubber industry of Lagos was started in 1894. In 1895 the exports amounted to 2,263 tons, of the value of 269,893*l.* Latterly the exports show a greater falling off, owing to the excessive bleeding of the trees.

*Niger Protectorate.*—The exports of rubber are assuming importance.

*Congo State.*—More than one-half of the external trade is in rubber. The value exported in 1897 was 114,000*l.*

*Loanda.*—The Portuguese possessions in South-west Africa yielded a large share of African rubber of good quality, but very dirty. The value of the exports in 1896 amounted to 639,532*l.*

*East Coast rubber plants.*—On the mainland of the East Coast rubber is yielded almost exclusively by climbing species of Landolphia, of which four have been discovered in this part of Africa. The rubber industry in this part of Africa has reached very considerable dimensions, but is seriously threatened by the rapid destruction of the rubber vines.

*Madagascar rubber.*—The principal rubber-yielding plant is *Landolphia madagascariensis*, a climbing plant of considerable size. It produces a very large quantity of excellent rubber. During recent years a considerable rubber industry has arisen in the southern parts of Madagascar, due to the discovery of a new rubber plant, a species of Tabernoemontana. But already the results of the reckless cutting down of the plants by the collectors are being experienced.

*Adriatic rubbers* are chiefly derived from Assam through Calcutta; from Upper Burmah through Rangoon; from the Straits Settlements through Penang and Singapore; from Sumatra, Java, Borneo, and New Guinea.

*Assam rubber* is the product of the familiar *Ficus elastica, Roxb.*, which attains to the dimensions of a huge forest tree. This rubber was at one time of excellent quality, which of late has much deteriorated, owing to careless collecting, which also led to the most outrageous destruction of trees. In 1873 the Bengal Government started regular plantations of Ficus elastica in Assam, and with slight interruption continued this movement until the present time. The amount of rubber obtained shows a singular irregularity year by year, coming as high as 36 lb. per tree in one year, and falling to a little over 2 lb. in another. Good rubber is also obtained from *Cryptostegia grandiflora*, probably a native of Madagascar, but now naturalised in India.

*Upper Burmah or Rangoon rubber* is for the most part also produced by Ficus elastica, although there are probably other sources of india-rubber in Burmah. One promising species seems to be Urceola (Chavanneria) esculenta, Benth., a climbing forest plant.

*Penang and Singapore rubber* is largely obtained from *Willughbeia firma, Bl.*, a large climber, the sap of which is coagulated by the addition of salt water. From *Willughbeia flavescens* a fair quality of rubber is also obtained, but the rubber yielded by *W. coriacea*, a jungle climber, is only used for mixing with other rubbers. Leuconotis eugenifolius probably yields some of the rubber from the Straits Settlements. There is no doubt, however, that some of the Malay rubber is obtained from Ficus elastica.

*Sumatra and Java rubber* appear to be obtained chiefly from Ficus elastica. Encouraging experiments in the cultivation of F. elastica have been made in the province of Kranoy in Java.

So far little or no rubber comes from the Philippine Islands, although there have been observed some 16 genera of the order Apocynaceæ. In Cochin China, a stout climber, *Parameria glandulifera*, which appears to be widely distributed through the Eastern tropics, is stated to yield an excellent rubber. From consular reports it would appear that rubber trees exist also in Formosa and Central China (*Eucommia almoides*), but very little is known about them.

*Borneo rubber* is the product of several creepers. Those yielding the best qualities are *Willughbeia firma*, *Leuconotis eugenifolius*, and a third species, probably also a Willughbeia. The exports of Borneo rubber in 1897 amounted to 768,320 lb.

*New Guinea rubber* is the latest addition to the list. The plants so far have not been determined. In 1896 the exports of this rubber, which appear to be of excellent quality, amounted to 67,000 lb.

In the Fiji Islands several rubber plants have been observed, of which *Tabernoemontana thurstoni* and *Alstonia plumosa* appear to be the most interesting, but at present they are little used by the natives.

In view of the possible falling off in the supply of commercial india-rubber, it has often been suggested that efforts should be made, in the countries where the trees are found wild, to replant exhausted areas. In various districts of South America and Central America, cultivation experiments have been made, more or less successfully, showing an annual revenue of from 30*l.* to 40*l.* per acre. The climbing habit of East and West African rubber plants renders them unsuitable in regular plantations, but they should be carefully protected in their present localities. The native rubber plants of Asia, with the exception of *Ficus elastica*, do not offer favourable conditions for systematic cultivation. The rubber trees offering the best inducement for regular cultivation in other than their native countries, arranged in the order of their value, from a cultural point of view, are: *Hevea brasiliensis*, *Castilloa elastica*, *Kickxia africana*, *Manihot Glaziovii*. Hevea requires a hot and moist climate, and a deep rich soil. A 20 years' experience in Ceylon shows a return of 20 per cent. on the capital invested. Castilloa trees have not succeeded on a large scale, so far, in any part of the Old World, but deserve further trial. Kickxia rubber trees probably grow more vigorously than any other rubber tree, and on the coast lands in West Africa, this tree should be adopted in preference to any of the American trees. Manihot trees were tested with great energy in Ceylon, but the yield was disappointing.—C. O. W.

*Leather and Caoutchouc, Substitute for.* Brandon et fils. Rev. Prod. Chim. **1**, [10], 148.

The caoutchouc substitute consists of: wood pulp, 50; caoutchouc solution, 25; chalk, 25; lampblack, 2½; sulphur, 2½ parts; whilst the leather substitute, with the above proportions of the two latter ingredients, contains 60 parts of wood pulp, 20 of caoutchouc solution, and 15 of chalk.

The caoutchouc solution and sulphur are first mixed together, then the chalk is added—to protect the caoutchouc from the injurious influence of heat—and the mass is incorporated thoroughly with the wood pulp previously beaten up into a flocculent condition. The mixture is formed into sheets, exposed to a temperature of between 93° and 107° C.; and, after the caoutchouc solvent has evaporated, is rolled in order to ensure greater solidity. —C. S.

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Tannin in the Oak Bark of the Geneva Canton.* W. Borel and H. W. de Blonay. Arch. Soc. phys. nat. Genève, **6**, [4], 160—168; Chem. Centr. 1898, **2**, [14], 796.

The author has tried to ascertain at which age the trees give bark richest in tannin, and what influence the thinning of the trees exercises on the formation of tannin. For the determination of the tannin, Jean's method was used, the extract being titrated with iodine, before and after extracting the tannin with hide powder. The difference between the two amounts of iodine is multiplied by a factor which is ascertained from the amount of iodine used with pure digallic acid. The bark examined was obtained from the stems of *Quercus pedunculata*, which grows at a height of 430 m. on heavy glacier clayey soil. A strip was cut off, from all the trees, all round the stem at a height of 1 metre above the ground.

The average tannin content decreases with the age of the tree, and increases again when the age is over 50 years. The average tannin content in the case of the stems amounted to:—

| | Per Cent. | | Per Cent. |
|---|---|---|---|
| 5 years............ | 8·40 | 34 years............ | 5·35 |
| 20 „ ............ | 7·15 | 41 „ ............ | 5·05 |
| 25 „ ............ | 6·60 | 50 „ ............ | 5·60 |
| 32 „ ............ | 6·30 | 51 „ ............ | 5·90 |

If, however, the total weight of bark obtained be also taken into consideration, then a plantation of 1 hectare between 1 and 60 years yields more tannin the older the trees are. The maximum annual production of tannin is attained after 26 years, with 23·8 kilos. per hectare. As, however, the value of the wood has also to be considered, it is better to allow the trees to become old before felling them.

The thinning of the trees exercises no influence on the tannin content, but causes an increase of 25—40 per cent. on the weight of the bark. The trees felled for the clearing contain, in the bark, considerable quantities of tannin, and should therefore be barked.—A. S.

*Leather and Caoutchouc, Substitute for.* Brandon et fils. Rev. Prod. Chim. **1**, [10], 148.

*See under* XIII. C., *preceding column.*

### PATENTS.

*Plastic Compositions.* C. Ives, London. Eng. Pat. 2887, Feb. 1, 1898.

A patent for the manufacture of articles of plastic composition from gelatin, glycerin, and potassium bichromate, either alone or with ground cork, or similar material, as a basis. Good results are obtained by grinding together 1 part of bichromate and 10 parts of gelatin, to which is added anhydrous glycerin. The whole, when mixed, is moulded to any desired shape, and is subjected, under pressure, to a temperature of 300° F.; the mass becomes soft, and is thus readily moulded to any form, and at the same time produces a composition elastic, tough, and insoluble.—J. G. P.

*Treatment of Skins for Use in Furriery and Tawing Operations, Impts. relating to the.* F. and G. de Rechter, Brussels. Eng. Pat. 14,398, June 29, 1898.

By means of formic aldehyde or its polymers, unhaired skins are treated so that the hairs or feathers thereon are permanently retained without affecting their quality in regard to the work in connection with furriery and tawing operations. This may be carried out by either submitting the skins to the vapours of formic aldehyde or by immersion in a solution of 1 part in 20 of formalin, either in water or alcohol.—J. G. P.

*Tanning Hides and Skins, An Improved Method of.* S. Thomson and J. A. Todd, of Titusville, Pa., U.S.A. Eng. Pat. 16,419, July 28, 1898.

Hides and skins which have been prepared for tanning in the ordinary way are treated with formaldehyde gas in a closed chamber until the gelatinous mass is converted into leather—usually from 12 to 48 hours.—J. G. P.

## XV.—MANURES, Etc.

*Nitrogenous Manure, Production of, from Indigenous Sources.* J. König. Neue Zeits. Rübenzucker-Ind. 1898.

Nitrogen, for manure, may be obtained, without importation, by utilising the nitrogen-assimilating power of legu-

[illegible] plants and green-manuring, by more extensive [illegible] of human and animal excrements, and by avoiding loss of ammonia in gasworks and coke-furnaces. It is to the last source that attention is chiefly directed, as it would be possible to furnish a great portion of the nitrogen required in Germany for manure in the form of ammonium salts.

During the four years 1895 to 1898 the amounts of nitrogen actually produced in Germany in the form of ammonia were 10,800, 15,000, 18,000, and 19,600 tons, but it is estimated that at least 230,000 tons of ammonium sulphate, containing 46,000 tons of nitrogen, could be produced annually.

The objections to the increased production of ammonium salts are the depression of price (ammonium sulphate was 17s. per 100 kilos. in 1897) and the common idea that nitrogen in this form is less valuable than in the form of nitrates. For spring manuring, nitrates are preferable to ammonia, since most plants probably only assimilate nitrogen in the form of nitrates. On the other hand, nitrates have the disadvantage that they are liable to render the soil difficult to work. Nitrates are also unsuited for autumn manuring, owing to loss in drainage; and when used in large amounts injure the quality of the crops.

With regard to ammonium salts, it is pointed out that their use in the case of soils poor in lime involves the more or less frequent application of marl, lime, or basic slag. The last two must, of course, not be applied with the ammonium salts; marl and ammonium salts may be applied simultaneously, but should be at once ploughed in.

The relative value of the nitrogen of ammonium sulphate, compared with that of nitrate of soda, has been shown by experiments to be 90 or 95:100. But when the disadvantages of nitrate of soda are taken into account, both forms of nitrogen may be considered equal. Ammonium salts, containing 20 per cent. of nitrogen, and costing 17*s.* to 18*s.*, are, therefore, at least equal to nitrate of soda, containing 15 to 15·5 per cent. of nitrogen, and costing 15*s.* to 16*s.* per 100 kilos.—N. H. J. M.

*Nitrates, Assimilation of, and the Effect of Different Nitrates.* W. Schneidewind. J. Landw. 1898, **46.**

The results of experiments with sugar-beet manured with nitrate of soda and nitrate of potash respectively, indicated that nitrate of soda gave a greater yield of dry matter and sugar in the roots, whilst nitrate of potash produced a greater amount of leaf. The more favourable effect of nitrate of soda is attributed to its greater solubility and diffusibility.

In experiments with oats manured with different nitrates, the yield of grain was greatest with the magnesium salt; nitrate of soda gave the next largest amount of grain, then nitrate of lime, and last nitrate of potash. Magnesia has a special *rôle* in the production of grain, but it is supposed that the better result of nitrate of soda, as compared with the potassium salt, is due, as in the case of sugar beet, to the greater solubility of sodium salts, and to the consequent more rapid supply of nitrogen, and phosphoric and sulphuric acids, to the plant. Straw production was least with magnesium and greatest with potassium nitrate.

The composition of the grain did not vary to any extent, except in the case of the magnesia plants, the grain of which was somewhat richer in magnesia. Even in this case most of the magnesia, taken up as nitrate, was deposited in the straw.

It is not supposed that soda can in any way replace potash in its functions connected with the migration of starch and sugar.—N. H. J. M.

*Humic Matters, Natural; Action of Lime and Calcium Carbonate on.* G. André. Comptes Rend. 1898, **127,** [13], 416—418.

Vegetable soil and heath soil, mould and peat, were subjected to the action of lime, calcium carbonate, and water respectively for 15 days at 100°, and the nitrogen evolved as ammonia, as well as the nitrogen rendered soluble, determined. The ammonia produced by contact with cold lime water for 82 days in a closed vessel was also determined.

Lime at 100° liberated from 9 to 12 per cent. of the total nitrogen in the form of ammonia, as against 11 to 17 per cent. liberated by potash in a previous experiment (Comptes Rend. 1898, **127,** 414). Calcium carbonate and water liberated less than 1 per cent. of the nitrogen in the form of ammonia in the case of the first two soils, but a good deal more from peat, and still more from mould. The soluble nitrogen produced under the influence of lime at 100° amounted to 19·5, 21·26, 40·91, and 16·56 per cent. of the total nitrogen of the four substances. The more prolonged action of lime (cold) resulted in the production of ammonia representing, on the average, about 6 per cent. of the total nitrogen.

Calcium carbonate (at 100°) and water (at 100°) produced very little soluble nitrogen (excluding the ammonia evolved) from peat; a greater amount (6·40 and 5·61 per cent. of total) was formed in the case of vegetable soil; and considerable amounts (16·91 and 17·40 per cent.) in the case of mould. In the previous experiments (*loc. cit.*) it had been found that peat was greatly changed by the action of potash.

In acid soils the production of ammonia seems to be due entirely to the action of microbes; it is, however, suggested that, in such soils, plants may directly absorb certain amides without their being previously converted into ammonium salts properly so-called.—N. H. J. M.

## PATENTS.

*Fertilising Material, New or Improved Manufacture or Production of.* J. E. J. Johnson, Stratford. Eng. Pat. 20,660, Sept. 8, 1897.

The process claimed consists in depriving yeast of a large part of the liquid contained by it, by pressure or evaporation, and further drying and pulverising the product. This may then be used, either alone or mixed with dry loam, phosphate, or other suitable material, as a fertiliser. The preliminary evaporation is best performed in a vacuum vessel, provided with a steam jacket and mechanical stirrer, the final drying being effected by exposing the material on trays in a current of warm air. If the preparation is required for storage, a proportion of a suitable antiseptic is also added.—J. F. B.

*Potash Salts, Impts. in the Manufacture of.* O. Imray, London. From W. T. Gibbs, Buckingham, Canada. Eng. Pat. 24,525, Oct. 22, 1897.

*See under* VII., *page* 1016.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Sugar Works, Indeterminate Losses in: Means of Reducing them.* H. Pellet (Reported by H. Robert). Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16,** [2], 161—166.

From a comparison of the purity of beetroot juice and diffusion juice, the author concludes that when there is a loss by destruction of sugar, it is shown by a diminution of purity, and is therefore no longer indeterminate. He also finds that the purity of the liquor during ordinary work, as compared with the filtered juice after the second carbonation, does not show any evidence of transformed sugar, but that, where a loss does occur, it may be exactly estimated from the diminution of the coefficient of purity.

Knowing the importance of these new determinable losses, it is easy to seek means to be employed in reducing them.
—L. J. de W.

*Diffusion Process for the Extraction of Sugar from Beetroots, Impts. in the.* F. F. Garez. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16,** [1], 44—46.

The juice is supplied to the diffuser by a pipe which enters near the top and is then bent so as to pass vertically down the centre to near the bottom. The whole length of the pipe is perforated and the lower end is open. There is an arrangement for heating the juice before it reaches the diffuser. By this means a high and uniform temperature is ensured from the commencement of the charge, and the operation proceeds without disturbance.—L. J. de W.

### *Beetroot Juice [Sugar], Phenomena attending the Carbonating of.* J. Weisberg. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16,** [2], 167—184.

On submitting diffusion juice, defecated by lime, to a fractional treatment with carbonic acid, it was observed that the polarisation of the filtered juice diminished to a minimum, and finally rose again to that of the juice before carbonating commenced. In one experiment the figures obtained were:—

| | | | | |
|---|---|---|---|---|
| Polarisation of filtered juice..... | 12·80 | 12·73 | 12·50 | 12·45 |
| Alkalinity: Grms. CaO per litre | 16·20 | 13·00 | 6·50 | 3·10 |
| Polarisation of filtered juice..... | 12·60 | 12·85 | 12·87 | 12·95 |
| Alkalinity: Grms. CaO per litre | 2·30 | 2·00 | 1·60 | 1·40 |

As carbonating proceeds there is a gradual thickening of the liquid and attendant difficulty of filtration, the greatest diminution of polarisation corresponding with the moment when filtration is slowest. Before this point is reached, the filtered juice remains dark, but as soon as it is passed the polarisation increases and the juice becomes more and more decolorised.

The reaction is ascribed to the formation of the gelatinous precipitate of a basic double salt of calcium sucrate and carbonate, since it is found that at each stage of the experiment a higher polarisation is obtained if the juice is heated before filtration, and heat favours the decomposition of the basic calcium sucro-carbonate hydrocarbonate of lime. Moreover the liquid, filtered cold, becomes turbid on standing or when heated, whilst that filtered hot remains clear.

These phenomena were observed by Felz (Les Mondes, **16,** 1869), but he attributed them to a simultaneous precipitation of carbonate of lime, hydrate of lime, and sugar in variable and irregular proportions, and consequently is in complete antagonism to the view of Loiseau, that the sucro-hydrocarbonates are definite chemical compounds.

—L. J. de W.

### *Beetroot Juice [Sugar], Liming of; Differences of Solubility of Quick and Slaked Limes.* D. Loiseau. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16,** [2], 209—225.

In order to find the best form in which to employ lime for liming beetroot juice, the author arranged a method for ascertaining the degree of solubility of different limes in sugar liquors. From his numerous fully-detailed results, he draws the following conclusions:—That slaked lime formed in contact with an excess of water is always less soluble in sugar solutions than that obtained directly in the form of a dry powder; that the contact of an excess of water injures the quality of the slaked lime not only during its formation, but after it is formed; that a solution of sugar is very suitable, not only for slaking the lime, but also for preserving the lime when slaked; that the lime which possesses the highest degree of solubility is undoubtedly quicklime in the form of a fine powder.

As regards the liming of beetroot juice, he concludes that the lime least suitable is slaked lime formed in an excess of water; that slaked lime in the form of a dry powder is preferable to milk of lime, but is not of better quality than that formed in the beetroot juice itself; but that powdered quicklime should only be used when employed in successive fractions; otherwise preference is given to quicklime employed in small pieces.—L. J. de W.

### *Beetroot in the Manufacture of Sugar, The Resin-Acid of.* K. Andrlik. Zeits. für Zuckerind. in Böhmen, 1898, **23,** [1], 25—37.

The waste waters of raw-sugar factories are often covered with large masses of white foam. This foam is carried off and dries, being deposited as a light, greyish dust with a characteristic irritating odour. This dried foam is the best source for the preparation of the resin-acid of the beet, and by digesting with hot dilute hydrochloric acid, washing with water, and extracting with alcohol, the author obtained extracts containing from 14·2 to 26·2 per cent. of resin acid with $[\alpha]_{\rm D} = + 78{\cdot}67^\circ$. Part of the resin in the foam appears to be in the form of a salt, and the preliminary digestion with acid is necessary to render the whole of it soluble in alcohol. The author also detected and estimated the resin acid in the deposit from the diffusion-juice heaters and in the scums from the first saturation. It was found in the foam of the diffusion-juice and of the unfiltered saturation-juice. The diffusion-juice itself gave a precipitate with hydrochloric acid, in which the resin acid was estimated to the extent of 0·019 to 0·026 per cent. on the juice of 14° Brix.

The resin-acid was detected but not estimated in the molasses. The quantity here did not exceed 0·01 per cent., and is due to the slight solubility of the calcium salt of the resin in the hot saturation-juice. The author has not as yet worked out a method for the estimation of the resin-acid in the beet itself. Apparently it exists partly free and partly as the calcium salt. The free acid is extracted by the ether in the estimation of the fat, and is the cause of the slight dextro-rotation of the fat of beetroots. The residue yields, on digestion with hydrochloric acid, an alcoholic extract containing the originally combined resin.

No doubt some other dextro-rotatory substance exists along with the resin, since in no case was the author able to isolate anything like the quantity of pure resin-acid indicated by the optical rotation of the alcoholic extracts.

The alkali salts of the resin-acid have an extraordinary viscosity, a solution containing 0·1 per cent. of salt having the appearance of a thick syrup. From a technical point of view, this property might be expected to have an influence on the filtration of the juice. Solutions of the potassium salt filtered with the greatest difficulty through paper, but, on the other hand, only a slight difference in the rate of filtration was observed when the resin-acid was added to the juice and the latter limed and saturated in the usual manner.

The author has noticed, on the large scale, quick-filtering juices whose scums contained as much as 0·4 per cent. of resin-acid, and very slow filtering juices with only 0·16 per cent. He does not think that the mere presence of resin-acid is the cause of slow filtration. It might possibly happen that excessive saturation, by liberation of potassium carbonate, might bring about the formation of the highly viscous potassium resin-salt, which would cause bad filtration and subsequent viscosity in the syrups; but in a properly regulated factory this should not occur.

As regards the influence of the optical rotation of the resin-acid on the analytical results, the author observed than an alcoholic solution of the resin gave a gelatinous precipitate after a time with basic lead acetate. This precipitate was filtered with difficulty and the filtrate was dextro-rotatory. Where the proportion of resin in the saccharine product is large, the rotation of an alcoholic solution of the product should be corrected, but in the estimation of the sugar in beets, the author found the error to be negligeable. He found that a solution of the precipitate, formed by the addition of water to the alcoholic beet extract in Scheibler's process, had practically no optical power. He also calculated that the quantity of resin-acid which he extracted, together with the fat, from unripe beets, only amounted to an error in the polarisation of 0·011° on the normal weight of fresh beet-pulp.

—J. F. B.

### *Beet-Sugar Works, Baryta Used in.* A. Gallet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16,** [2], 187—193.

Four years of work with baryta on an industrial scale at Bléraucourt (Aisne) have rendered its use eminently practical and remunerative in partially replacing lime in the first carbonation.

When 1 kilo. of crystallised barium hydrate per ton of beetroots is used, the lime may be reduced by 15 kilos., the same purification being obtained. Under the conditions obtaining at Bléraucourt a saving is effected of 2·4076 francs per ton of beetroots worked, and the working season may be reduced by 39 days, thus avoiding the deterioration of the beetroots and the diminished yield of sugar which occurs at the end of the campaign.—L. J. de W.

*Sugar, Determination of Crystallised, in Massecuites.* H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [1], 20—23.

In a reply to the criticism of Lallemant it is maintained that indirect processes for the determination of the crystals in massecuites cannot have the accuracy, the simplicity, and advantage of the direct mechanical process, based on the employment of a laboratory centrifugal machine, which acts as a rotatory sieve.

The objection that the centrifugal force of this machine differs from that of the machines used on the large scale, does not hold, as the massecuite is not treated in the same condition, but with the crystals freed by dilution of the massecuite with a saturated syrup.—L. J. de W.

*Raffinose, The Behaviour of, on the Fermentation of Molasses.* K. Andrlik. Zeits. für Zuckerind. in Böhmen, 1898, **23**, [1], 1—25.

*See under* XVII., *page* 1064.

## PATENT.

*Saccharine Juices or Sugar Solutions, Impts. in the Purification of.* E. W. Gerbracht and F. G. Weichmann, New York. Eng. Pat. 16,292, July 26, 1898.

The process claimed consists in the addition to the saccharine liquid of a proportion of a salt, such as monocalcium hydrogen phosphate, $CaH_4(PO_4)_2$, or the ammonium hydrogen orthophosphates, $(NH_4)_2HPO_4$ and $NH_4H_2PO_4$, or other suitable salt, which, when boiled with the liquid at a low temperature under vacuum, is decomposed into an alkaline salt and an acid, expelling the volatile acid constituents as well as the volatile alkaline constituents of the saccharine solution. The process is applicable either to raw beet-juice or to after-products, and removes all objectionable taste and smell. The proportions of salt added are from 0·5 to 1·5 per cent. of the dry substance of the juice.—J. F. B.

# XVII.—BREWING, WINES, SPIRITS, Etc.

*Diastase: Its Preparation, Nature, and Estimation.* W. J. Sykes and H. N. Hussey. J. Fed. Inst. Brewing, 1898, **4**, 527—544.

The authors give an historical summary of the investigations which have been carried out on malt-diastase by C. O. Sullivan, C. J. Lintner, Osborne, Wróblewski, and Seyffert.

Sykes and Mitchell (Analyst, **20**, 233) found that a solution of the precipitate obtained by saturating malt extract with magnesium sulphate, when diluted to the same volume as the malt extract, possesses the same diastatic power as the latter; the present authors state that in their experiments they have observed under these conditions a considerable loss of diastatic power.

It appears that the precipitate produced by saturating an aqueous extract of barley with ammonium sulphate possesses considerable diastatic power. A similar result was obtained when magnesium sulphate was used. It is known that by heating a solution of diastase, partial coagulation takes place, accompanied by diminution of diastatic activity. If heated to a higher temperature the saccharifying action is destroyed, and only the liquefying property remains. On these and other grounds it has been supposed that diastase is a mixture of active enzymes, and the authors have carried out experiments to effect the splitting up of diastase by fractional salting out with ammonium sulphate. Several precipitates were obtained, but no difference could be detected in any of them, save that the second precipitate was more highly diastatic, since they all liquefied starch paste, and under proper conditions carried the reaction down to the No. 8 equation. Malt-invertase is also precipitated by salting out with magnesium sulphate. Salting out with Rochelle salt or sodium thiosulphate yielded active preparations of diastase.

The paper concludes with an account of Sykes and Mitchell's method of estimating diastatic power (this Journal, 1896, 621).—J. L. B.

*Barley and Malt, On the Carbohydrates of, with Special Reference to the Pentosans. The Behaviour of the Pentosans during the Preparation of Malt, and during Mashing and Fermentation.* B. Tollens. J. Fed. Inst. Brewing, 1898, **4**, 438—454.

In barley and malt there are contained, besides the substances soluble in water and those rendered soluble by the mashing process, which together constitute the extract, certain insoluble substances classed as fibre or cell tissue. Naturally the same substances are present in malt as in barley, but they are to some extent modified and transformed by the germination process; and the distinctly sweet taste of malt as compared with the mealy taste of barley indicates the presence of a larger amount of sugars in the former.

The author reviews our knowledge of the carbohydrates of barley and malt. Besides certain sugars there are present in malt, amorphous transformation products of starch, *e.g.*, dextrins. If finely-ground (German) malt be boiled with alcohol to destroy the diastase, and the residue shaken with cold water and a little very dilute iodine solution, a distinct violet coloration is produced, which, on addition of more iodine, passes into dark blue. Of the insoluble carbohydrates, may be mentioned cellulose, and those substances related to it which are soluble in solution of caustic soda, and to which E. Schulze has applied the collective name hemicellulose. Those hemicelluloses which when heated with dilute sulphuric acid are converted into glucose, galactose, &c., are known as hexosans; others belong to the pentosans.

If the substances forming the tissues of plants are moistened with a solution of phloroglucinol in hydrochloric acid, they either remain unaltered or are coloured red, and this coloration is known as the lignin reaction. The substances which give this reaction also yield considerable quantities of furfural when distilled with 12 per cent. hydrochloric acid, and when boiled with dilute sulphuric acid they yield the pentoses, arabinose or xylose.

The pentoses are not, as a rule, present as such in plant substances, but rather as the parent compounds, which yield pentoses on hydrolysis, and, accordingly, formulæ must be assigned to them containing less of the elements of water than the pentoses. These parent substances of the pentoses are known generally as pentosans, whilst those which yield arabinose and xylose on hydrolysis are denoted respectively as arabans and xylans. The most probable empirical formula for the pentosans is $C_5H_8O_4$, and their conversion into pentoses, as well as that of the pentoses into furfural, may be represented by the equations—

$$\underset{\text{(Pentosan)}}{C_5H_8O_4} + H_2O = \underset{\text{Pentose}}{C_5H_{10}O_5} \text{ and } C_5H_{10}O_5 = \underset{\text{(Furfural)}}{C_5H_4O_2} + 3H_2O.$$

The pentosans are not the only substances which, on distillation with hydrochloric acid, yield furfural; thus glycuronic acid and also, according to Cross and Bevan, a large series of oxidation products of the carbohydrates, yield furfural under similar conditions. Cross and Bevan designate the vegetable substances which yield furfural, furfuroids; the author prefers the old term pentosan, because almost all of these substances produce pentoses on hydrolysis. It has been recognised, however, that vegetable tissues on hydrolysis yield, besides arabinose and xylose, a considerable amount of syrupy substances which may contain other pentoses (perhaps ritose, lyxose, &c.), and also glycuronic acid, together with those oxidation products of the carbohydrates concerning which very little is known.

The results obtained by the author and Glaubitz (J. für Landwirtsch. 1897, 106), and also by Cress and Bevan (Ber. 1894, **27**, 1061, 2604), show that barley contains 9—10 per cent. of pentosans, whilst the first-mentioned chemists found 9·88 per cent. of pentosans in malt, and in malt which had been grown for a long period, 11·8 per cent. They conclude that the pentosans contained in barley are not diminished during the process of germination, there being, indeed, a slightly increased quantity of them in the malt.

Regarding the question as to whether pentosans pass into the wort, the author and Glaubitz (*loc. cit.*) made a malt mash, with the following results :—

| Amounts taken. | Pentosans. | | |
|---|---|---|---|
| | Per Cent. | Weight in Grms. | Weight in Grms. in Wort and Grains. |
| Malt, 82 grms. ....... | 11·18 | 4·17 | .. |
| Grains, 20·5 grms. .... | 29·43 | 6·03 | 8·33 |
| Wort, 328 c.c. ........ | .. | 2·30 | |

From these figures it will be seen that the greater part of the pentosans remain in the grains ; only a fraction passes into the wort. Lager beer was evaporated, and found to contain 0·25 per cent. of pentosans, or 5·3 per cent. calculated on the total solids.

The author, in conjunction with Stone, and, subsequently, with Schöne, has proved that arabinose and xylose are not capable of being fermented with yeast. It is, however, possible that the small quantity of pentosans and pentoses which occur in wort, are decomposed by other fermentative changes, which proceed side by side with alcoholic fermentation, and produce acetic, lactic, and succinic acids. The possibility that the pentoses take part in fermentation is supported by the observation of Cross and Bevan (this Journal, 1897, 252), that the pentosans or pentoses in liquid extracts, obtained by heating brewers' grains with dilute sulphuric acid, are fermented by yeast, sometimes partially and sometimes entirely. It is possible that the cause of this difference in the behaviour of the pentosans is not due to alcoholic, but to an acid, fermentation, as the yeast used in breweries is not always a pure culture. In order to solve this question, Schöne has, at the author's suggestion, carried out the following experiments. Extracts were obtained by treating brewers' grains with dilute sulphuric acid, and these were sown with brewery yeast, the amount of pentosans in the liquid before and after fermentation, as well as the cupric reducing power, being determined. The amount of alcohol, and also of volatile and non-volatile acids, was estimated.

| — | Percentage before Fermentation. | Percentage after Fermentation. | Percentage fermented. |
|---|---|---|---|
| Pentosans ............ | 26·9 (=30·5 pentoses). | 5·1 (=5·8 pentoses). | 21·8 |
| Total reducing sugars as glucose. | 29–29·5 | 5·2 | 24·0 |

Since the 21·8 per cent. of pentosans removed by fermentation yielded only 3 per cent. (on the grains) of alcohol, it follows that other products were formed, and, in a fermented extract, the presence of acetic and lactic acid was proved.

When an extract was fermented with a pure yeast, the amount of pentosans fermented was considerable, although not so great as when ordinary brewery yeast was employed. Thus, in a fermentation which was allowed to proceed for 8 days at 27° C., 2·27 per cent. of alcohol, 0·4 of acetic acid, and 0·97 per cent. lactic acid, calculated on the grains, were obtained.

An experiment is described, which shows that arabinose remains unaltered when in contact with a pure yeast; the following result was, however, obtained when a solution of arabinose rendered nutrient was inoculated with an acid producing bacillus, and kept in an incubator for seven days.

| | Per Cent. |
|---|---|
| Loss of arabinose........................... | 20·10 |
| Yield of alcohol ............................ | 3·12 |
| „ volatile acids as acetic acid......... | 4·23 |
| „ non-volatile acids as lactic acid..... | 1·10 |

—J. L. B.

*Taka-diastase.* W. E. Stone and H. E. Wright. J. Amer. Chem. Soc. 1898, **20**, [9], 639—647.

After a short summary of the present state of our knowledge of this enzyme, an account is given of a series of experiments which were carried out with the object of comparing its action on starch with that of ordinary malt diastase.

Pure potato-starch, in the form of paste, was subjected to the action of each kind of diastase under precisely similar conditions, the degree of conversion of the starch being determined (1) by the iodine-test, (2) by determining the optical activity of the solutions, (3) by the amount of sugar formed. (1) The iodine-tests showed that the taka-diastase invariably worked more rapidly at the outset, but the complete conversion of the starch was effected earlier by the malt-diastase. (2) In the optical tests the saccharifying powers of the enzymes were taken to be proportional to the decrease of the rotatory powers of the solutions, as the intermediate products of conversion have a higher specific rotation than the mixture of maltose and dextrose finally produced. The products of the action of taka-diastase were in all cases of lower specific rotation than those from malt-diastase, indicating a more rapid change to sugar. (3) The sugar-test showed that for a given time, taka- is superior to malt-diastase in saccharifying power.

Further experiments confirmed the results obtained by the iodine-test : that whilst taka-diastase was able to transform a large quantity of starch in a short space of time, yet, even after long periods, there remained some starch unaltered ; hence it cannot be recommended as a reliable analytical agent.—E. H. T.

*Diastase, The Destruction of, during Fermentation.* G. Heinzelmann. Zeits. f. Spiritusind. 1898, **21**, [41], 357.

In the author's experiments a pure culture of distillery-yeast, Race II., was used, and precautions were taken to keep the fermentations pure.

A malt-wort was prepared and its corrected diastatic power determined by Lintner's method, before and after fermentation. The results showed that the diastatic power of the wort had decreased 66·6 per cent. during its fermentation.

Two experiments were made with mashes of potato-starch, containing nutrient salts, and saccharified by a solution of prepared diastase. To one mash asparagine was added and from the other it was omitted.

When asparagine was present the fermentation was completed in 45 hours, and the diastatic power of the mash had diminished 72 per cent. during fermentation. In the absence of asparagine the fermentation required 70 hours, and the decrease in diastatic power was 90·2 per cent. Hence it is concluded that asparagine and other nitrogenous foods preserve the diastase from attack by the yeast.

The author found, on calculating the results of the two last experiments on the diastase originally added to the starch-mash, expressed in terms of comparable diastatic units, that in the mash containing asparagine 71·4 per cent. of the diastatic units added in the diastase solution were destroyed during the saccharification of the starch at 50—51° R., and 20·6 per cent. during fermentation, leaving a balance of 8 per cent. In the mash without asparagine, 63·7 per cent. of the diastatic units were destroyed during saccharification, 32·7 per cent. during fermentation, and a balance of 3·6 per cent. remained in the fermented liquid.

A separate experiment was performed to determine the progressive destruction of diastase during the mashing process. A mash was prepared with 16 per cent. of starch, and 2 grms. of diastase were added to 1,000 grms. of the mash. After 10 minutes at 49° R., 14·3 per cent. of the diastase had disappeared ; after 20 minutes further mashing at 50° R., 60 per cent. of the original diastase had been destroyed. The residual 40 per cent., however, suffered no further diminution after 30 minutes further heating at 50° R.

The author believes that during fermentation the diastase is destroyed in the interior of the yeast-cell by the proteohydrolytic enzyme present, but that this enzyme has no effect on the diastase outside the cell. He intends to clear up this point by a study of the action of pressed extract of yeast on diastase.—J. F. B.

### *Alcoholic Fermentation without Yeast Cells.* H. Abeles. Ber. 1898, **31**, 2261—2267.

The possibility is discussed of Buchner's expressed yeast extract consisting of surviving particles of yeast protoplasm—a view which has been expressed by Voit, Kupffer, and Hans Buchner. E. Buchner, however, has shown by a series of experiments that yeast extract is active when in presence of poison fatal to protoplasm, and in high concentrations of sugar and of glycerin—conditions which would annul the action of ordinary yeast. The author insists on the point that the action of poison upon the ferment is not only dependent upon the concentration of the poison, but also, in a higher degree, on the relation of the amount of poison to that of protoplasm. Experimental evidence is adduced to show that whilst a certain quantity of poison, such as Buchner employed, is fatal to the fermentative power of a given quantity of yeast; yet, if the amount of yeast be increased, an active fermentation is brought about.

The second principal argument against the living protoplasm theory is the fact that dried yeast extract heated to 100° C. is still capable of fermentative power. Weisner (Sitzungsber. d. kaiserl. Acad. der Wissenschaften, **59**, 500), however, has shown that when dried yeast is heated to 100° C. a portion of the yeast cells—notably the young ones—remain active.—J. L. B.

### *Alcoholic Ferments, The Vitality of the, and their Preservation in Nutrient Media and in the Dry State.* E. C. Hansen. Compte rend. du Labor. de Carlsberg, 1898; through Wochenschr. für Brau. 1898, **15**, [41], 540—542.

After an historical survey of the previous work on this subject, the author publishes in this paper the results of a series of experiments started in the year 1881. Pure cultures of various fermentation organisms were then sown in different media, and others were dried on cotton-wool and on filter-paper. The organisms investigated comprise 42 species and 5 varieties of saccharomyces, nine yeast-like, non-sporogenous organisms, and seven species of mould fungi.

For a comparison of the periods of vitality of the different ferments, reference must be made to the original paper. On the whole, the results of duplicate experiments are somewhat discordant. All the organisms lived longest in solutions of cane-sugar (10 per cent.). The shortest-lived species of saccharomyces were *S. Ludwigii* and Carlsberg yeast No. 2, the former varying from one to six years and the latter from three to eight years. Most of the other species had not attained this limit of vitality after 16 years, whilst Carlsberg yeast No. 1 and some wild yeasts were still alive after 17 years. The lifetime of the yeast-like organisms, comprising *Monilia candida*, *S. apiculatus*, and seven species of Torula, varied from 3 to 14 years. All the mould fungi lived for seven years, and some for 11 years. When the access of air was cut off, all the organisms died within two years. In beer wort the period of vitality was somewhat shorter than in cane-sugar solutions. Some cultures, however, lasted for 11 years, whilst others, duplicates of the same, died after five months. In water many of the yeasts lived for 11 years, but some only lasted a few months.

Streak cultures on wort gelatin of seven of Hansen's species were still alive after two years, in spite of the fact that the gelatin had dried up.

In the dry state on filter-papers, the Hansen species lived from 5 to 11 months, whilst spores of some Pastorianus species retained their vitality for three or four years. The mould fungi lived somewhat longer.

Dried on cotton-wool, the yeasts lasted longer than on filter-paper, and spores were formed. When kept on platinum wires in the desiccator they lived only a few days, or at the most for a few weeks, whilst outside the desiccator, they did not last much longer. The author remarks that the period of vitality of single cells is only correct when preserved in the dry state, as the results obtained in culture media represent the vitality of the whole colony of several generations. In the dry state, however, several disturbing factors come into play, such as the thickness of the layer and the access of light and air. He states that in cane-sugar solutions the budding and multiplication can only be limited, and the results approximate to those for single cells. [The article from which this abstract is taken, *Wochenschr. für Brau.*, *loc. cit.*, is by Lindner, who disagrees with this statement, and remarks that when the majority of the cells are dead, one or two still go on multiplying under easier conditions of existence, and thus keep up the chain of generations.]

The author states that since the evolution of new varieties is least to be feared when the multiplication is most restricted, preservation of the yeast in the dry state is most likely to keep its properties constant.

In the Carlsberg breweries, solutions of cane-sugar have been found to answer best for the preservation of yeast, but cultures should not be kept too long, or variations in the properties may result.—J. F. B.

### *Fermentation, Vacuum.* O. Reinke. Wochenschr. für Brau. 1898, **15**, [41], 528.

The vacuum process was first adopted in the United States, where it has been fairly thoroughly worked out, resulting in a considerable economy of space and buildings, and a reduction in the time of fermentation, and storage. In some parts it is used for the top-fermentation or else only for the after-fermentation; in others the carbon dioxide is pumped off at first, and a slight pressure allowed to develop afterwards, by which means the beer retains sufficient gas and its full aroma. The process has been taken up to a greater extent in England and Australia, but attempts to introduce it in South America have only met with limited success. Latterly it has been gaining a footing on the Continent. In some breweries, where the beer was much exposed to infection during fermentation by the old method, the use of the closed vessels has shown an improvement, but as the process was only cautiously applied, the results cannot be considered as representative. On the other hand, it is stated that beer fermented under vacuum has a flat taste, and the advantages claimed are only obtained at the expense of the flavour. The enamelled vessels are very expensive, and hard deposits are liable to form, which are very difficult to remove, even by steaming under pressure. The author is of opinion that the vacuum should not be applied as soon as the wort is set up. The yeast requires a supply of fresh, filtered air, which is used up during fermentation, and the vacuum should only be employed when this air has been absorbed. If the gases pumped off are led through well-cooled tubes, certain products are condensed which may afterwards be added to the beer, but even then some of the aromatic constituents of beer fermented by the older methods are lacking.

The subject requires careful, systematic investigation, and the process may very likely be applicable to the manufacture of dark beers, where the caramelised products may disguise the absence of aroma.—J. F. B.

### *Alcoholic Fermentation of Molasses, Method of Facilitating.* —Bauer. Rev. Prod. Chim. **1**, [9], 130.

In order to obtain a cheaper yeast food than that yielded by cereal grains, the author exposes beer yeast—previously freed, by sifting, from hop resins and other impurities—to the action of lactic acid bacteria, in open vessels for 12—14 days at a temperature of about 30° C. This treatment results in the liquefaction of the mass, the yeast cells disappearing entirely, whilst 1·3 per cent. of lactic acid is formed from the plasmic mucilaginous matter, and the albuminoid substances are almost entirely converted into amides. The product is sterilised by steam heat, deprived of its volatile acids, and is employed as yeast nutriment in the proportion of 2·5 parts per 170 parts of sugar in the (dilute) molasses to be fermented. Since under the same conditions 10 parts of malt would be ordinarily required, a considerable saving is effected by this method.

When the production of aërated yeast is in question, vigorous growth being a prime factor, the quantity of liquefied yeast nutriment is raised to 40 or 50 parts.—C. S.

### *Raffinose, The Behaviour of, in the Fermentation of Molasses.* K. Andrlik. Zeits. fur Zuckerind. in Bohmen, 1898, **23**, [1], 1—25.

Raffinose, either alone, in presence of saccharose, or in so-called raffinose-molasses, was fermented by top-fermentation yeast only to the extent of about one-third of its weight, corresponding to the fructose residue. In 72 hours, with raffinose alone, only about 73 per cent. of the calculated quantity of alcohol was obtained, and even after 137 hours, in presence of saccharose, the whole of the fructose residue was not completely converted into alcohol. The presence of raffinose had no influence on the fermentation of the saccharose of the molasses.

From molasses rich in raffinose, an average of about 89·2 per cent. of the theoretical quantity of alcohol, calculated from the analysis of the molasses, was obtained. This result is attributed by the author partly to contamination of the top-fermentation yeast employed, and partly to the inaccuracy of the existing methods for the determination of saccharose and raffinose in the presence of other optically active substances found in molasses.

The addition of raffinose to molasses, already containing only small quantities of that sugar, gave rather better yields of alcohol.

The raffinose calculated from the residual melibiose, on analysis by Creydt's mucic acid method after fermentation, averaged 92·9 per cent. of that determined in the original molasses, showing that the melibiose residue was almost unattacked after 72 hours' fermentation with top-fermentation yeast.

With bottom-fermentation yeast the raffinose was attacked as a whole. After 72 hours, the fermentation was by no means complete, but after 144 hours, practically the whole of the raffinose and saccharose were fermented, a yield of 97·9 per cent. of the theoretical quantity of alcohol, calculated on the analysis by Herzfeld's optical method, being obtained. The addition of raffinose, even up to a total of 14 per cent., had a favourable effect on the fermentation of the molasses by bottom-fermentation yeast. It was found that a mixture of top- and bottom-fermentation yeasts gave a more complete fermentation and a higher yield of alcohol in 72 hours than either species used separately. The melibiose residue took part in the action, and a yield of 94·4 per cent. of the calculated quantity of alcohol was obtained.

By boiling the molasses containing raffinose with dilute sulphuric acid and fermenting with top-fermentation yeast, a greater yield of alcohol was obtained than from the untreated molasses. In some cases this yield was very slightly higher than the theoretical quantity calculated for the fructose residue, but the melibiose residue did not take part in the production of alcohol to any considerable extent by this treatment.—J. F. B.

### *Proteo-hydrolytic Enzyme Contained in Extract of Yeast, Further Communications on the.* L. Geret and M. Hahn. Ber. 1898, **31**, [14], 2335—2344.

It was found that in 6—8 days the enzyme in the pressed-extract was able to decompose, not only the coagulable albuminoids present, but also quantities of other added albumins, such as fibrin, egg-albumin, and the coagulated albumin from other samples of extract. After one hour's digestion of the extract, leucin and tyrosin were found in the filtrate after coagulation, but no true peptone, and only traces of albumose. When 2 per cent. of peptone or albumose was added to the extract, the biuret reaction disappeared after three days. The nitrogenous compounds were separated by phospho-tungstic acid into bases and amido acids, and the relation of these groups at different periods was determined. At first the proportion of bases increased, but after two weeks the ratio was the same as at the start. Will has recently observed that, with deficiency of soluble nutrients, especially nitrogen, the secretion of the enzyme in the living cell is favoured by the absence of air. The authors passed currents of air and hydrogen through samples of pressed-extract at 37° C., and compared the amounts of coagulate, obtained at various periods, with a control sample. They found that the action of air on the enzyme in the pressed-extract was unimportant, whilst hydrogen had a slight retarding action.

The behaviour of the xanthine compounds in fresh and in old extracts is peculiar. Salkowski has described a "latent" state of the xanthine compounds in yeasts after prolonged digestion. If the yeasts were boiled before digestion, the xanthine could not be directly detected by silver solutions afterwards; but if they were boiled after the digestion, a precipitate was readily formed. The "latent" state of the xanthine in the yeast filtrates in the first case was destroyed by boiling for one hour with 1 per cent. sulphuric acid, and the xanthine could then be precipitated in the usual manner. The authors found that fresh pressed-extract gave no xanthine reaction, but that, after prolonged digestion, xanthine was present in the "latent" state and could be precipitated after boiling with acid. The formation of hypo-xanthine increased with the time of digestion. The passage of gases during the period of digestion did not increase the production of xanthine, but after 14 days the xanthine in the samples which had been treated with gas was directly precipitable without boiling with acid. In untreated samples the passage of gas subsequent to the digestion did not obviate the necessity of boiling with acid to obtain the xanthine precipitate. Continuous shaking was found to have the same effect as treatment with gases. The organic phosphorus in pressed-extract is converted into phosphoric acid after a few hours' digestion.

On studying this conversion, the authors found that after one hour's digestion, about two-thirds of the total phosphorus was in the state of phosphoric acid, but that even after several days the whole was not converted, and there remained a residue of organic phosphorus amounting to one-fifth or one-sixth of the total. The organic sulphur was converted into sulphuric acid only to a very slight extent. It was found that the activity of the enzyme was only weakened, not destroyed, by the presence of 1 per cent. of hydrogen cyanide, and this weakening may be due to the acidity only.

On mixing the pressed-extract with eight times its volume of absolute alcohol, the enzyme is thrown down together with the albumin. On treatment with water this precipitate is only partially redissolved. The enzyme is all present in the filtered solution, and, after keeping this for a short time, it can be separated in small quantity and in a fairly pure state.

A proteo-hydrolytic enzyme is also present in the pressed-extracts obtained from other micro-organisms, and the authors give results of quantitative experiments, showing the decrease of coagulable albumin on keeping extracts of tubercle bacilli, typhus bacilli, and *sarcina rosea* for different periods. These enzymes appear to be very widely distributed in animal and vegetable organisms, and have been found in the juices of lupin seedlings and of the cells of the liver.
—J. F. B.

### *Beer Wort, Influence of Mineral Constituents of Brewing-Water on.* De Smet. La Bière, **6**, [8], 117—118.

From the experiments detailed, the author concludes that no injurious influence on the saccharification of the wort is exerted by sodium sulphate, calcium nitrate, or magnesium sulphate, nitrate or chloride.—C. S.

### *Lactic Fermentation, The Prevention of, in the Distillery, by Acidification with Commercial Lactic Acid.* Wehmer. Zeits. für Spiritusind. 1898, **21**, [39], 342, and [40], 349—350.

The process of acidification of the yeast-mash with lactic acid, with the object of preventing the development of lactic bacteria, has not hitherto been adopted on the industrial scale, in spite of much that has been said in its favour, on account of the high price of lactic acid. There is now on the market a commercial acid, costing in Germany 60—80 marks per 100 kilos, which contains 50 per cent. of lactic acid, and which, although not quite pure, is free from any objectionable volatile impurities, such as butyric acid, which might affect the flavour of the spirit. The author has studied the effect of acidifying the preliminary mash, used for bringing the yeast into an active condition, with the above commercial lactic acid, and the process has been carried on successfully on the large scale in a distillery for more than six months.

In the laboratory experiments, proportions of 1, 0·5, 0·25, 0·125, and 0 per cent. of lactic acid were respectively added to two sets of beakers, each containing 200 c.c.

the yeast mash. The mash in one set was sown with 1 grm. of pressed yeast to each beaker, whilst that in the other was fermented by air-borne organisms alone.

With 1 per cent. of lactic acid, microscopical examination, at the end of seven days, showed that the yeast in the sample which was sown had remained perfectly pure, and that the unsown mash had been fermented by air-borne yeasts only, foreign bacteria being either absent or rare. With 0·5 per cent. of lactic acid, the sample sown with yeast contained only a few of the rod-shaped lactic bacteria, whilst the unsown sample was largely contaminated.

With diminishing proportions of lactic acid, the purity of the yeast diminished, and the samples containing 0·125 per cent. were as impure as those with none at all. The addition of 1 per cent. of lactic acid to the mash had no harmful effect on the development of the yeast, the fermentation in all cases being perfectly normal.

On the large scale, it was found that when the preliminary yeast mash was acidified with 0·5 per cent. of lactic acid, it had a somewhat thicker consistency and slightly lower temperature than when acidified in the usual manner with sulphuric acid. When the mother-yeast already contained lactic bacteria, the addition of 1 per cent. of lactic acid to the preliminary mash did not entirely prevent the development of these bacteria during fermentation: but when the mother-yeast was pure, absolutely no bacteria were found after the preliminary fermentation, and only very few after the main fermentation. The multiplication of the yeast was slightly increased (about 1 per cent. higher), and the yield and flavour of the spirit were in every respect satisfactory.

Perhaps, at the present price of the acid, the process does not show any considerable economy in the cost of production of the spirit and yeast, but, on the other hand, everything proceeds much more smoothly, and is more under control than when the ordinary method of acidification is used. Probably, with increased consumption of lactic acid in the dyeing industry, the price will be reduced in the near future.

By the old method of acidification with sulphuric acid, the acidity of the mash increases during fermentation at the cost of the sugar, owing to the activity of the bacteria, whilst by acidification with lactic acid it remains constant to the end, and the fermentation is pure.—J. F. B.

*Lupulinic Acid (B Hop-bitter Acid).* G. Barth and C. J. Lintner. Ber. 1898, **31**, [13], 2022.

*See under* XX., *page* 1070.

*Wine Extract, Determination of.* A. Cellerin. Rev. intern. falsific. **11**, 126; Chem. Centr. 1898, **2**, [10], 611.

*See under* XXIII., *page* 1076.

*Hops, their Valuation, and the Volatile Acids of.* R. Wetschorkewitsch and M. Bjalobrsheski. Farmazeft. 1898, **6**, 525.

*See under* XXIII., *page* 1075.

## PATENTS.

*Yeast, Improved Process for the Extraction of the Plasma (Cell Contents) and Separation of Cellulose from.* A. D. Watson, Mitcham. Eng. Pat. 22,816, Oct. 5, 1897.

Two processes and the extracts prepared by them are claimed:—

*Process* 1.—Washed, pressed yeast is desiccated either by heat or by contact with some dry inert agent, such as anhydrous starch. After separation from the starch by sifting, the yeast can easily be ground to an impalpable powder. If desired, this can then be mixed with water, and the liquid extract filtered from the cellulose and concentrated *in vacuo*. When the extract has reached a specific gravity of 1·160—1·180 it is again filtered from the precipitate formed by the coagulation of some albuminoids in combination with hop-resins.

*Process* 2.—The washed, pressed yeast is placed in a vessel and allowed to break down spontaneously into a liquid state. The temperature is best kept constant at 95—98° F. After the yeast has liquefied, an antiseptic, such as salicylic acid, and any desired flavouring matter, are added, and the constant temperature maintained for some 20—40 hours longer, until the extract has reached a certain specified specific gravity. It is then filtered, and raised to a temperature of 180—200° F., to coagulate certain albuminoids. After filtration, the extract may be concentrated to a paste, if desired, in which form it contains 60—70 per cent. of albuminoids.—J. F. B.

*Spirits and other Liquids, Treating with Gases; Impts. in.* J. B. Hilliard, Glasgow. Eng. Pat. 23,724, Oct. 15, 1897.

SPIRITS or other alcoholic liquids are treated with gases, such as air, oxygen, or ozone, without evaporation or loss of gas, by passing the gas through and over the liquid in a closed circuit, in such a way that the gas emerging from the liquid is again utilised for the same purpose. Any gas which is absorbed by the liquid is continuously replaced from a reservoir in the circuit.

The process can be conveniently carried out in two ways: either with a force-pump, which injects the gas through the liquid and sucks the gas emerging at the other end after replenishment from a reservoir, or else by alternately passing the gas from one reservoir to another by the pressure of movable weights, and letting it travel, in its passage, through a vessel containing the liquid to be treated.

—J. F. B.

*Saccharometer for Testing Beer Worts, An Improved.* M. Preu and G. Himmel, Tübingen, Germany. Eng. Pat. 29,930, Dec. 17, 1897.

*See under* XXIII., *page* 1072.

*Alcohol by Saccharification and Fermentation by Means of Mucedineæ, Impts. in Apparatus for the Manufacture of.* A. Collette, jun., and A. Boidin, Seclin, France. Eng. Pat. 15,561, July 15, 1898.

THE claims of the present patent relate to the combination and application of certain mechanical devices, some of which taken separately were previously well known, to the process of saccharification and fermentation by means of the mucedineæ (Eng. Pat. 1897, 19,858).

The objects of the devices patented are to preserve antiseptic conditions in all parts during the whole operation, to agitate in such a way as to keep the ferments continually immersed, to introduce the sterilised air necessary for the propagation of the organisms, to introduce cultures and to withdraw samples during the operation without infection, and to discharge the gaseous products of fermentation continuously without sudden inrush of air.—J. F. B.

# XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

## (*A*.)—FOODS.

*Albumose Milk, A New.* Schreiber and Waldvogel. D. med. Wochenschr. 1898, **24**, 505; from Chem. Zeit. Rep., 1898, **22**, 236.

THE authors have found that the addition of a relatively small quantity of albumose to milk sensibly increases the digestibility of the casein. Albumose, unlike other substances added to milk for this purpose, does not cause diarrhœa, so that albumose milk can be given to children with a tendency to that complaint. The following three kinds of prepared milk may be used:—

| —— | I.<br>From One to Three Months. | II.<br>From Three to Six Months. | III.<br>From Six Months on. |
|---|---|---|---|
| Skimmed milk .. | 350 | 480 | 720 |
| Cream .......... | 300 | 280 | 280 |
| Water .......... | 350 | 240 | .. |
| Milk sugar ..... | 20 | 15 | .. |
| Caseose ......... | 3·2 | 2·4 | 1·6 |

—J. L. B.

*Butter; On the Volatile and Insoluble Fatty Acids in.* R. Henriques. Chem. Rev. Fett- u. Harz-Ind. **5**, [9], 169—172.

In view of the fact that the Reichert-Meissl number corresponds to different percentages of volatile acids according as their molecular weights vary—*e.g.*, R.-M. No. 29 = 5·10 per cent. of butyric acid or 6·73 per cent. of caproic acid—the author was led to inquire whether the abnormally low value of this constant in some pure butters was due rather to a high mean molecular weight of the volatile acids, than to smallness of quantity of the same, and whether on this basis, a method of distinguishing such abnormal butters from mixed butters—wherein the *quantity* of the volatile acids is necessarily lower—could be devised.

To this end he examined a series of butters with abnormally low Reichert-Meissl values, by drying the soap from the R.-M. test, its weight, multiplied by $\frac{2 \cdot 2}{100}$, being termed the "soap number." Representing this value as $a$, and the R.-M. value as $b$, the percentage of volatile soluble acids is revealed by the formula $a - \frac{76b}{1000}$; and their mean molecular weight $= \frac{1000a}{2b} - 38$.

The results hitherto obtained are, however, only negative, so far as their value for analytical practice is concerned, the mean molecular weights coinciding with those in normal butter (93·3—99·8), whilst the actual quantities of the volatile acids are lower (4—5 per cent. instead of 5—6 per cent.).

The author then proceeded to the examination of the insoluble fatty acids, and found the Reichert-Meissl number in normal pure samples 86·5—89·1, whilst in abnormal—also pure—butters it ranges from 88·96 to 90·68. Difficulties, however, arose in the determination of the mean molecular weights, samples which gave values between 267 and 270 when dried at 105°—110° C., showing lower values (256·3—263) when dried *in vacuo* without the assistance of heat.

He concludes that, in view of the assumption that butter contains a large percentage of oleic acid, there must also be—from the above figures—a considerable quantity of another acid, of lower molecular weight than palmitic acid; and that the isolation of this acid is desirable. Also, that if the assumption of a very low mean molecular weight for the saturated insoluble acids be correct, their separation may perhaps afford a means for distinguishing pure butter from the other fats, wherein, by reason of the high proportion of stearic and palmitic acids, the corresponding molecular weights are relatively high.—C. S.

*Chocolate, Estimation of Sugar in.* P. Carles. J. de Pharm. et de Chim. 1898, **8**, [6], 245

*See under* XXIII., *page* 1076.

*Food, Detection of Formaldehyde in.* E. Rimini. Ann. de Farm. e Chim. **1**, 9; Pharm. J. 1898, **61**, 363.

*See under* XXIII., *page* 1076.

## PATENTS.

*Organic Substances, Preservation of.* F. W. H. Graeff, Berlin. Eng. Pat. 21,387, Sept. 17, 1897.

The substance is introduced into an air-tight vessel, from which the air is then expelled and an indifferent gas, *e.g.*, carbon dioxide, or an antiseptic gas, *e.g.*, formalin, introduced. Pasteurised in this manner, the freshness and appearance of the substance remained unimpaired. This process can be used for the preservation of animal flesh, vegetables, milk, effervescing drinks, &c., &c. There are 19 claims.—E. H. T.

*Malt Preparations, An Impt. in the Manufacture and Treatment of.* W. Jago, Hove, Sussex. Eng. Pat. 27,376, Nov. 22, 1897.

The patentee claims the preparation made by mixing an infusion of malt, either in its dilute state or after concentration *in vacuo*, with bran or germ or some cereal product, such as is obtained by the milling of wheat or other cereal, or else with starch. The mixture is dried first in a steam-jacketed vacuum-pan and finally on trays by warm air, and then pulverised. If it is desired to retain the diastatic properties of the malt, the temperature at any stage must not be higher than 160° F. The preparation is a valuable foodstuff and can be used, mixed with flour or meal, for baking purposes or as a substitute for malt-extract.—J. F. B.

*Soluble Casein Preparations, Impts. in Method of Obtaining.* A. Backhaus, Königsberg, Prussia. Eng. Pat. 30,877, Dec. 31, 1897.

The object of the invention is to make possible the greater consumption of casein and other albuminous matter contained in milk, by rendering them soluble in water. Casein, separated from milk by means of acid or rennet, is triturated in a moist state with tertiary sodium citrate, which must react slightly alkaline to litmus. 2·5 gms. of salt are necessary for every litre of milk treated. Instead of the tertiary salt the neutral citrate, mixed with either sodium carbonate or tri-sodium phosphate in certain proportions, may be employed. To obtain a perfect preparation, the milk must first be freed from fat and other precautions taken.

Compound casein preparations are obtained from milk by heating the latter with the citrate, after freeing from fat, and then evaporating to dryness in a vacuum.—E. H. T.

*Slaughtered Animals, Impts. in the Method of, and Apparatus for, Preserving.* A. Fjelstrup, Odense, Denmark. Eng. Pat. 6323, March 15, 1898.

The blood of the slaughtered animal is driven out by injecting sterilised water into the vascular system under suitable pressure. The water is then displaced by sterilising liquid, *e.g.*, a solution of common salt and saltpetre, or by the latter mixed with a disinfecting agent. To effect the injection the animal's chest is opened along the middle line and a faucet introduced into the aorta through the left hand ventricle, the walls of the aorta being pressed tightly round the faucet by means of a pair of tongs. The other end of the faucet is forked, and each branch is provided with a stop-cock which communicates with the liquid to be injected, the latter being either pumped in, or run in from tanks standing at a suitable height.—E. H. T.

*Sterilised Raw Meat, Improved Process for obtaining as Powder.* F. Grognet, Nanterre, France. Eng. Pat. 12,612, June 4, 1898.

The process consists in grating the raw meat either to a coarse or fine powder, as required, with lentil, wheat, pea, or other flour. The excess of the latter is then removed, and the meat sterilised by dipping for a few seconds into a bath containing vinegar, salt, ginger, various spices, and potassium acetate, in certain proportions. It is then dried in a current of air.

Thus sterilised, meat can be preserved, it is said, for an indefinite period, provided it be kept dry.—E. H. T.

*Sterilising Milk, Improved Method of, and Apparatus for.* N. Bendixen, Copenhagen. Eng. Pat. 12,869, June 8, 1898.

The method employed is to saturate the milk with carbonic anhydride, boil for about half an hour under pressure at a temperature of about 120° C., and then to pass a current of pure, sterile air through it. By this process the lime, lactose, albumin, &c., contained in milk, should remain unaffected, and consequently the latter retains the taste and other properties of the fresh article.

The apparatus consists of a closed container, heated by means of a steam-hood, and having feeding tubes for carbon dioxide and stirrer; it has also two tubes communicating with a cooling apparatus, one in the upper and one in the lower part, the former to lead the steam developed from the milk, the latter to lead the liquid, through the cooling apparatus into a second container, which in its turn has two tubes, one to pass air in by, the other, provided with faucets to run off the sterilised milk.—E. H. T.

## (*B.*)—SANITATION; WATER PURIFICATION.

### PATENTS.

*Clarifying Sewage and Reducing the Refuse, Sewage Filtering Screen or Tank for.* B. Harlow, Macclesfield, Cheshire. Eng. Pat. 22,622, Oct. 1, 1897.

The screen consists of a double row of movable iron bars, from 4 to 7 ft. high, and about 1½ to 2 ins. square or round, fitting in sockets cast in, and flush with, the foundation plate forming the bottom of the screen. The space between the bars is 1—2 ins., and the space between the rows, which is filled with filtering material, about 1 ft. or more. To prevent clogging, the bars are fixed at right angles to each other not square with the screen.—N. H. J. M.

*Purification [by Ozone] of Water and other Liquids, A Process of and Apparatus for.* M. Otto, Neuilly-sur-Seine, France. Eng. Pat. 17,257, Aug. 10, 1898.

The apparatus consists of a closed cylinder containing an inner cylinder open at the top. The water to be purified passes through sieves into the intermediate space between the two cylinders where, by means of emulsers and baffle plates, it is thoroughly saturated with ozonised air which is admitted at the bottom of the cylinder through a valved tube. The water overflows into the inner cylinder and from thence passes directly to the mains. There are special outlets for the excess of ozonised air which may be again utilised. The destruction of organic matter, &c. is accompanied by phosphorescence, the water retaining its luminosity for several seconds.—N. H. J. M.

## XIX.—PAPER, PASTEBOARD, Etc.

*Cellulose, Technical Applications of, for Textiles, Paper, &c.* M. Kitschelt. Farben-Zeit. 1898, **12**, [17], [18], 261, and 282

*See under VI., page* 1043.

*Sulphite Wood Pulp Manufacture, Waste Liquor from [Wood Mordant].* H. Seidel. Rev. Gén. des Mat. Col. **2**, [22], 370.

*See under VI., page* 1043.

### PATENT.

*Cornstalks, Pith of, Obtaining Products from the, and Utilising same.* C. A. Allison, London. From L. Silverman, Chicago, U.S.A. Eng. Pat. 15,758, July 19, 1898.

Pith free from fibres of any kind, is obtained from cornstalks by removing the pith as a whole, and then subjecting it to a shredding or cutting action, lengthwise of the contained fibres. The pith is rendered highly absorbent by gently steaming which causes it to swell up, and then either artificially drying it, or allowing it to dry itself. Prepared in this way it can be used as a pad or bandage for surgical purposes or as an absorbent substance for oils, &c.—[illegible] M.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Thoria, Industrial Extraction of.* Wyrouboff and A. Verneuil. Comptes Rend. **127**, [11], 412—414.

The authors state that their process has given good results in the treatment of many tons of monazite, and that it extracts all the thoria. The mineral is first attacked by one of the known processes, in the case of monazite, for instance, by treatment with $H_2SO_4$. The product is diluted with water, sufficient acid being present to prevent the precipitation of phosphates; the liquid is decanted the following day and is precipitated by half of the amount of oxalic acid required for complete precipitation. The oxalates are washed till free from phosphoric acid, and are heated with 10 per cent. $Na_2CO_3$ solution until completely changed into carbonates, any dissolved $ThO_2$ being precipitated by adding NaOH solution to the liquor. The carbonates are washed and dissolved in hydrochloric acid, when any unconverted oxalates will separate as a slight precipitate, which is worked up separately. To the solution is added barium peroxide in small quantities till the solution gives no further precipitate with hydrogen peroxide. The precipitate, coloured orange-red by cerium peroxide, contains all the $ThO_2$ with 20—30 per cent. of other rare earths; it is filtered off, washed, dissolved in cold concentrated hydrochloric acid; barium is removed by $H_2SO_4$, the solution is diluted so as to contain 15 per cent. of acid and then precipitated by oxalic acid; the oxalates are washed till free from iron, and are extracted by ammonium carbonate solution, as concentrated as possible, to which enough ammonia is added to keep only neutral carbonate present. By using two or three earthenware vessels and working, methodically, all the $ThO_2$, containing about 7 per cent. of other earths, particularly yttria, is quickly extracted. The earths are precipitated by soda, washed by decantation till free from oxalic acid, dissolved in just enough cold nitric acid, diluted till the solution contains 2 per cent. of $ThO_2$ and precipitated by hydrogen peroxide using 7—8 litres of "10 volume" peroxide per kilo. of $ThO_2$. The exceedingly bulky precipitate is drained on a filter and washed till the wash-water gives no precipitate with ammonia. The filtrates are worked up afterwards to recover the little $ThO_2$ which they contain.

Up to this point tap-water and ordinary commercial chemicals are used, and the $ThO_2$ now contains about 0·1 per cent. of $Ce_3O_4$; for the remaining steps, pure reagents and distilled water are required. The precipitate is dissolved in hot, strong nitric acid, and re-precipitated by $H_2O_2$; it is now quite free from Ce, but contains $SO_3$, $P_2O_5$, and CaO. It is dissolved in hydrochloric acid, precipitated by oxalic acid, the oxalates are decomposed by cold sodium hydrate, the hydroxides are washed, dissolved in hydrochloric acid, re-precipitated by ammonia, and finally dissolved in nitric acid, and evaporated to crystallisation.

The authors have found no indications of the alleged new element *Russium*, said to accompany thorium.—H. B.

*Iodoborates.* H. Allaire. Comptes Rend. 1898, **127**, [16], 555—557.

By allowing the vapour of the iodide of the metal to act on the corresponding borate, the author has prepared the iodoborates of magnesium, zinc, cadmium, manganese, nickel, cobalt, and iron.—J. S.

*Silver Sulphophenate.* F. Zanardi. Annali Chim. Farm. 1898, 317; Chem. Centr. 1898, **2**, [13], 712.

The silver salt of *p*-phenolsulphonic acid may be used for antiseptic purposes. It forms needles soluble in 2 parts of water. The best method of preparation is to decompose the barium salt with the calculated quantity of sulphuric acid, and then treat the free sulphonic acid with silver carbonate.—A. S.

*Iodoform, Decomposition of, in Solution.* J. Bougault. J. Pharm. Chim. 1898, **8**, [5], 213—214.

Light has generally been considered the sole cause of the decomposition of iodoform in solution. An ethereal solution of iodoform and a solution of sodium thiosulphate were contained in a closed flask exposed to direct sunlight. The iodoform solution soon became coloured, the flask was shaken so that the iodine was absorbed by the thiosulphate After several repetitions the iodoform no longer decomposed, but did so immediately air was admitted to the flask. The same result follows if mercury, silver, or any other substance without action on iodoform, but capable of combining with iodine, is substituted for the thiosulphate. The solution as obtained colourless in an atmosphere free from oxygen also remains colourless if it be separated from the substance used for removing iodine. These experiments indicate that the decomposition of iodoform in the light is a process of oxidation. Formic acid was isolated as one of the products of the oxidation.—A. C. W.

*Chloroform, The Action of, upon Aqueous Alkali.* J. Thiele and F. Dent. Annalen, 1898, **302**, [3], 273—274. See also this Journal, 1897, 1035; 1898, 78, 387.

If warm 40 per cent. potash be shaken with a few drops of chloroform in a nitrometer, 50—100 c.c. of carbon monoxide are obtained. The reaction was quantitatively studied by warming about 2 grms. of pure chloroform with excess of dilute caustic soda solution on the water-bath at a constant temperature, the liquids being mixed by a Witt's centrifugal stirrer. The formic acid produced was determined at the conclusion of the reaction by acidifying with acetic acid and precipitation with mercurous chloride. At 25° C., 21 per cent. of the chloroform is converted into formic acid, and at 100° C., 66 per cent. The authors consider that the formic acid is produced by the secondary reaction of carbon monoxide on the alkali.—A. C. W.

*Oil of Lemon Grass, Contribution to the Knowledge of.* W. Stiehl. J. Prakt. Chem. 1898, **58**, [13, 14, and 15], 51—101.

The author has isolated three aldehydes from oil of lemon grass by the difference in the behaviour of the respective sodium bisulphite compounds.

*Citriodoric aldehyde*, $C_7H_{13}CH:CH.CHO$, is obtained by dissolving the bisulphite compound of the total aldehydes in water, treating the clear solution with caustic soda in excess, and subjecting the resulting oil to distillation *in vacuo*. It boils at 108°—109° under 12 mm. pressure, and at 228°—229° under 760 mm., has a density of 0·8883 at 20°, a refractive index of $n_D = 1·48538$ at 20°, and is optically inactive. Its molecular refraction is 49·07, compared with 47·53, required by $C_{10}H_{16}O$.

*Geranial* (*Citral*).—Semmler and Tiemann (this Journal, 1894, 417. 1898, 598), have isolated citrol from oil of lemon grass, which is identical with geranial obtained by the oxidation of geraniol with chromic acid. This aldehyde was prepared by the author by treating 1 part of oil of lemon grass with 2 parts of sodium bisulphite (40 to 50 per cent.) and 2 parts of water in the cold. It boils at 111° to 112° under 12 mm. pressure, and at 225°—227° under 760 mm., has a density of 0·8868 at 20°, a refractive index of $n_D = 1·48752$ at 20°, and is optically inactive. Its molecular refraction is 49·34.

*Allo-limonal*, $C_8H_{13}CH_2.CHO$, is obtained by treating 1 part of the total aldehydes of oil of lemongrass with 2 parts of sodium bisulphite and 2 parts of water, when, owing to the instability of the bisulphite compound, the latter is split up and yields allo-limonal. It boils at 117°—119° under 12 mm. pressure, and at 233°—235° under 760 mm. It has a density of 0·9017 at 20°, and a refractive index $n_D = 1·48306$ at 20°. Its molecular refraction is 48·15, and its rotation in 1 dm. tube $[\alpha]_D = -5°·05'$.

The following yields were obtained:—

| | Per Cent. |
|---|---|
| Hydrocarbons | 15 to 20 |
| Citriodoric aldehyde | 40 to 50 |
| Allo-limonal | 25 to 30 |
| Geranial | 8 to 10 |

—D. B.

*Lemon Oil, Further Note on the Constituents of.* J. C. Umney and R. S. Swinton. Pharm. J., 1898, **69**, [1475], 370.

The authors have recently reported (this Journal 1898, 863), that both the Palermo and Messina varieties of lemon oil contain from 1·2 to 1·4 per cent. of geranyl acetate. As the result of further investigation, it is now stated that the acetic ester of linalool, as well as that of geraniol, is one of the constituents of Palermo lemon oil. These alcohols are present in the proportion of 3 parts of linalool and 1 part of geraniol. Messina oil contains no linalyl acetate but only the geraniol ester.—J. O. B.

*Ionone from Lemon-Grass Oil.* F. Tiemann Ber. 1898, **31**, [13], 2313—2329. (See also this Journal, 1897, 1030; and 1898, 598, 599.)

In the isolation of citral from lemon-grass oil by means of the bisulphite compound, repeated treatment with saturated bisulphite solution and ether will not extract the whole of the aldehyde. The non-aldehydic constituents may be isolated from the residue after decomposing the remaining citral by alcoholic potash. The reaction between citral and semicarbazide hydrochloride may be employed for the approximate estimation of citral, thus 50 grms. of lemon-grass oil, 50 grms. of semicarbazide hydrochloride, and 80 grms. of sodium acetate dissolved in dilute alcohol gave, after two days' standing, 55 grms. of semicarbazone, corresponding to 42 grms. of citral. The same essential oil had previously been found to contain 15 per cent. of non-aldehydic constituents. No other aldehyde than citral could be found in lemon-grass oil.

The author criticises the statements of Ziegler (this Journal 1898, 793), as to the "pseudo-violet oil" and "technical violet oil," prepared according to Eng. Pat. 26,350, 1896 (this Journal 1897, 1058). Doebner has already shown that the condensing action of the chloride of lime is due to the calcium hydrate formed by its decomposition by means of acetone (this Journal, 1898, 867). The author has examined the oils in question, prepared according to the method described in the patent specification. From "pseudo-violet oil" pseudoionone is isolated as the semicarbazone; "technical-violet oil" contains ionone as its chief constituent mixed with pseudoionone and a terpene, to which last the commercial violet oil owes its activity.
—A. C. W.

*Essence of Violets, Preparation of Artificial.* F. Fritzsche. Rev. Prod. Chim. **1**, [7], 99.

In place of citral it is proposed to employ lemongrass oil, the yield being said to be superior in point of both quantity and quality.

The following method and ingredients are proposed:—acetone, 45 kilos.; 96° Tr. alcohol, 75½ kilos.; lemongrass oil, 38 kilos.; cobalt nitrate, 30—300 grms. (according to the free-acid content of the lemongrass oil and the amount of basic lime present) are rapidly heated to 50° C., mixed with 75½ kilos. of "chloride of lime" solution (prepared in the cold), and the whole boiled for 18 hours.

The product is distilled in a current of steam, the fraction coming over at and above 155° C.—at 12 mm. pressure—being treated with 15 times its own quantity of a solution of sodium bisulphite of 11° B. (b. p. 140° C.), and stirred continually. The transformation is complete in 3 to 10 days; and the addition of a little iron oxide or chloride is found advisable.—C. S.

*Savine Oil* (*Oleum Sabinæ*). E. Fromm. Ber. 1898, **31**, [13], 2025—2031.

By fractional distillation three main fractions can be obtained from this oil. The first distilling below 195° C. consists, in the main, of terpenes (pinenes) passing over between 156° and 170° C. The second fraction (195°—235° C.) consists of saponifiable esters, whilst the third (235°—310° C.) contains, in addition to resins, the *Cadinene* of Wallach (Ann. d. Chem. **238**, 82). It was with the second fraction that the author's present investigation was made. By repeated fractionation of the esters a substance was isolated, which boiled between 222° and 224°, and which corresponded in elementary composition with the acetic ester of an alcohol of the formula $C_{10}H_{15}.OH$ (not $C_{10}H_{18}O$, as inferred by Schimmel and Co. (Geschäft. Ber. 1895, 39)). This alcohol, for which the name of *Sabinol* is suggested, is present in the oil principally in combination with acetic acid, though a small portion is also in the form of an ester of an acid with a high boiling-point (247° C.), which has not yet been further examined. It can be obtained directly from the oil without previous isolation of the ester by saponifying with alcoholic potash, and distilling the saponification products in a current of steam. The residue contains the higher boiling constituents, together with potassium salts of the acids, whilst the distillate consists essentially of terpenes and sabinol, which can be separated by fractional distillation.

Sabinol boils at 208°—209° C. It is an unsaturated substance readily absorbing halogens and their hydrogen compounds, but the author did not succeed in isolating the addition compounds in a pure state, as he was unable to crystallise them, and on attempting distillation they decomposed with violence. On oxidising sabinol by means of a cold saturated neutral solution of potassium permanganate, it is quantitatively converted into a dibasic acid, $C_9H_{14}O_4$. When crystallised from water this acid melts at 140° C., and on treatment with acetic anhydride yields an anhydride which crystallises from petroleum spirit in needles, melting at 55° C. It is in every respect identical with the α-tanacetogendicarboxylic acid described by Semmler (Ber. **25**, 3348). When subjected to dry heat it begins to decompose at about 200° C. with evolution of carbon dioxide, and at 210° C. there is violent decomposition. The greater part of the decomposition products are soluble in soda solution, and on the addition of a mineral acid to the solution yield an oil which, when purified by repeated distillation, boils at 229° C., and is a monobasic acid corresponding in composition with the formula $C_8H_{14}O_2$.

On heating sabinol with a strong dehydrating agent a violent reaction occurs, in which part of the substance becomes resinated, whilst a smaller part is converted into a new substance, which boils at about 175° C. This is probably a hydrocarbon, but has not yet been obtained in a pure state, or in sufficient quantity for further examination.
—C. A. M.

*Fenchone, Terpenes, and Ethereal Oils, Study of.* O. Wallach. Annalen, 1898, **302**, [3], 371.

*See under XXIV., page 1077.*

*Linalool, Conversion of, into Terpineol Melting at 35°.* K. Stephan. J. Prakt. Chem. 1898, **58**, [13, 14, and 15], 109—120.

From the results of this investigation the author draws the following conclusions:—

1. Acetic anhydride converts linalool into solid *d*-terpineol, so that Barbier's licarhodol can only be a mixture of linalool, geraniol, and terpineol.

2. Glacial acetic acid, when warmed, acts slowly on linalool, with partial conversion into terpineol.

3. In the presence of sulphuric acid, glacial acetic acid readily converts linalool into terpineol.

4. Concentrated formic acid readily converts *l*-linalool into *d*-terpineol, and *d*-linalool (coriandrol) into *l*-terpineol.
—D. B.

*Lupulinic Acid (β Hop-bitter Acid).* G. Barth and C. J. Lintner. Ber. 1898, **31**, [13], 2022—2025.

From hop resin Hayduck (Wochenschr. f. Brau. 1888, 937) separated two bitter resins, which he termed α and β, and one tasteless resin (γ). Crystals were separated from the α and β resins, and as they were of a feebly acid nature they were called α and β lupulinic acid. Following the directions of Bungener (Zeits. f. das ges. Brauwesen, 1884, 94), the authors extracted the acid of lupulin with petroleum ether. From the extract a resinous mass was obtained, and by careful purification with petroleum ether and methyl alcohol, a crystalline β-lupulinic acid was isolated, melting at 92° C., of a bitter taste, and soluble in the ordinary solvents. It resinified with ease, especially on evaporation of its solution. From the results of determinations of carbon and hydrogen and of the molecular weight, the acid would appear to have the formula $C_{25}H_{36}O_4$. β-lupulinic acid reduces ammoniacal silver solution, but does not form crystalline compounds with phenylhydrazine, semi-carbazide, or hydroxylamine. On oxidation with potassium permanganate in alcoholic solution, valerianic acid is formed. The authors believe that α- and β-lupulinic acids belong to the terpenes, and bear a close relationship to the constituents of hop oil.
—J. L. B.

*Lactic Acid, Preparation of, for the Dyer and Printer.* C. Dreher. Färber-Zeit. 1898, **12**, [18], 277—279.

Lactic acid now finds extensive application in the dyeing industries, partly as a reducing agent, and partly as a substitute for tartaric acid.

There are two modifications of ethylidene lactic acid, the optically inactive form, called common or fermentation lactic acid, and an optically active variety known as sarcolactic acid. Only the former is of technical importance, and results when solutions containing certain of the sugars undergo lactic fermentation.

The lactic ferment, as the bacillus is termed, which changes sugar into lactic acid, grows best in solutions at 35°—40° C., to which the usual foodstuffs for its growth—phosphates and nitrogen in some form as nitrates or ammonium salts—have been added. For the production of a pure acid it is essential to use a pure ferment; hence great cleanliness must be observed when working with the cultures; all vessels must be sterilised by the usual methods before use, and contamination with the dust of the air as far as possible avoided.

As a source of a pure ferment, a small culture of the lactic bacillus is procured from a bacteriological laboratory. A gelatin solution is inoculated in the usual way, with some of the culture, and placed in an incubator at 35° C. for 24—36 hours. The colonies containing the pure ferment are picked out, examined microscopically, and fresh cultures made therefrom.

Grape sugar, invert sugar, or molasses, may be employed as a source of sugar; in the latter case, however, a dark-coloured and impure acid results; but, technically, starch is more commonly used.

The conversion of the starch into sugar is effected by the action of diastase. In a well-closed room, sterilised wooden vessels, provided with mechanical stirrers, and containing a boiled solution of starch, mixed with 5 to 10 per cent. of malt, are maintained at 65° C. for a few days. The milky solution becomes transparent, and by Fehling's sugar test, 80 per cent. of the starch will be found to have been converted into sugar.

The contents of the vessels are boiled to sterilise them, the requisite foodstuffs, saltpetre, and phosphates for the ferment added, and, when cooled down to 35° C., they are inoculated with some of the pure culture of the lactic ferment. In a separate room, and likewise in wooden vessels containing about 500 litres, the fermentation is conducted at 35° C. for from four to eight days.

Complete conversion of the sugar into lactic acid only takes place when the acid is neutralised as fast as it is formed, for the growth of the bacilli is stopped by the product of their own life. As soon as the fermentation vat shows an acid reaction, chalk is added.

The completion of the reaction is shown by the bath no longer becoming acid. The contents are then boiled and passed through a filter press. The filtrate contains calcium lactate, and is concentrated until it nearly solidifies on cooling; a quantity of a 60—70 per cent. solution of sulphuric acid added, and the precipitate of calcium sulphate removed by filtration. In this way a solution containing 25—30 per cent. of lactic acid is obtained. By evaporation *in vacuo* the acid is further concentrated to 50—70 per cent., the strength of the commercial article. The yield from 100 kilos. of starch, containing 80 per cent. of the dry substance, is 130 kilos. of 50 per cent. lactic acid solution.

A pure pharmaceutical product may be obtained by recrystallising the calcium lactate several times, converting it into the lead salt, and finally decomposing the latter with sulphuretted hydrogen.—H. I.

*Anisol and Phenetol, Direct Synthesis of.* C. Moureu. J. Pharm. Chim. 1898, **8**, [5], 211—213.

Phenols are obtained by the fusion of sulphonates with caustic potash. If the potash be replaced by an alcoholate, the phenol ethers result—

$$C_6H_5.SO_3Na + NaOCH_3 = Na_2SO_4 + C_6H_5.OCH_3.$$

The reacting substances must be perfectly dry, the sulphonate should be dried at 150° C., and the alcoholate, obtained by solution of sodium in absolute alcohol, should be heated at 200° on the oil-bath in a current of dry hydrogen. The substances are mixed in theoretical proportion as rapidly as possible, and the mixture heated at 350°, moisture being excluded. The reaction is more difficult to accomplish with the higher homologues of the benzene sulphonic acid and of sodium ethylate.—A. C. W.

*Methylenediantipyrin, Identity of Formopyrin with.* A. Ferro. Gaz. chim. ital. **26**, [2], 411; Chem. Centr. 1898, **2**, [11], 633.

The author states that the identity of formopyrin with methylenediantipyrin is confirmed by the similarity between the constants of measurement of crystals of the two preparations.—A. S.

*Mandragorine.* H. Thoms and M. Wentzel. Ber. 1898, **31**, [13], 2031—2037.

The name mandragorine has been applied to a substance, related to atropine, found in various plants of the genus *Mandragora*, growing in the region of the Mediterranean Sea, and which have been employed in medicine since very early times. The dried roots of the plant found near Trieste yield 0·32 per cent. of the alkaloid, which is obtained in the form of a glassy mass melting at 79°. A very dilute solution of it in sulphuric acid placed in the eye produces prolonged mydriasis. It gives a well-crystallised gold-chloride double-salt, melting at 153°—155°. This is, however, a mixture. By fractionally precipitating the double-salt, and recrystallising the fractions, about 90 per cent. of the substance was obtained in the form of a gold-chloride double-salt melting at 163°—164°, and possessing the composition and all the properties of hyoscyamine gold chloride. The base prepared from this salt was hyoscyamine. The nature of the remaining 10 per cent. is not yet definitely ascertained. It is not, however, atropine.
—T. E.

*Oroxylum Indicum, Seeds of.* D. Hooper. Agricultural Ledger, (6), 1898; through Pharm. J. 1898, **69**, [1476], 390.

The seeds of *Oroxylum indicum*, known by the natives of India as "damree," are employed by them as a remedy for ringworm in cattle. When examined, they were found to contain: moisture, 3·65 per cent.; oil, 20·34 per cent.; resin and bitter principle, 12·96; mucilage and albumin, 20·54; fibre, 32·71; ash, 9·80 per cent. The oil was green in colour and bitter in taste, the latter being due to a yellow crystalline bitter principle, doubtless identical with oroxylin, of Naylor and Chapman, isolated by them, in 1890, from the bark of the same tree. The seeds lose their therapeutic activity on heating, and in times of famine have been ground and made into bread, and even eaten raw. It would thus seem that they are devoid of active poison.
—J. O. B.

*Piperidine Series, Syntheses in.—II.* F. B. Ahrens. Ber. 1898, **31**, [13], 2272—2276.

In the electrolysis of a sulphuric acid solution of nitrosopiperidine, a base, $C_{10}H_{18}N_2$, is formed at the anode (Ber. **30**, 533). The base forms a monobenzoyl derivative, but endeavours to prepare a dibenzoyl compound were fruitless. One of the nitrogen atoms is thus of secondary nature. By reduction with tin and hydrochloric acid the base yields two isomeric amines of the fatty series, $C_5H_{11}N$, which differ widely from one another and from piperidine. Both are secondary bases, and both evolve hydrobromic acid on treatment with bromine, forming substitution compounds. This is also the case with the base $C_{10}H_{18}N_2$, which must be regarded as a fatty diamine.

If the original product of the electrolysis of nitrosopiperidine be freed from sulphuric acid by baryta, and distilled with excess of baryta, ammonia and piperidine are to be found in the distillate. The residue contains nitric acid arising from the nitroso-group, and amidovaleric acid, which was identified by means of its platinum double-salt.
—A. C. W.

*Piperidine Series, Syntheses in.—III.* F. B. Ahrens. Ber. 1898, **31**, [13], 2278—2280.

The pyridine bases condense under the influence of sodium to form dipyridyl compounds. Piperidine does not act on sodium in the same manner. But a similar condensation occurs with benzoyl- and nitroso-piperidine. If these substances are left in contact with sodium in anhydrous ethereal solution for 8—14 days, from the products of the reaction of nitrosopiperidine, γ-piperidyl can be isolated, and piperylhydrazine also appears to be formed. From benzoylpiperidine, the compounds $C_{10}H_{18}N_2(C_7H_5O)_2$ and $C_{10}H_{19}N_2(C_7H_5O)$ result.—A. C. W.

*Racemism, Partial.* A. Ladenburg and G. Doctor. Ber. 1898, **31**, [13], 1962. (This Journal, 1898, 614.)

*See under* XXIV., *page* 1078.

*Vanillin Testing.* Chem. and Druggist, Oct. 22, 1898, 676.

*See under* XXIII., *page* 1076.

*Morphine, Estimation of.* A. B. Prescott and H. M. Gordin. J. Amer. Chem. Soc. **20**, [9], 706.

*See under* XXIII., *page* 1076.

## PATENTS.

*Oxyphenylguanidines and their Derivatives, Production of.* J. C. Mewburn, London. From Chemische Fabrik von Heyden, Radebeul, Germany. Eng. Pat. 24,287, Oct. 20, 1897.

The fundamental process claimed consists in causing a carbodiimide to react with an amidophenol, to produce simple or mixed oxyphenyl-guanidines of the general formula $R'O.C_6H_4.N:C(NHR)(NHR'')$. Three claims refer to different methods by which these guanidines may be obtained from the reaction product of carbon bisulphide and *p*-phenetidine. This probably contains the compounds

$$CS(NH.C_6H_4.OC_2H_5)_2 \text{ and}$$
$$C_2H_5O.C_6H_4.NH.CS.SNH_3.C_6H_4.OC_2H_5.$$

These substances, when treated with desulphurising agents (lead oxide) alone, or in the presence of amidophenols, or when heated to 150°—200° C., yield, as intermediate products, carbodiimides, which at once pass into guanidines.

$$C(NC_6H_4.OC_2H_5)_2 + NH_2.C_6H_4.OC_2H_5 =$$
$$C_2H_5O.C_6H_4.N:C(NH.C_6H_4.O.C_2H_5)_2.$$

Another claim is for the process which consists in heating the ureas corresponding to the thioureas mentioned, with alkalis or alkaline alcoholates, which act as dehydrating agents. The new class of oxyphenylguanidines are "characterised by their powerful anæsthetic action."
—A. C. W.

*Tobacco for Ordinary and Medical Purposes, An Improved Process for the Preparation of.* F. J. Kidgell, Thurton, Norwich. Eng. Pat. 26,259, Nov. 11, 1897.

The claim is for the extraction of soluble substances from tobacco by dilute alcohol, steam, or hot water. The extracted substances are distilled and the purified product returned to the tobacco. Drugs may also be added.
—A. C. W.

*Dihydroxytartaric Acid, An Improved Process for the Production of.* H. J. H. Fenton, Cambridge, and E. S. St. B. Sladen, London. Eng. Pat. 27,032, Nov. 18, 1897.

The process for the production of dihydroxytartaric acid is claimed, which consists in the gradual addition to a mixture of dihydroxymaleïc acid, with four times its weight of glacial acetic acid, at a low temperature, of bromine and water, drop by drop, slightly in excess of the quantities indicated by the equation—

$$C_4H_4O_6 + 2H_2O + Br_2 = C_4H_6O_8 + 2HBr.$$

—A. C. W.

*Blood Serum of Swine Immunised against Swine Fever, Process for the Production of a Permanent Preparation containing the White Corpuscles of the.* G. Lorenz, Darmstadt, Germany. Eng. Pat. 13,710, June 28, 1898.

The serum is purified by the addition of calcium chloride in strong solution, 2—4 per cent. of the water contained in the serum being employed, then further by partial precipitation with ammonium sulphate. After filtering, the white corpuscles are precipitated by the further addition of ammonium sulphate, dried on porous plates, and dissolved in a solution of sodium salicylate and glycerin. To the solution obtained there is then added about 0·5 per cent. of sodium carbonate and carbolic acid. The mixture is finally clarified in a centrifugal machine. The process described is claimed.
—A. C. W.

*Medicinal Compounds [Tasteless Quinine], Impts. in, and Process for Preparing the same.* E. W. Grove, Saint Louis, Missouri, U.S.A. Eng. Pat. 17,615, Aug. 16, 1898.

The claims are for the production of a medicinal compound of quinine and paraffin wax or a similar substance insoluble in water, by melting the substances together or by dissolving them in solvents, removing the solvents, and powdering the residue.—A. C. W.

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Explosives, Influence of Temperature on.* B. Heise. Eng. and Mining J. 1898, **66**, [16], 451.

It is stated that, although the technology of safety explosives has made considerable progress in Germany, the theory at present received as to the influence of the explosion temperature on the safety of the explosive has received but little attention, and the progress made in this direction during the last few years is almost exclusively confined to practical experiments, whilst the explosion temperatures of the various explosives have not in any case been determined. The author, in an article published in "Glückauf," has: (1) Determined by calculation both the explosion temperatures and also the useful effect of the safety explosives in general use; (2) Carried out several experiments as regards the safety, explosive action, and shattering effect of the various substances.—A. S.

*Acetaldehyde and Ethyl Aceto-acetate, Action of Mercuric Nitrate on.* K. A. Hofmann. Ber. **31**, [13], 2212.

*See under XXIV., page 1078.*

### PATENTS.

*Manufacture and Production of Explosives, Impts. in the.* A. Luck, Dartford, and C. L. Nichols, London. Eng. Pat. 24,1[illegible]6, Oct. 19, 1897.

In gunpowders composed principally of nitrocellulose a soluble salt, such as sodium nitrate, is incorporated with the mass before it is formed into grains. The dried grains are then hardened by treatment with a weak solvent, such as ether-alcohol. After hardening, the grains are treated with water, when the whole or part of the sodium nitrate can be dissolved out, leaving the grains correspondingly more or less porous.—W. M.

*Matches, Impts. in Manufacture of.* J. Joscht, Vienna. Eng. Pat. 29,[illegible], Dec. 9, 1897.

The sticks, cut to the requisite length and bound together in large "slices" of about 1[illegible],000 to 20,000, are impregnated at one or both ends with the igniting mass, consisting of sodium chlorate, copper sulphate, gum arabic, and tragacanth gum, by pressing these "slices" on an elastic substance, such as felt, flannel, asbestos, &c., which is drenched with the igniting mass. After drying, the matches are paraffined.—W. M.

*Explosives, An Impt. in.* F. L. Thomas. From La Société des Poudres et Dynamites, Paris. Eng. Pat. 29,389, Dec. 11, 1897.

A "flame-extinguishing" salt, such as "sodium hyposulphite" (thiosulphate), ammonium sulphide or sulphate, is mixed with the blasting explosive. The following examples are given:—Ammonium nitrate, 87·5 parts; binitronaphthalene, 10·0 parts; hyposulphite, 2·5 parts. Also: Dynamite, 40 parts; potassium nitrate, 15 parts; binitronaphthalene, 10 parts; ammonium sulphate, 5 parts.
—W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

*Lubricants for Glass Stop-Cocks.* F. C. Phillips. J. Amer. Chem. Soc. 1898, **20**, [9], 678—681.

The following two recipes are given for preparing serviceable lubricants for glass stop-cocks:—

I.

| | Parts. |
|---|---|
| Pure rubber | 70 |
| Spermaceti | 25 |
| Vaselin | 5 |

II.

| | |
|---|---|
| Pure rubber | 70 |
| Yellow unbleached wax | 30 |

The materials are in each case thoroughly mixed when hot, the rubber, which must be pure and fresh, being melted first, and the others stirred in. It is claimed that these preparations satisfy all the requirements of a good lubricant, *i.e.*, they adhere to the glass, lubricate well, are translucent, and not saponifiable.—E. H. T.

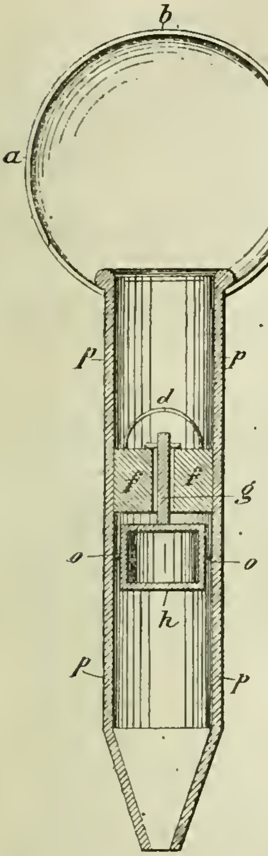
### PATENTS.

*Saccharometer for Testing Beer-Worts, An Improved.* M. Preu and G. Himmel, Tübingen, Germany. Eng. Pat. 29,930, Dec. 17, 1897.

The saccharometer carries on its stem a movable float, which slides up and down. When the saccharometer is immersed in the liquid, the float remains on the surface, and indicates, by its position on the stem, the specific gravity of the liquid. By calibration of the instrument for reading from the top of the movable indicator, the true specific gravity may be determined directly in the vat or pan, and the difficulties caused by the foam and the curvature of the surface, when ordinary saccharometers are used, are avoided. A device is also claimed for clamping the movable indicator on the stem in any position. For this purpose a flat bar extends the whole length of the stem, and terminates at the top in a knob containing a forked crank, operated by a small handle. When the saccharometer is in the liquid, the handle in the knob at the top is pressed inwards. The movement of the handle causes the flat bar to rotate about its axis through an angle of 90°, and the rotation makes it press outwards the jaws of a spring clamp attached to the movable indicator through which it passes, whereby the latter is firmly clamped in the position on the stem which it occupied in the liquid. The whole instrument is then withdrawn from the liquid and the position of the indicator read off.
—J. F. B.

*Pipette for Taking up any desired Quantity of Liquid, A New or Improved.* P. Weissgerber, Lauterbach, Germany. Eng. Pat. 14,519, July 1, 1898.

The pipette *p* has a suction or pressure air ball, *a*, *b*, *c*, mounted thereon, and contains within it a float *h*, having spacing or distance pins *o*, and furnished with a pin *g* passing through and resting upon a stopper or piston *f*, fitting air-tight within the tube. The stopper is adjusted in position, to allow any determined quantity of liquid to be taken up, by means of the loop *d*. Modifications in the shape of the float and piston are illustrated in the specification.—R. S.



*Colorimeter, more especially applicable for Use as an Ozonometer; An Improved.* M Otto, Neuilly-sur-Seine, France. Eng. Pat. 17,524, Aug. 13, 1898.

(*a*) A SYSTEM of adjustable coloured prisms adapted to slide upon one another, and forming a blade having parallel faces of a variable thickness, one of the prisms carrying a vernier adapted to move opposite a graduated scale; (*b*) a series of parallel tubes in two compartments traversed by pencils of rays emanating from one and the same source of light, in such a manner that the path traversed is the same for both gases or liquids to be compared; (*c*) an eye-piece or lens for observing the images formed by the apparatus; (*d*) a pressure-gauge with a movable scale for determining the pressure of the gases. When used as an ozonometer, blue prisms are employed, the coloration due to the ozone being compared with the colour produced by the prisms on a similar column of air. The prisms are adjusted until the colours are equalised; the proportional amount of ozone is read on the vernier scale, and corrected for pressure and temperature.—R. A.

## *INORGANIC CHEMISTRY.—QUANTITATIVE.*

*Hydrogen Sulphide, Estimation of, in Natural Gas.* F. C. Phillips. J. Amer. Chem. Soc. 1898, **20**, [9], 678—691. (Compare this Journal, 1896, 562.)

ANALYSES of the natural gas obtained from borings on Point Abino, a promontory on the northern side of Lake Erie, 10 miles west of Buffalo, point to the following percentage composition:—

| | Per Cent. |
|---|---|
| Hydrogen sulphide | 0·74 |
| Nitrogen | 2·69 |
| Hydrocarbons (paraffins) | 96·57 |

—E. H. T.

*Portland Cement Mortar, The Free Lime* ($CaH_2O_2$) *in Hardened.* — Schuliatschenko. Thonind. Zeit. **22**, 400—401.

LIAMIN separates the crystals of free calcium hydroxide from the other constituents of the finely powdered sample by a method of sedimentation washing in a mixture of methylene iodide and benzene in such proportions that the liquid has a specific gravity of 2·3, and consequently allows the hydroxide to remain in suspension, so that it can be separated and weighed. To confirm the results obtained by this method, he estimates the amount of combined water expelled by a temperature of 450°—480° C., and calculates the result to hydroxide. The figures obtained correspond within 0·4—0·7 per cent.

The actual amount of free hydroxide appears, from analytical data, to increase during the setting of the cement mortar, up to the end of the second month, the increase corresponding to the progressive hardening of the mortar, and seemingly confirming the theoretical view of Le Chatelier, *viz.*, that $3CaO.SiO_2$—the only silicate of lime that is capable of setting hard—decomposes under the influence of water into hydroxide and monosilicate.

On the other hand, the opinion expressed that the free hydroxide is inimical to the durability of the cement, is controverted by practical experience in regard to the stability of cement mortar exposed to the action of sea water. In this connection, experiments recently made by Emelianoff on samples of Portland cement concrete taken from the harbour works at Poti, after 30 years' immersion in the Black Sea, show that only a thin superficial layer (about 3 mm. in thickness) was saturated with carbon dioxide (20 per cent.), the proportion sinking to 5—6 per cent. at a depth of 6 mm., and to mere traces at 2 cm. from the surface, whereas the interior of the mass contained over 33 per cent. of free calcium hydroxide.

It is therefore considered unnecessary to add puzzuolane to Portland cement mortar, in order to increase the hardness of the latter.—C. S.

*Tin, Electrolytic Determination of.* E. D. Campbell and E. C. Champion. J. Amer. Chem. Soc. **20**, [9], 687—690. (Compare this Journal, 1896, 122 and 219; 1897, 1043.)

IN the electrolytic estimation of tin in tin ores, the best results are obtained by electrolysing a solution of the double oxalate of tin and ammonium.

The modification of the usual method here recommended, concerns the method of converting the sodium sulphostannate—obtained by fusing the ore with sodium carbonate and sulphur—into the double oxalate; the necessity of precipitating and filtering out the tin after it has once been obtained in solution as sulphostannate being avoided.

The crushed ore having been converted into sodium sulphostannate in the usual way, the crucible contents are treated with water, and after the insoluble residue has been re-fused, lixiviated, and the solution added to the main body of the sulphostannate, the tin is precipitated as stannic sulphide with hydrochloric acid. After boiling down to 80 c.c., 10 c.c. of the same acid, but of sp. gr. 1·2, and 2·3 grms. of sodium peroxide, are added, thus securing the complete conversion of the sulphide into chloride.

The liquid is then boiled, filtered into a large platinum dish, and ammonia solution and 50 c.c. of a 10 per cent. solution of acid ammonium oxalate are added. The electrolysis is effected over night with a current of 0·10 ampère and E.M.F. = 4 volts, and the analysis completed in the usual manner.—E. H. T.

*Zinc in Ores, Electrolytic Determination of.* Paweck. Eng. and Mining J. 1898, **66**, [15], 424.

THE author uses as cathodes, discs of brass wire gauze, the conductor being joined to the centre of the disc. The zinc is said to settle as a light grey layer, free from spongy zinc, and the whole analysis may be completed in two or three hours. The cathodes may also be amalgamated in a solution of mercuric chloride in nitric acid. The completion of the analysis is determined with the help of potassium ferrocyanide or of sulphuretted hydrogen, any zinc precipitated being redissolved in strong hydrochloric acid. Perforated platinum dishes are used as anodes.

—A. S.

*Nickel, Separation of, from Iron by Means of Electrolysis.* B. Neumann. Chem. Zeit. 1898, **22**, [72], 731—732.

ONLY three electrolytic methods of determining nickel have been employed to any extent in practical analysis, *viz.*, (1) Electrolysis of a solution of the sulphate containing ammonium sulphate and ammonia in excess; (2) Electrolysis after conversion of the salt into nickel ammonium oxalate; (3) Electrolysis of a neutral nickel solution after decomposition with ammonium carbonate. Of these methods the first is the most frequently used, any iron present being first precipitated with ammonia and the filtrate electrolysed, which is not possible in the case of the oxalate method. The author finds that the second and third methods give accurate results with pure nickel solutions. When a filtrate containing only nickel and iron is obtained, he considers that it is not possible to effect their separation by means of electrolysis, and a previous precipitation of the iron is essential in methods 1 and 3. In the double oxalate method, the nickel, cobalt, and iron are readily kept in solution as oxalates, but the decomposition points of the double salts lie too close to one another to permit of a separation by varying the strength of the current.

Classen's method of depositing the nickel and iron simultaneously, dissolving the deposit in dilute acid, and determining the iron volumetrically, is tedious and, in the author's opinion, inaccurate. When the iron is previously removed by precipitation (after oxidation) with ammonia or as a basic salt, it is necessary to redissolve and precipitate it two or three times, in order to free it from the last traces of cobalt or nickel—a course which considerably increases the bulk of the filtrate. Attempts have therefore been made to electrolyse the nickel solution without filtering off the precipitated ferric hydroxide. Engels (Eclairage élect. 1898, **14**, 106) regarded the deposited nickel as pure, but Ducru (Chem. Zeit. 1897, **21**, 780) showed that it invariably contained iron, and recommended its determination and deduction. He further asserted that the quantity of iron deposited was fairly constant, varying between 1 and 2 mgrms., when from 0·4 to 0·5 of the original substance had been taken, and that for technical purposes the error was negligeable. As this statement appeared improbable to the author, he has made experiments with solutions of iron and nickel sulphates under varying conditions of time and strength of current, the results of which show that the amount of iron deposited increases with the amount contained in the solution, and that the time and strength of current have an influence on the results, whilst with incomplete oxidation of the iron, the amount is greatly increased. The quantity of iron, reduced must be attributed—partially, at least—to the action of the hydrogen evolved during the electrolysis. Hence, the conclusion is arrived at that in precipitating nickel electrolytically from a solution containing ferric hydroxide in suspension it is necessary to determine and deduct the amount of iron simultaneously deposited. If the amount of iron in the original substance exceed from 0·05 to 0·1 grm., it is not advisable to neglect it even in technical analyses.

The modifications of the method which the author recommends for the determination of nickel in nickel steel and nickel slag ("Nickelstein"), are:—

*Nickel Steel.*—5 grms. of borings are dissolved in dilute sulphuric acid, the solution oxidised with hydrogen peroxide, bromine water, or nitric acid, ammonium sulphate solution added, the iron precipitated with ammonia, the liquid boiled, and, after the addition of more ammonia, made up to 500 c.c. and filtered. 100 c.c. (= 1 grm. of steel) of the filtrate are treated in the usual way with ammonium sulphate and ammonia, slightly diluted, warmed, and electrolysed with a current of ampèrage 1 to 2, and voltage 3·4 to 3·8. The deposition is complete in from 1½ to 2 hours. The error due to the volume of the precipitated ferric hydroxide is less than 0·5 per cent. A specimen of nickel steel, which, according to a control test in which the iron was removed by repeated precipitation, contained 4·12 per cent. of nickel, gave as the mean result of several determinations by the above method, 4·02 per cent.

*Nickel Slag.*—The sample is dissolved in hydrochloric acid with the subsequent addition of a little nitric acid, the solution evaporated with sulphuric acid until white fumes begin to appear, the residue taken up in hot water, and the copper, arsenic, and antimony precipitated as sulphides. After expelling the sulphuretted hydrogen, the iron in the filtrate is oxidised with bromine water or hydrogen peroxide, and the process continued as in the case of nickel steel. The cobalt present is precipitated simultaneously with the nickel. In one experiment a sample was found to contain 6·02 per cent. of nickel and cobalt, as against 6·15 per cent. by the control method. An analysis of the same sample by Rothe's method, in which the metals are brought into solution as chlorides, the iron and cobalt chlorides shaken out with ether, and the residual nickel chloride solution electrolysed, gave 5·60 per cent. of nickel. Determinations of the cobalt by Ilinski and v. Knorre's nitroso-$\beta$-naphthol method gave 0·48 and 0·51 per cent.

The author considers that for technical purposes the methods described above fulfil the required conditions of accuracy and speed.—C. A. M.

### *Cyanide Solutions, Estimation of Oxygen in Working.* [*Gold.*] A. F. Crosse. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 107—112.

Brief descriptions of Romyn's and Thresh's methods of estimating oxygen in water are described. The author's method for testing cyanide solutions is an adaptation of Thresh's method, as described in Sutton's *Volumetric Analysis.* Before the latter method can be used, all cyanides and absorbents of iodine must be removed. Hence, in practice, the author first treats the solution to be examined with zinc sulphate. A bottle capable of holding 2½ litres of the liquid to be tested is carefully filled and well stoppered, its exact capacity being known. 100 c.c. of the solution are withdrawn for a preliminary test, and are titrated with zinc sulphate solution (200 grms. of $ZnSO_4.7H_2O$ per litre), using phenolphthalein as an indicator, the zinc solution being run into the cyanide until the magenta colour of the latter is just destroyed. The quantity of the standard zinc solution required for the bulk of the cyanide in the large bottle is calculated from this result, and the correct amount is added, without allowing air to enter with it, the stopper of the bottle being replaced as soon as possible. The mixture of cyanide and zinc sulphate is then thoroughly shaken and set aside for the resulting heavy flocculent precipitate of zinc cyanide to settle, which happens after some time, a small scum usually remaining on the surface. The clear liquor is then siphoned off without undue access of air by using a bent glass tube passing through one hole of a doubly perforated cork fitted into the bottle; the second hole carries a short glass tube (arranged as in a washing-bottle), through which air is blown momentarily to start the action of the siphon. The end of the immersed limb of the siphon is covered with a small bag of lint, which filters off any floating particles of precipitate. Two or three (290 to 300 c.c.) pipettes full of the siphoned solution are drawn off and retained. A preliminary test of the iodine-absorbing power of the solution (due to unprecipitated double cyanides) is then made by adding to a quantity equal to that used in the test, 0·9 c.c. of sulphuric acid (half acid, half water), and a few drops of potassium iodide and starch. Dilute bromine water (1 bromine water: 2 water) is added until a blue colour is obtained. Another pipette full of the liquid is now taken, 0·9 c.c. of sulphuric acid and the required amount of bromine water (found from the preliminary experiment) are added, the stopper is put into the wide-mouthed bottle used in Thresh's test, and the pipette is turned over several times. 1 c.c. of the potassium iodide and sodium nitrate solution is then added, and the free iodine—freed in proportion to the oxygen in the solution—is determined by means of standard sodium thiosulphate solution.

By this method the amount of oxygen per litre in certain cyanide solutions was found to be as follows:—Solution from Siemens-Halske process before precipitation, 4·65 to 4·69 mgrms.; tap-water, 7·7 mgrms.; the same tap-water, with 0·2 per cent. KCy and a little ferrocyanide, 7·6 mgrms.; solution as pumped on to a leaching vat, 6·3 mgrms.; the same solution as run from the vat 30 hours later, 0·6 mgrm.; and the same from the end of the zinc boxes, 0·3 mgrm.—W. G. M.

### *Metals, Separation of, by means of Hydrobromic Acid Gas.* E. A. Atkinson. J. Amer. Chem. Soc. 20, [10], 797—810.

The process of separation consists in heating compounds (usually oxygen compounds) of the metals in a stream of hydrobromic acid gas, and collecting in water in a receiver the volatile bromides. In general, the same separations may be effected by hydrobromic as by hydrochloric acid, but in some cases hydrobromic acid does its work more rapidly. Using one or two tenths of a gramme of the material, the bromides of arsenic and of antimony can be driven off in about an hour and a half; that of bismuth in two hours and a half. The lead compound which remains after the action, is not of constant composition, but approximates to $PbO.5PbBr_2$. Copper remains behind as cuprous bromide. No satisfactory separation of arsenic from iron could be effected, but the results were quite accurate in the separation of arsenic, antimony, or bismuth from any of the metals, lead, copper, silver, or cobalt.—J. T. D.

### *Lead Peroxide, The Volumetric Determination of, by Alkaline Arsenite.* C. Reichard. Chem. Zeit. 22, 774—775.

The finely powdered lead peroxide is weighed off into a flask and suspended in water. Excess of a strongly alkaline solution of arsenious acid of known strength, containing about 0·01 grm. of $As_2O_3$ per c.c., is added, and the liquid boiled with addition of more strong soda solution, till the whole of the peroxide has dissolved to a colourless or slightly yellow solution. The liquid is now diluted, excess of sulphuric acid added, and the unfiltered liquid titrated with permanganate. Or, the excess of acid can be neutralised by acid sodium carbonate, the liquid filtered hot, and the filtrate, after cooling, titrated with iodine and starch.

—J. T. D.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

### *Cotton-Seed Oil, The Reactions of.* K. Mazhar. J. Pharm. Chim. 1898, 8, [5], 214—215.

The Turkish law forbids the importation of cotton-seed oil or its admixtures with other edible oils, except they be coloured with alkanet. The author has made comparative tests of 300 samples of oils imported at Salonica, and concludes—

1. The reaction with nitric acid (5 c.c. of oil with 10 drops of acid of density 1·4, free from nitrous acid) is uncertain. 2. Hirschsohn's reaction (5 c.c. of oil with 10 drops of 0·5 per cent. chloroform solution of gold chloride) is not delicate. 3. Becchi's reaction (5 c.c. of oil, 25 c.c. of absolute alcohol, and 5 c.c. of 1 per cent. alcoholic silver nitrate solution) gives doubtful results. The modification of this reaction (10 c.c. of oil, 1 c.c. of 0·5 per cent. alcoholic silver nitrate solution, and 10 c.c. of a mixture of 85 parts of amyl alcohol, 15 parts of colza oil) is more sensitive, but also gives doubtful results. The processes of Millan, and Tortelli and Ruggeri (this Journal, 1898, 607), are long, and do not give absolute certainty. 4. Halphen's reaction (equal parts of oil, amyl alcohol and carbon bisulphide saturated with sulphur), is trustworthy, and superior to the other reactions for cotton-seed oil. (See this Journal, 1897, 1045, where 1 per cent. solution of sulphur in carbon bisulphide is specified.)—A. C. W.

### *Phytosterol and Cholesterol, Detection of, in Fats.* H. Kreis and O. Wolf. Chem. Zeit. 1898, 22, [78], 805—806.

The authors consider that Bömer's methods of extracting these substances (Zeits. Untersuch. Nahr.- u. Genussmittel, 1898, 81 and 545) have the drawbacks that if 50 grms. of the fat are taken, the process is tedious, and enormous quantities of ether are required, whilst if only 10 grms. are taken, the amount of cholesterol or phytosterol obtained is often too small for examination, and the extraction must be repeated in doubtful cases with 50 grms. of the fat. Their objection to v. Raumer's method (this Journal, 1898, 774) is that it is a difficult matter to completely dry the soap obtained from 50 grms. of fat. The following simple process is brought forward as requiring the use of a minimum amount of solvent:—50 grms. of the fat are boiled with 125 c.c. of 95 per cent. alcohol and 25 c.c. of a 40 per cent. aqueous solution of soda until saponified. The alcohol is evaporated off, the soap dissolved in 500 c.c. of boiling water, and the excess of alkali neutralised with hydrochloric acid (sp. gr. 1·124). 100 c.c. of a 10 per cent. solution of calcium chloride are then added, and the flask shaken until the calcium soap separates out, a further addition of calcium chloride being made if the liquid still froths after shaking. The calcium soap is collected on a filter of cotton-cloth, pressed between filter-paper, and boiled for an hour with 100 c.c. of 95 per cent. alcohol. When cold, the alcohol is filtered, the filtrate treated with 3 c.c. of a 40 per cent. solution of soda, in order to saponify any remaining fat, and evaporated to dryness. The small residue is powdered and shaken in a flask with 50 c.c. of ether at intervals during an hour. The residue obtained on filtering and evaporating the ether is dissolved in as small as possible a quantity of hot alcohol, and on cooling, the cholesterol or phytosterol is deposited in pure white crystals. The results of the quantitative application of this method are promised in a future communication.—C. A. M.

## *ORGANIC CHEMISTRY.—QUANTITATIVE.*

### *Beeswax and its Adulterants, The Iodine Value of.* K. Dieterich. Chem. Zeit. 1898, 22, [72], 729—731.

In the determination of the iodine value of beeswax, the author modifies the usual method by taking from 0·5 to 0·75 grm. of the substance, adding 40 c.c. of chloroform, which is allowed to remain in contact with the wax overnight, and then adding the iodine solution. It is shown that the solubility of the substance in chloroform is often an additional test of its purity.

The iodine values thus obtained with 16 specimens of pure yellow wax varied from 8·779 to 10·697 with the Hübl solution, and from 8·427 to 10·466 with the Hübl-Waller solution. All the samples were completely soluble in chloroform. In the case of white wax, which did not dissolve completely in the chloroform, the limits were: with Hübl's solution, 4·164 to 4·373, and with the Hübl-Waller solution, 3·975 to 4·193.

Three specimens of wool-fat wax were also examined. This substance is prepared from the residue in the wool-fat manufacture, and has recently been introduced into commerce. Being cheaper than beeswax, its use as an adulterant is quite possible. As is seen in the subjoined table, the variation in the iodine value is too great for a determination of that constant to afford any information as to its presence in beeswax; but it differs from beeswax in being incompletely soluble in chloroform.

| | Iodine Value. | |
|---|---|---|
| — | Hübl. | Hübl-Waller. |
| 1 | 7·787 | 3·125 |
| 2 | 10·035 | 3·655 |
| 3 | 14·179 | .. |

Of the substances commonly employed to adulterate beeswax, tallow, lard, colophony, and stearic acid, as the author states, increase the iodine value, cerasin and paraffin lower it, whilst Japan wax and Carnaüba wax do not cause any appreciable difference. An addition of Carnaüba wax, however, is indicated by the wax not dissolving completely in chloroform, as is also the case with ceresin and paraffin.

—C. A. M.

### *Hops: Their Valuation and their Volatile Acids.* R. Wetschorkewitsch and M. Bjalobrsheski. Farmazef. 1898, 6, 525.

The value of a sample of hops is decided by the aroma. The ethereal oil of the lupulin consists of a hydrocarbon $C_{10}H_{16}$, and a substance which undergoes atmospheric oxidation to valerianic acid. As the latter is volatile, a diminution of the ethereal oil, and also of the quality of the hops, occurs in proportion to the oxidation. For the estimation of the volatile acid, 25 grms. of hops are distilled in a current of steam, until the distillate ceases to be acid to litmus, after which it is titrated with N/10 caustic alkali. The following number of c.c.'s of alkali were required for 25 grms. of hops of different ages.

| 1897. | 1896. | 1894. |
|---|---|---|
| 32·5 | 22·4 | 15·1 |
| 28·1 | 20·5 | 14·3 |
| 28·6 | 21·7 | .. |
| 27·9 | 19·9 | .. |
| 30·1 | .. | .. |
| 27·6 | .. | .. |

The volatile acid which distilled over was shown to be valerianic acid.—J. L. B.

*Wine Extract, Determination of.* A. Cellerio. Rev. intern. falsific. **11**, 126—127; Chem. Centr. 1898, **2**, [10], 611.

The apparatus used somewhat resembles that proposed by Duclaux for the determination of the dry substance in milk. The narrower limb of a U-tube is connected with a Liebig's bulb apparatus containing sulphuric acid; the wider limb contains a piece of sponge. The sponge is first washed and then dried with the apparatus on the water-bath, a current of air being passed through the tube. 10 c.c. of wine are introduced into the apparatus, and the sponge compressed with a glass rod until it has absorbed all the wine: it is then again somewhat extended by means of a piece of bent platinum wire. The apparatus is then dried on the water-bath for seven and a half hours at 80° C., a current of dry air being passed through the tube. The method is stated to avoid loss of glycerin, and to give results closely approximating to those obtained by the method of determining *in vacuo*, which requires three and a half days.—A. S.

*Chocolate, Estimation of Sugar in.* P. Carles. J. de Pharm. et de Chim. 1898, **8**, [6], 245—247.

16·2 GRMS. of the finely-divided chocolate are introduced into a 100 c.c. flask, with about 80 c.c. of water, and placed on a water-bath for 30 minutes. 2 to 3 c.c. of a solution of basic lead acetate (1/10) are now added, after which the contents of the flask are cooled, made up to the mark with water, well shaken, and filtered. The clear filtrate is then examined in a saccharometer. As the volume of the chocolate amounts to about 50 per cent. of the weight taken, the number read on the saccharometer must be corrected in the ordinary way to obtain the correct percentage of sugar.

An alternative method is to filter the whole of the aqueous extract, and to wash the precipitate with water until the filtrate is brought up to 200 c.c., a portion of the latter then being examined in the saccharometer. As cocoa powder alone yields a small quantity of dextro-rotary substances, a correction of about 0·5 per cent. may be made; but this is not necessary, as the increase is counterbalanced by the small amount of sugar which is obstinately retained by the precipitate.—W. P. S.

*Formaldehyde in Food, Detection of.* E. Rimini. Ann. de Farm. e Chim. **1**, 9; Pharm. J. 1898, **61**, [1471], 363.

If 15 c.c. of a very dilute, aqueous, formaldehyde solution be mixed with 1 c.c. of a 1 per cent. solution of phenylhydrazine-chloral hydrate, 3—4 drops of freshly-prepared sodium nitroprusside solution added, and the mixture rendered alkaline with strong caustic soda, there is formed, on warming, a marked blue coloration, changing to a deep red. On this characteristic reaction a method is based for the detection of formaldehyde in foodstuffs. Milk containing formaldehyde, when tested thus, turns blue to ash-grey, changing after 15 minutes to a red colour.—A. S.

*Ptomaïne resembling Strychnine.* A. Meeke. Nederl. Tijdschr. Pharm. **10**, 258; Chem. Centr. 1898, **2**, [12], 659.

IN the examination of a corpse already putrefying, the author found a ptomaine very similar to strychnine, with the following reactions:—With picric acid, crystalline precipitate, turning violet with sulphuric acid and bichromate of potash; with potassium bichromate, precipitate, turning blue with sulphuric acid; with potassium ferrocyanide, yellow precipitate changing to violet with sulphuric acid. It is distinguished from strychnine by its freedom from bitter taste and physiological action. On evaporating with chlorine water, the residue left behind turned dirty green with ammonia, whilst in the case of strychnine, it remains yellowish-white. The solution in sulphuric acid is first yellow, then red; with Froehde's reagent, first dirty violet, then green.—A. S.

*Vanillin Testing.* Chem. and Drug., Oct. 22, 1898, 676.

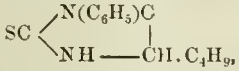
IN the course of an article in the Pharm. Zeit., Welmans mentions that the price of vanillin in 1873 was 7,000 m. (350*l.*) per kilo. (35 oz.), in 1890 it had been reduced to 700 m., in 1896 to 560 m., and last year to 125 m. Hence there is now great temptation to adulterate the article, and, as a matter of fact, such adulteration is taking place, especially with acetanilide. Welmans refers to recent exposures of the matter, and points out that pure vanillin in fine crystals melts in the open air to a colourless fluid, but if the crystals are large the melt is apt to be yellowish, so that a brown or red colour indicates impurity of tarry origin. Pure vanillin dissolves in sulphuric acid to form a lemon-yellow solution similar to that of citric acid in the same solvent. He also points out that vanillic acid is a probable impurity of vanilla made by the oxidation of aceto-eugenol with potassium permanganate, and although vanillic acid has a much higher melting-point (207° C.) than vanillin (83° C.), its presence serves to depress the latter by a few degrees, so that 10 per cent. of vanillic acid in vanillin gives a mixture, melting at 80°—81° C. As a simple means of estimating vanillin he suggests saturation with standard alkali. If 1 grm. of vanillin be shaken with excess (25 to 26 c.c.) of decinormal potash (alcoholic) the mixture becomes viscous and white almost instantly, but on warming to 40°—50° C. it liquefies, and the excess of alkali can be determined with decinormal hydrochloric acid with phenolphthalein as indicator. The proportions recommended are:—Vanillin 1 grm., rectified spirit 25 c.c., decinormal KOH 25 c.c., and phenolphthalein two or three drops. Shake in a 200 c.c. stoppered flask, and titrate excess of alkali with decinormal acid. As 1 grm. of vanillin is equivalent to 0·36842 grm. of KOH [since $C_6H_3.COH.OCH_3.OH$ (152) = KOH (56)], 1 c.c. of decinormal KOH = 0·076 vanillin. Acetanilide does not affect decinormal alkali; vanillic acid does, 1 grm. requiring 0·3 grm. of KOH, so that the amount of either can be calculated if present.

*Morphine, Estimation of.* A. B. Prescott and H. M. Gordin. J. Amer. Chem. Soc. **20**, [9], 706—728. (Compare this Journal, 1898, 282, 612.)

AT the conclusion of a long paper on the preparation and properties of certain alkaloidal periodides, the following volumetric method for the estimation of morphine in pharmaceutical preparations is suggested:—The crude morphine obtained by the pharmacopoeial method is taken up with lime-water, and, after the solution has been made up to a certain volume, an aliquot portion is filtered off and acidulated with hydrochloric acid. An excess of $\frac{1}{10}$N iodine solution is then added, drop by drop, until the periodide of morphine separates out, and the supernatant liquid has a dark red colour. This liquid is made up to a certain volume, half of which is taken and titrated with standard sodium thiosulphate. The amount of the latter used gives the uncombined iodine, and, by calculation, the combined iodine. With 10 grms. of opium, $\frac{1}{10}$ of the lime-water solution would represent 1 grm. of opium, and the percentage of morphine would be given by the product of the iodine consumed and the factor 71·918, or by the product of the volume of $\frac{1}{10}$N iodine solution consumed and the number 0·94793.—E. H. T.

*Pure Leucin, Separation of, from Crude Leucin, or the Nature of the Products derived from Casein, by Digestion with Trypsin.—II.* F. Röhmann. Ber. 1898, **31**, [13], 2188.

Pure leucin can be suitably isolated from the crude leucin (Ber. **30**, 1978) by converting it into its phenyl thiohydantoïn—



$$\mathrm{SC}\begin{matrix}\diagup\ \mathrm{N(C_6H_5)C} \\ \qquad\qquad | \\ \diagdown\ \mathrm{NH — CH.C_4H_9,}\end{matrix}$$

a substance first prepared, together with the thiohydantoïn, of other amido acids, by Aschan (Ber. **16**, 1544; **17**, 426). The method of separation adopted by the author was essentially that proposed by Marckwald, Neumark, and Stelzner (Ber. **24**, 3278):—Eight grms. of the dry, finely powdered, crude leucin are mixed with four grms. of potassium hydroxide and a little water, and decomposed by adding 9 grms. of phenyl mustard-oil dissolved in alcohol, the flask being cooled to moderate the energy of the reaction. The excess of alcohol is evaporated on the water-bath and the mass diluted with water. Any sulpho-carbanilide which is deposited is removed, and the filtrate shaken with ether. The aqueous solution is heated on the water-bath to expel any ether, and hydrochloric acid is added in excess. This causes the impure thiohydantoic acid to separate out as a resinous mass, which, after standing until the next day, changes spontaneously into the thiohydantoïn. The resinous substance is then washed with water and dissolved in hot alcohol, from which, on cooling, a mass of crystals is deposited. These are separated from the mother-liquid and purified by repeated crystallisation from alcohol, and finally from acetic ether. As thus obtained the crystals are fine colourless prisms, several mm. in length. They melt at 178° C., and when crystallised from alcohol, group themselves radially into ball-like masses.—C. A. M.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

*Double Carbide of Iron and Tungsten.* P. Williams. Comptes Rend. **127**, [11], 410—412.

A mixture of 150 grms. of pure calcined $WO_3$, 250 grms. of iron, and 80 grms. of paraffin coke was heated in a carbon crucible for 5—6 minutes in Moissan's electric furnace with a current of 900 ampères and 45 volts. The fused mass obtained was very complex, containing a carbide, WC, probably a certain amount of a carbide $W_2C$, and several double compounds of W and Fe, of which the carbide $3W_2C.2Fe_3C$ was isolated as follows:—The fused mass was treated with hot concentrated HCl till completely disintegrated; the crystalline powder was collected, and was again treated with HCl: it now consisted of two double carbides of W and Fe, the carbide WC, and graphite. The crystalline double carbides, being strongly magnetic, were removed by a magnet, and boiled with *aqua regia*, which dissolved half of the crystals, leaving a compound of definite composition corresponding to $2Fe_3C.3W_2C$. Under the microscope this was seen to consist of very bright little prisms, resembling pyrites. It scratched glass easily, but not quartz, and was very magnetic. Its density at 18° was 13·4. Heated to redness in dry H, it was unaltered; heated similarly in O, it slowly oxidised. At a red heat Cl attacked it, forming chlorides of Fe and W; Br and I acted more slowly; S not at all. Water vapour did not alter it at the softening-point of Bohemian glass, but moist air oxidised it slowly. $HNO_3$ and $H_2SO_4$ dissolved it quickly, and it was decomposed by KOH, $KClO_3$, $KNO_3$, $K_2CO_3$, and $KHSO_4$.—H. B.

*Double Carbides of Iron and Chromium, and of Iron and Molybdenum; The Preparation and Properties of.* P. Williams. Comptes Rend. **127**, [14], 483—484.

Two new carbides, $3Fe_3C$, $2Cr_3C_2$ and $Fe_3C$, $Mo_2C$, have been prepared in the electric furnace. Like a carbide of iron and tungsten similarly prepared (Comptes. Rend. **127**, [11], 410; see previous abstract), they may be considered as compounds of iron carbide, $Fe_3C$, with the metallic carbides prepared by Moissan in the electric furnace. The chromium preparation takes its place in a series with two compounds isolated by Carnot and Goutal, having the composition, $3Fe_3C$, $Cr_3C_2$, and $Fe_3C$, $3Cr_3C_2$. The quantity of carbide obtained is approximately inversely proportional to that of the graphite formed at the same time.—J. A. R.

*Nitric Nitrogen Produced by the Pea.* J. L. Beeson. J. Amer. Chem. Soc. **20**, [10], 793—795.

To ascertain whether the nitrifying organisms in pea roots produce more nitric nitrogen than the pea requires, and whether, therefore, the pea will help to feed another crop, growing among it, experimental plots were planted as below, and the nitrates in the soil determined at the end of September. A blank experiment was made by leaving a plot fallow, but treating it in all other respects like the planted plots. The following are the results; the figures being milligrams of nitric nitrogen in 1 kilo. of soil:—

| | N. |
|---|---|
| Fallow | 1·01 |
| Peas, growing, pods nearly grown | 3·333 |
| Peas, growing, pods about ripe | 8·670 |
| Peas, grown, and dead two or three weeks | 10·510 |
| Corn (hard) and peas same as above | 7·503 |
| Corn (hard) alone | 0·340 |
| Cotton, growing | 0·423 |
| Sorghum, growing | 0·333 |

These results indicate that the peas produce more nitric nitrogen than they require; that the organisms continue to produce it after the pea is dead; and that the corn crop grown along with the peas was fed by the nitric nitrogen which the peas produced.—J. T. D.

*Arsenic, Action of Sodammonium on.* C. Hugot. Comptes Rend. 1898, **127**, [16], 553—555.

By acting on arsenic with sodammonium or by liquefying ammonia in presence of sodium and arsenic, the author has obtained a brick red compound having the composition $Na_3As, NH_3$. The compound is slightly soluble in liquid ammonia, and is usually contaminated by sodamide produced by its own decomposition.—J. S.

*Bismuthous Oxide.* L. Vanino and F. Treubert. Ber. **31**, [13], 2267—2272. (See this Journal, 1898, 613.)

In reference to Schiff's observation that the supposed bismuthous oxide was not attacked by mercury, the authors find that a very small quantity of oxide or metahydrate of bismuth is sufficient to prevent the amalgamation of even freshly precipitated bismuth. Attempts to reduce the chloride by means of hypophosphorous acid, hyposulphurous acid, and formaldehyde gave only grey to black metallic bismuth. In faintly alkaline solutions these reducing agents yield the metal in an extremely fine state of division, and the precipitated oxychloride is coloured blue by it. The same colour was similarly obtained with salts of lead.
—E. W. W.

*Camphor, Solubility of.* C. Istrati and A. Zaharia. Comptes Rend. 1898, **127**, [16], 557—559.

The authors find that water dissolves camphor, the density of a saturated solution at 15° C. being 1·00071, whilst the rotation in a tube 22 cm. long is $[\alpha_D] = +0°·4$. Camphor is also readily soluble in hydrochloric acid at 0° C., 100 c.c. of the solution containing 40,276 grms. of camphor. On warming this solution with the hand, a solid substance separates out, but is again redissolved on cooling.—J. S.

*Fenchone, Terpenes, and Ethereal Oils, Study of.* O. Wallach. Annalen, 1898, **302**, [3], 371—387.

In the oxidation by potassium permanganate of fenchene obtained from the same *d*-fenchone, two different acids are obtained, of the composition $C_{10}H_{16}O_3$, melting at 137°—138° C., and 152°—153° C. Fenchone does not appear to alter in rotatory power under the influence of reagents, and by reduction it yields a single alcohol, *l*-fenchylalcohol, melting at 45° C. The fenchylchloride obtained from this alcohol by the action of phosphorus pentachloride varies in rotatory power according to the means employed to separate it from the reaction product; $[a]_D$ was found to lie between −13° and +5·1°. Levorotatory fenchylchloride, by the action of quinoline or aniline, is converted into *d*-fenchene, whilst *d*-fenchylalcohol yields *l*-fenchene. By long warming with dilute alcoholic sulphuric acid, *d*-fenchene is completely converted into *l*-fenchene. Potassium permanganate readily oxidises *d*-fenchene into the oxyfenchenic acid, $C_9H_{14}C(OH).CO_2H$, of melting-point 138°—139° C., whilst *l*-fenchene is oxidised with more difficulty into *l*-oxyfenchenic acid of melting-point 152°—153° C.

In order to distinguish between the derivatives of *d*- and *l*-fenchone, the author proposes to affix to their names *D* or *L*; thus the fenchenes above mentioned are *D-d*-fenchene and *D-l*-fenchene.

From *l*-fenchone, by a similar series of reactions, *L-d*-oxyfenchenic acid of melting-point 152°—153° C. was obtained. The racemic mixture of *D-l*- and *L-d*-oxyfenchenic acids melts at 142°—143° C. In a previous paper (Annalen, **300**, 315) the oxidation of *D-l*-oxyfenchenic acid in acid solution to a homologue of camphor, fenchocamphorone, $C_9H_{14}O$, was described.—A. C. W.

*Acetaldehyde and Ethylic Aceto-acetate, Action of Mercuric Nitrate on.* K. A. Hofmann. Ber. **31**, [13], 2212—2218.

By the action of acetylene on a solution of mercuric nitrate, or by treating the mixture of acetaldehyde and alcohol obtained in the ordinary method of preparation of the former, with a solution of mercuric nitrate acidified with nitric acid, the author has prepared a white crystalline substance, $C_2Hg_2NO_4H$. It crystallises from aldehyde, is very sparingly soluble in water, and in 3 per cent. nitric acid at the ordinary temperature, is decomposed by stronger acid on warming, and with concentrated sulphuric acid and ferrous sulphate gives the nitric acid test. By the action of warm dilute hydrochloric acid, aldehyde and mercuric chloride are formed, and when heated with an alkaline solution of potassium cyanide, it gives the aldehyde reaction. When heated with ethylic iodide it yields a yellow compound, $C_2Hg_2I_2$, which is insoluble in water, potassium iodide solution, and dilute sulphuric and hydrochloric acids, and becomes rapidly grey on exposure to light. When $C_2Hg_2NO_4H$ is warmed with dilute caustic soda solution, it gives a precipitate which contains metallic mercury and yields $C_2Hg_6Cl_6$ when treated with HCl (Ber. **31**, 1907; this Journal, 1898, 948). The solution probably contains $CHg_3CO_2H$, for hydrochloric acid, nitric acid, and carbonic acid, give white precipitates of salts of the type $C_2Hg_3Cl_3.CO_2H$, which no longer give the aldehyde reactions. When these precipitates are again treated with caustic soda they yield oxymercarbide (*loc. cit.*). By shaking a very dilute solution of mercuric nitrate acidified with nitric acid, with ethylic aceto-acetate at +5°, a white precipitate, $C_6HgO_3Hg$, is formed, which, on heating, decomposes with production of a caramel-like odour and liberation of carbon. It is insoluble in hot water, and when warmed with dilute hydrochloric acid or with potassium cyanide solution, ethylic aceto-acetate is liberated. From its solution in dilute caustic soda solution, carbonic anhydride precipitates a yellowish transparent gelatinous mass.

By the action of mercuric nitrate solution on acetone, a light yellow crystalline compound, $C_3Hg_3H_4O(NO_3)_2$, is formed.

By shaking a mixture of mercuric chloride and potassium hydrogen carbonate in solution, with acetaldehyde, the author obtained a white powder, which, with hydrochloric acid and with alkaline potassium cyanide solution gave the characteristic aldehyde reactions, and with alkalis behaved like Poleck and Thummel's compound obtained from commercial ether (Ber. **22**, 2863), but contained 4 per cent. less of carbon.—E. W. W.

*Partial Racemism.* A. Ladenburg and G. Doctor. Ber. 1898, **31**, [13], 1969—1971. (See also this Journal, 1898, 611.)

The authors sought a "half-racemic" salt of racemic acid. Quinine, quinidine, cinchonine, cinchonidine, and brucine separate racemic acid into *d*- and *l*-tartaric acids. Acid strychnine racemate exhibits a feeble dextro-rotation. Neutral strychnine racemate was prepared by dissolving 2 mols. of base in the solution of 1 mol. of racemic acid. The salt, on decomposition by ammonia, conversion into lead salt, and decomposition by sulphuretted hydrogen, left a completely inactive acid. The properties of this neutral racemate were compared with those of the *d*- and *l*-tartrates. The following table shows the salt to be an individual compound and not a mixture:—

| — | Melting Point. | Water of Crystallisation. | Solubility in 100 parts of Water. | | Specific Gravity. |
|---|---|---|---|---|---|
| | | | At 40° C. | At 20° C. | |
| | ° C. | Mols. | | | |
| Racemate......... | 222 | 6½ | 3·69 | 2·45 | 1·4986 |
| *d*-Tartrate........ | 228 | 7 | 3·10 | 2·03 | 1·5129 |
| *l*-Tartrate........ | 212 | 3½ | 2·04 | 1·70 | 1·6080 |

Additional proofs of the presence of a racemate are as follows:—A molecular mixture of strychnine *d*- and *l*-tartrates melts at 234° C.; on recrystallisation the racemate is obtained melting at 222° C. The same molecular mixture combines with water to be converted into the racemate. The transition temperature does not lie between −15° and +50° C.—A. C. W.

## New Books.

Hygienisches Taschenbuch, für Medicinal- und Verwaltungsbeamte, Aertzte, Techniker und Schulmänner. Von Dr. Erwin von Esmarch, o. ö. Professor der Hygiene an der Universitat Konigsberg i. Pr. 2te vermehrte und verbesserte Auflage. Julius Springer, Berlin. 1898. Price M. 4. H. Grevel and Co., 33, King Street, Covent Garden, London.

This small volume for the pocket, contains 260 pages of subject-matter and 7 pages of an alphabetical index. The following subjects form the groundwork of its construction, and indicate the chapters into which the work is divided. Air, Earth and Soil. Water. Hygienic Conditions. Water Supplies, &c. Hygiene of Buildings and Dwellings generally. Foundations. Details of Building itself. Supply of Light to Rooms. Ventilation. Heating. Removal of Excreta and Refuse, &c., in connection with Hygiene. Schools and Schoolhouses. Hospitals. Avoidance of Infectious Diseases. Disinfection. Disinfectants and Disinfecting Apparatus. Disinfecting, Institutes for.

An Introduction to the Science and Practice of Qualitative Chemical Analysis (Inorganic). By Chapman Jones, Senior Demonstrator of Practical Chemistry at the Royal College of Science, London, &c. Macmillan and Co., Ltd., London. 1898. Price 6*s*. The Macmillan Company, New York.

The aim of the book, as stated by the author, is "to guide the student towards the attainment of systematic work in his qualitative analysis—to help him to learn a sound method, rather than to fill his mind with innumerable isolated reactions." The work is of small 8vo size, and contains 209 pages of subject-matter and an alphabetical index. It contains a few illustrations of apparatus. The subject-matter is classified as follows:—Apparatus. Division of Basic (or Metallic) Radicals into Groups. The Basic Radicals of Group 5. Basic Radicals of Group 4. Basic Radicals of Group 3, Section B. Basic Radicals of Group 3, Section A. Basic Radicals of Group 2, First Section. Remaining Members of Group 2, Second Section. The Acid Radicals. Examination of Group 3A Precipitate when it contains Phosphates (Group 3c). Comparative remarks on the Acids. Method of Analysis. Example Analysis. Insoluble Substances or Residues. Metals and Alloys. Proposed Substance in a Solution. Reagents. Atomic Weights.

Chemistry in Daily Life. Popular Lectures by Dr. Lassar-Cohn, Professor in the University of Königsberg, &c. Translated by M. M. Pattison Muir, M.A. Second Edition, revised and augmented. H. Grevel and Co., 33, King Street, Covent Garden, London, W.C. 1899. Price 5*s*.

In this volume, of small 8vo size, are contained the subject-matter of 12 lectures delivered by Prof. Lassar-Cohn. The matter fills 328 pages of the volume, and there are eight pages devoted to an alphabetical index.

The leading subjects of the 12 lectures, which are stated in the preface to have "caused quite a sensation in German circles," are as follows:—

Lecture I. Breathing. Physics and Chemistry. Air, &c., Combustion. Matches, Yellow and Red Phosphorus. II. Nature of Flame. Candles. Fats. Oils. Petroleum, &c. Cooking by Gas. Incandescent Gas Lights, &c. III. Food of Plants. Manuring. Artificial Manures, &c. Food of Men and Animals. Digestion, &c. IV. Mixed Diet. Butter. Margarine. Starch. Cooking. Sugar. Saccharine. V. Quantity of Food to Eat, &c. Fermentation. Wines, &c. VI. Wine Vinegar. Wood Vinegar. Gunpowder and Explosives. Textile Materials. VII. Tanning. Leather. Bleaching. Bleaching Powder. Coal-Tar Dyestuffs, &c. VIII. Oil Painting. Drying and Non-drying Oils. Inks. Paper and its Manufacture. Patents. IX. Potash. Soda (Leblanc). Acids. Ammonia-soda Process, &c. Soaps. Hard and Soft Waters, &c. X. Glass. Clay. Bricks, Mortars, &c. Photography. The X Rays. XI. Noble and Base Metals. Ores. Relations of Value between Gold and Silver. Bimetallism, &c. Gas Furnaces, &c. XII. Alloys. Alkaloids, Chloral, Ether. Chloroform. Iodoform. Carbolic Acid, &c., &c.

A History of Chemistry from Earliest Times to the Present Day. Being also an Introduction to the Study of the Science. By Ernst von Meyer, Ph.D., Professor of Chemistry in the Technical High School, Dresden. Translated, with the Author's sanction, by George McGowan, Ph.D. Second English Edition. Translated from the Second German Edition, with numerous Additions and Alterations. Macmillan and Co., Ltd., London. 1898. Price 15*s*. The Macmillan Company, New York.

Octavo volume containing prefaces, table of contents, list of abbreviations of names of journals referred to, list of errata, introduction, and subject-matter, filling 594 pages. The work concludes with copious indexes of authors and subject-matter. As historic literature the matter is classified as follows:—I. From Earliest Times to Birth of Alchemy. II. The Age of Alchemy. General History. Special History. III. History of the Iatro-Chemical Period. IV. History of Period of Phlogiston Theory, Boyle to Lavoisier. V. History of Most Recent Periods (from Time of Lavoisier up to now). Lavoisier, Dalton, Berzelius, Gay-Lussac, Liebig, Wöhler and Dumas, Graham, Kolbe and Frankland, Kekulé, Hofmann, Perkin, Baeyer, Ladenburg, Fittig and V. Meyer, Newlands, L. Meyer, and Mendeléeff. VI. Special History of Various Branches of Chemistry, from Lavoisier to the Present Day.

# Trade Report.

## TARIFF CHANGES AND CUSTOMS REGULATIONS.

(*From the Board of Trade Journal.*)

### Belgium.

*Reduction of Duty on Alcohol used in Manufactures.*

A Royal decree, published in the *Moniteur Belge* of the 26th October, extends the relief from excise duty granted to alcohols utilised in the manufacture of fulminate of mercury, in the preparation of pharmaceutical products, and in the manufacture of imitation leathers, from 64 francs to 90 francs per hectolitre of alcohol.

### Netherlands.

*Tariff Modifications.*

A Royal decree, dated 7th October last, grants exemption from taxation for vinegar and ligneous acid on behalf of lucifer match manufactories, on the basis of the regulations enacted by the Royal decree of 12th July 1847, and by the Royal decree of the 26th November 1876, and under such further precautions against abuse as shall be considered necessary by the Minister of Finance.

### Norway.

*Calcium Carbide.*

Carbide of calcium, which is not specially mentioned and which it was intended to treat as indefinite goods, duty 15 per cent. *ad valorem*, it is now proposed to make duty free—on the grounds that as Norway will probably produce large quantities of this particular article, it will be possible to export large quantities of it. It would therefore be injurious to Norway if other countries were to impose a duty upon this product.

### Russia.

*Tariff Classification.*

The Russian Customs Department notifies, under Circular dated the 19th September (1st October) 1898, Sub. No. 19,690, the following classification of goods:—

Solution of coal tar used for coating iron for the purpose of protecting the latter from rust, and usually known under the name of "Siderosten," to be cleared under section 112 of the tariff, duty roubles 1·50 copecks gold, per poud, as a chemical product not specially named.

### Spain.

*Import Duty on Vanilla.*

A Royal order has been published stating that crystallised vanilla (*vainilina cristalizada*), a product used in perfumery and confectionery, will in future be assessed for import duties under No. 318 of the Custom-house tariff.

### Switzerland.

*Tariff Classifications.*

Powders for washing purposes and other similar products will pay 1·25 frs. or 2·50 frs. per quintal, according to the method of packing, on condition that they do not contain more than 15 per cent. of pure soap.

The same products containing more than 15 per cent. of soap, as well as all those known in trade as soap powders, extracts of soap, &c., will pay duty as soap, according to the method of packing—*i.e.*, 5 frs. or 40 frs., whatever be their relation to pure soap.

This decision will enter into force the 1st January, 1899.

### United States.

*Customs Decisions.*

The following decisions relating to the classification of articles in the Customs tariff have recently been given by the authorities:—

*Sulphur, ground*, in sacks, is exempt from duty under paragraph 674, Act of 1897, as sulphur not otherwise provided for; and sulphur in barrels free of duty under the same paragraph as crude sulphur.

*Fancy soap* in the form of artificial fruit is assessed for duty as soap at 15 c. per lb.

*Crude carbolic acid* is entitled to free entry, and also *creosote oil*.

*Pulsatilla*, used in making homœopathic medicines, is assessed for duty at 60 c. per lb. and 45 per cent. *ad val.*

*Calamus-root, orris, gentian, colchicum*, and *rhubarb*, on appeal, have been declared exempt from duty as non-edible drugs.

### *Duty on Cerium Ore.*

In the matter of a protest from Chicago as to duty levied on cerium ore, the Board of General Appraisers has decided as follows:—"The appraiser reports that the merchandise in question is cerite, which is an ore or earth found in Sweden, and contains a mixture of the silicates and oxides of cerium, didymium, and lanthanum, together with traces of other metals and impurities. Cerium is provided for in paragraph 518, but cerium ore is nowhere provided for, and it was accordingly returned for duty under the provision for 'raw or unmanufactured articles not otherwise provided for.' It is claimed that the article is the raw material for the manufacture of cerium, and that it is entitled to free admission under paragraph 518 as cerium, or under paragraph 614.

"We find upon the testimony, the sample, and the appraiser's report, that the merchandise is a crude mineral not specially provided for, and we sustain the claim that it is exempt from duty under paragraph 614."

### CUSTOMS TARIFF OF JAPAN.

The following is a statement of those *ad valorem* rates of import duties, to come into force on January 1st, 1899, which are not provided for in the list of specific duties, which is also given below:—

#### *General* ad Valorem *Tariff.*

| Tariff No. | Classification of Articles. | Rate of Duty. |
|---|---|---|
| | | *Ad Valorem.* |
| | GROUP I.—SCIENTIFIC INSTRUMENTS. | |
| 7 | Crucibles | 10 per cent. |
| 14 | Chemical and philosophical instruments | " |
| | GROUP II. BEVERAGES. | |
| 31 | Mineral waters | " |
| | GROUP IV.—DRUGS, MEDICINES, AND CHEMICALS. | |
| 71 | Antifebrin | " |
| 72 | Antipyrin | " |
| 76 | Bleaching powder | " |
| 88 | Collodion, photographic, with iodiser | " |
| 107 | Manganese, peroxide | " |
| 109 | Musk | 15 per cent. |
| 110 | " (artificial) | " |
| 113 | Pilocarpine, hydrochlorate | 10 per cent. |
| 116 | Potassium iodide | " |
| 118 | Quinine, hydrochlorate, or sulphate | " |
| 131 | Sodium, salicylate | " |
| 136 | All other drugs, medicines, and chemicals | " |
| | GROUP V.—DYES, COLOURS, AND PAINTS. | |
| 137 | Alizarin dyes | " |
| 138 | Anilin dyes | " |
| 140 | Carmine | " |
| 142 | Cochineal | " |
| 143 | Emerald green | " |
| 146 | Gold, silver, or platinum, liquid | 15 per cent. |
| 148 | Indigo, liquid | 10 per cent. |
| 149 | Indigo extract and indigo carmine | " |
| 157 | Smalt | " |
| 160 | Varnish | " |
| [illegible] | All other dyes, colours, and paints | " |
| | GROUP IX.—METALS. | |
| 202 | Antimony | 5 per cent. |
| 217 | Platinum | 10 per cent. |
| 230 | Solder | 5 per cent. |
| | GROUP X.—OILS AND WAXES. | |
| 273 | Gasoline | 10 per cent. |
| 281 | Oil, palm | " |
| 282 | " paraffin | " |
| 286 | All other oils and waxes | " |
| | GROUP XI.—PAPER AND STATIONERY. | |
| [illegible] | Ink, printing, writing, and lithographic. | 15 per cent. |
| 293 | All other paper | " |
| 296 | Sealing wax | " |

CUSTOMS TARIFF OF JAPAN—*cont.*

| Tariff No. | Classification of Articles. | Rate of Duty. |
|---|---|---|
| | GROUP XVI.—MISCELLANEOUS. | |
| 404 | Asbestos | 10 per cent. |
| 408 | Blasting gelatin and similar explosives. | 15 per cent. |
| 412 | Caoutchouc: Crude | 5 per cent. |
| | Caoutchouc: Sheet | 10 per cent. |
| | Caoutchouc: Manufactured | 20 per cent. |
| 420 | Chalk and whiting | 5 per cent. |
| 435 | Fireworks | 30 per cent. |
| 444 | Guncotton | 15 per cent. |
| 453 | Lard, tallow, and grease | 10 per cent. |
| 454 | Leather manufactures | 20 per cent. |
| 456 | Matches | " |
| 468 | Porcelain and earthenware | " |
| 476 | Shoe-blacking | " |
| 488 | Toilet articles and perfumery | 30 per cent. |
| 497 | All other articles, manufactured, unenumerated. | 20 per cent. |

#### *Duty-free Articles.*

Bone ash, guano, tea lead.

#### *Prohibited Articles.*

Adulterated drugs, chemicals, medicines, foods, and beverages considered injurious by laws, ordinances, and regulations.

Articles considered dangerous to public health or to plants and animals.

Articles violating laws of the Empire respecting patents, designs, trade marks, and copyright.

Opium, except imported by Government.

#### *List of Specific Duties.*

The Board of Trade have received a statement showing the specific equivalents which have been fixed by the Japanese Government for a number of the *ad valorem* rates of import duty given in the General Tariff. These specific equivalents are given in the list below, and such articles as as are not mentioned in the list will be charged the *ad valorem* rates. It should be understood that the articles marked (*) below, and printed in *italics*, will remain entitled, on their importation into Japan from the United Kingdom, to the application of the conventional rates of duty under the various treaties.

#### *Articles of Import on which Specific Duties will be Levied.*

| No. in the Statutory Tariff. | — | | Specific Duty. |
|---|---|---|---|
| | | | Yen. |
| 44 | Salt (sea or rock): | | |
| | *a.* Crude | Per 100 kin | 0·083 |
| | *b.* Refined | " | 1·370 |
| | DRUGS, CHEMICALS, AND MEDICINES. | | |
| 136 | Acid, boracic | Per 100 kin | 2·038 |
| 66 | Acid, carbolic (crystals) | Per kin | 0·030 |
| *67 | *Acid, salicylic* | " | 0·157 |
| 68 | Acid, tartaric | " | 0·073 |
| 69 | Alcohol | " | 0·036 |
| 70 | Alum | Per 100 kin | 0·198 |
| *75 | *Bismuth subnitrate* | Per kin | 0·206 |
| 77 | Borax | Per 100 kin | 1·238 |
| 78 | Camphor | Per kin | 0·377 |
| 79 | Cassia, or cinnamon bark | Per 100 kin | 0·732 |
| 80 | Cassia, or cinnamon oil | Per kin | 0·202 |
| 82 | Cinchona bark | Per 100 kin | 1·732 |
| 83 | Cinchonine (muriate or sulphate of). | Per kin | 0·200 |
| 84 | Cinnabar | " | 0·096 |
| 86 | Cocaine hydrochloride | " | 12·983 |
| 91 | Cutch and gambier | Per 100 kin | 0·927 |
| 94 | Glycerin | Per kin | 0·036 |
| 95 | Gum arabic | Per 100 kin | 1·307 |
| 96 | Gum benzoin | " | 1·124 |
| 98 | Gum olibanum | " | 0·560 |
| *100 | *Hops* | Per kin | 0·058 |
| 101 | Iodoform | " | 0·511 |
| 102 | Ipecacuanha | Per 100 kin | 36·620 |
| 104 | Lead, acetate or sugar of | " | 1·282 |

### CUSTOMS TARIFF OF JAPAN—*cont.*

| No. in the Statutory Tariff. | —— | | Specific Duty. |
|---|---|---|---|
| | DRUGS, CHEMICALS, AND MEDICINES—*cont.* | | Yen. |
| 108 | Morphine, hydrochlorate or sulphate. | Per kin | 4·043 |
| *112 | *Phosphorus, amorphous...* | " | 0·165 |
| 113 | Phosphorus, yellow ........ | Per 100 kin | 12·353 |
| 114 | Potassium bromide ........ | Per kin | 0·093 |
| *115 | *Potassium chlorate........* | Per 100 kin | 2·321 |
| 119 | Rosin..................... | " | 0·298 |
| *122 | *Saltpetre.................* | " | 0·980 |
| 123 | Santonine ................ | Per kin | 0·380 |
| 126 | Shellac .................. | " | 0·055 |
| 127 | Soda ash.................. | Per 100 kin | 0·351 |
| 128 | Soda bicarbonate .......... | " | 0·457 |
| 129 | Soda, caustic ............ | " | 0·454 |
| 130 | Soda crystals ............ | " | 0·227 |
| 136 | Nitrate of soda ........... | " | 0·471 |
| 131 | Soda, salicylate........... | Per kin | 0·142 |
| 133 | Sticklac ................. | Per 100 kin | 1·870 |
| 134 | Vaseline.................. | " | 1·642 |
| | DYES, COLOURS, AND PAINTS. | | |
| 139 | Blue, mineral (dry of liquid). | Per 100 kin | 6·690 |
| 141 | Cobalt, oxide of........... | " | 34·628 |
| 144 | Galls of all kinds ......... | " | 1·715 |
| 145 | Gamboge.................. | " | 6·802 |
| *147 | *Indigo, dry...............* | " | 12·953 |
| 150 | Lead (all colours).......... | " | 1·070 |
| *152 | *Logwood, extract of.......* | " | 2·397 |
| *154 | *Paint in oil ..............* | " | 1·304 |
| 158 | Turmeric.................. | " | 0·384 |
| 159 | Ultramarine............... | " | 1·749 |
| 162 | Verdigris ................. | " | 2·297 |
| 163 | Vermilion.................. | Per kin | 0·120 |
| 165 | White zinc................. | Per 100 kin | 1·230 |
| | GLASS AND GLASS MANUFACTURES. | | |
| *167 | *Glass, window, uncoloured or unstained.* | Per 100 sq. ft. | 0·400 |
| | HORNS, IVORY, SKINS, HAIR, SHELLS, &C. | | |
| 185 | Hides or skins, bull, ox, cow, and buffalo (raw, dried, salted, or pickled, and undressed). | Per 100 kin | 0·962 |
| 192 | Ivory or tusks, elephant ... | Per kin | 0·298 |
| *196 | *Leather, sole...............* | Per 100 kin | 7·441 |
| | METALS AND METAL MANUFACTURES. | | |
| | Brass:— | | |
| 203 | Bar and rod ............. | Per 100 kin | 3·070 |
| 203 | Plate and sheet.......... | " | 3·086 |
| 206 | Old (only fit for re-manufacturing). | " | 0·915 |
| | Copper:— | | |
| 208 | Bar and rod ............. | " | 3·464 |
| 208 | Plate and sheet.......... | " | 3·488 |
| 209 | Nails .................... | " | 3·956 |
| 211 | Wire ..................... | " | 7·496 |
| 213 | Old (only fit for re-manufacturing). | " | 0·799 |
| | German silver:— | | |
| 214 | Plate and sheet .......... | " | 6·020 |
| 214 | Wire ..................... | " | 6·257 |
| *232 | *Lead, pig, ingot, and slab ..* | " | 0·368 |
| 233 | Lead ...................... | " | 0·753 |
| *235 | *Mercury ..................* | " | 5·689 |
| 236 | Nickel..................... | " | 3·529 |
| *247 | *Tin, pig and slab..........* | " | 1·992 |
| | Yellow metal or Muntz metal:— | | |
| 250 | Sheet and plate........ | " | 2·871 |
| 251 | Rod and bar........... | " | 2·586 |
| *255 | *Zinc, block, pig, and slab ..* | " | 0·451 |
| *256 | *Zinc, sheet.................* | " | 1·303 |
| 257 | Zinc, old sheet ............ | " | 0·297 |
| 264 | Bronze powder ............ | " | 11·269 |
| | OIL, FATS, AND WAXES. | | |
| *272 | *Candles, all kinds of ......* | Per 100 kin | 3·522 |
| 274 | Oil, beans and peas ........ | " | 6·747 |
| 275 | Oil, castor (in tins, casks, and jars). | " | 1·060 |
| 276 | Oil, cocoa-nut.............. | " | 1·181 |
| 277 | Oil, ground-nut............. | " | 1·122 |
| 278 | Oil, kerosene:— | | |
| | *a.* In tins ................ | Per gallon | 0·016 |
| | *b.* In casks............... | " | 0·010 |
| 279 | Oil, linseed (in tins and casks). | Per 100 kin | 1·724 |
| | OIL, FATS, AND WINES—*cont.* | | Yen. |
| 280 | Oil, olive (in tins and casks) | Per 100 kin | 2·929 |
| 283 | Spirit of turpentine (in tins or casks). | Per gallon | 0·076 |
| *285 | *Wax, paraffin .............* | Per 100 kin | 1·088 |
| | PAPER. | | |
| *292 | *Paper, printing ...........* | Per 100 kin | 1·757 |
| | SUGARS. | | |
| 299 | Sugar (up to No. 14 standard of colour indicated in Dutch specimen colours). | Per 100 kin | 0·204 |
| *300 | *Sugar, refined:—* | | |
| | **a.* (*From No. 15 to No. 20 as indicated in Dutch specimen colours*). | " | 1·524 |
| | **b.* (*Upwards of No. 20 standard colour as indicated in Dutch specimen colours*). | " | 1·828 |
| 301 | Sugar, rock candy.......... | " | 2·213 |
| 302 | Molasses.................. | " | 0·157 |
| 370 | Leather cloths (for furniture, &c.). | Per sq. yd. | 0·043 |
| 371 | Oil-cloths and linoleum cloths (for floors). | " | 0·071 |
| | BEER. | | |
| 385 | Beer, ales. porter, and stout: In bottles containing not more than half a litre. | Per 12 bottles | 0·388 |
| 385 | Beer, ale, porter, and stout: In bottles containing not more than one litre. | " | 0·515 |
| | MISCELLANEOUS. | | |
| 418 | Celluloid (in sheet or rod).. | Per kin | 0·196 |
| *419 | *Portland Cement..........* | Per 100 kin | 0·089 |
| 423 | Coal....................... | Per ton | 0·879 |
| 424 | Coke...................... | " | 0·789 |
| *430 | *Dynamite .................* | Per kin | 0·100 |
| 443 | Glue (common)............ | Per 100 kin | 0·972 |
| 445 | Gunpowder (smokeless powder excepted). | " | 2·617 |
| 446 | Gypsum................... | " | 0·055 |
| 455 | Malt...................... | " | 0·544 |
| 464 | Pitch..................... | " | 0·187 |
| 464 | Wood tar.................. | " | 0·322 |
| 465 | Plaster of Paris............ | " | 0·174 |
| 467 | Plumbago, or black lead... | " | 0·730 |
| 471 | Pulp (for making paper) .. | " | 0·297 |
| 472 | Putty ..................... | " | 0·234 |
| 475 | Sandal wood............... | " | 1·434 |
| *478 | *Soap (for washing).......* | " | 1·085 |

NOTE.—The word "kin" in the above table means the Japanese weight. The words "yard, foot, and inch" are English measures. The words "pounds and tons" are English weights in avoirdupois. The word "gallon" is American standard gallon. The word "litre" is French measure.

## *LEGAL DECISIONS.*

### HIGH COURT OF JUSTICE, QUEEN'S BENCH DIVISION. BEFORE THE LORD CHIEF JUSTICE OF ENGLAND AND MR. JUSTICE WILLS. RIVER RIBBLE COMMITTEE *v.* HALLIWELL—SAME *v.* SHORROCKS.

*Chem. Trade J., Nov.* 5, 1898, 292.

These were two appeals from the decision of a county court judge on two river pollution cases. The two appeals were heard together.

Mr. Cripps, Q.C., and Mr. F. Mellor appeared for the appellants, the Ribble Joint Committee; Mr. Danckwerts appeared for the respondent Halliwell; and Mr. Bradbury for the respondent Shorrocks.

The court upheld the decision of the county court judge.

The Lord Chief Justice, in giving judgment, said that the case was important because, on the one hand it was important to prevent pollution of rivers, and on the other hand, it would be very important to the manufacturers if a certain view of the Act were taken. There were two questions. The first, whether there was such evidence as to make it the duty of the county court judge to find that an offence had been committed under section 2. Secondly, what was the effect of section 17? The scope of the Act was to prevent pollution of rivers. Section 2 created the offence. The only words therein applicable to the respondent in the case were, permitting "solid putrid matter" to fall into the river. The matter must be both putrid and solid, and that at the time it entered the river. If the matter were not both putrid and solid, the offence charged would not have been committed. Section 20 defined solid matter, section 6 dealt with liquid matter. His Lordship then went through the facts, and pointed out that the respondent did not add any solid matter to the water in the reservoir, but merely retained the water as it came to him. The definition in the Act, of solid matter, excluded "particles in suspension in water." Here 97·6 per cent. of the effluent at the time it came into the river was water, and only 2·4 per cent. solid. Could that be called solid matter? No one could say so. It did not, therefore, come within the material words of the section. It was largely a question of fact, and the county court judge had held that it was not solid matter. The court could not say that he was wrong. On the second question he was of opinion that, even if solid putrid matter were being put into the river by the respondents, under the circumstances section 17 of the Act protected him. He had the right of impounding the water and could only use it as it came to him with the pollution from above. It was a necessary consequence that the water should become putrescent, but he had the right to impound the water, and the section held him harmless from the necessary consequences of the reasonable exercise of that right.

Mr. Justice Wills concurred, pointing out that the right in the riparian owner to impound the water implied the right to return the water to the river when he had done with it.

Leave to appeal was given.

### A Patented Medicine not a Secret Medicine.

*Chem. and Druggist, Oct.* 29, 1898, 691.

The Berlin Supreme Court gave an important judgment on October 17 concerning Dr. Robert's "Hæmogallol." The maker, Merck, of Darmstadt, and Mr. Sandhagen, an editor, were accused of having praised this preparation in the press, against the police regulations as to secret remedies (*Geheimmittel*). The Court of Aldermen fined the defendants, but the Correctional Tribunal reversed this decision on the ground that the preparation was not a secret remedy, the process of manufacture having been patented by Dr. Merck. The State Attorney appealed against this decision, but the Supreme Court dismissed the appeal, declaring that a medicine which is patented is no "Geheimmittel."

## *GENERAL TRADE NOTES.*

### The Discovery of Mica in Coorg.

*Imp. Inst. J., Nov.* 1898, 315.

The annual report of the Director of the Geological Survey for India contains an interesting account of Mr, T. H. Holland's recent explorations in Coorg (*Indian Agriculturist*, Sept. 1898).

The discovery of pegmatites amongst the minerals of Mecara led to the institution of a careful search for mica with the result that at five or six localities sheets above the average market size have been obtained. The largest were found on Elk Hill, where the outcrop of the vein is immediately beneath the soil, the sheets being quite free from warping, and measuring some 30 ins. in width. All the samples are of good colour, and show a high degree of elasticity, though the value of some is reduced by the presence of ferruginous inclusions between the cleavage planes, and occasional warping. The extent of depreciation in value resulting from these defects is now being ascertained in London.

It seems extremely unlikely that the best veins should have been struck first in such a completely soil-covered area, but there is sufficient evidence that the mica-bearing pegmatites extend over a wide area, and, if worked with discretion, should be a source of considerable profit.

### Tartar Crystals in Italy.

*Ch. of Commerce J., Oct.* 1898, 218.

The manufacture of tartar crystals, a secondary industry in Calabria, but intimately associated with the wine-growing industry, and, for that reason, formerly flourishing in the district, has of late years fallen off and is now almost abandoned. In 1878 there were in Calabria not less than 28 factories producing bitartrate of potash, and nine in the town of Reggio alone; now only one or two remain working and without hopes for the future. Their output is, however, more in demand than ever, and traders state that it is capable of successful and fruitful exportation, even to Australia, but instead of reducing the tartar to "brown or white salt," it can be sold in original cakes at, consequently, very cheap prices.

### Terms for Copper Smelting.

*Eng. and Mining J., Oct.* 22, 1898, 481.

It is stated that the English smelters, after a conference, have agreed to modify still further their standard terms for treating copper furnace material. They will abandon the "miner's ton" of 2,352 lb. and the draft. While they still retain the returning charge on silver, they have reduced this 6*d*. to 2*s*. per unit of copper, and have agreed to buy at a price per unit of copper by assay.

These changes are a matter of interest to the copper trade; they will greatly simplify English invoices and will bring English terms of sale much nearer to the American. The reason is doubtless the increasing difficulty found by the English smelters in obtaining supplies of material.

### German Trade with South Africa.

*Chem. and Druggist, Nov.* 5, 1898, 737.

The official statistics given below relate to South African imports from Hamburg (the chief German port), during 1887, of articles allied to the drug trade:—

| Drugs, Chemicals, &c. | Cape Colony and Natal. | | Transvaal. | |
|---|---|---|---|---|
| | | £ | Tons. | £ |
| Carbolic acid ........... | 6 tons | 203 | .. | .. |
| Cyanide of potassium ... | 34 „ | 2,800 | 782 | 56,657 |
| Drugs and chemicals (various). | 88 „ | 6,664 | 14 | 526 |
| Essences and extracts... | 25 „ | 2,066 | 3 | 205 |
| Ether.................... | 7 „ | 379 | .. | .. |
| Glycerin................ | .. | .. | 645 | 36,358 |
| Perfumery.............. | 54 tons | 4,946 | 7 | 741 |
| Quicksilver............. | .. | .. | 2 | 397 |
| Quinine and salts of quinine. | 284 lb | 161 | .. | .. |
| Salicylic acid ........... | 1 ton | 204 | .. | .. |
| Sugar of lead ........... | 5 tons | 102 | .. | .. |

It should be noted that the figures refer only to Germany's direct over-sea trade with South Africa in German bottoms, *viâ* Hamburg, no shipments of any consequence being made to that country from other German ports. The German imports from Cape Colony and Natal include aloes, 40 tons, valued at 776*l*., and medicinal leaves and flowers, valued at 171*l*.

### Sugar-Cane Cultivation in the Hawaiian Islands.

*Chem. Trade J., Nov.* 5, 1898, 283.

The sugar industry of the islands prospered during the year 1897, all the plantations paying large dividends. Although the financial success of the sugar business is largely due to the treaty of reciprocity with the United States, there can be no doubt that a considerable measure of it is also due to the intelligent systems which have been adopted by the planting community, and to the introduction of improved methods of agriculture and manufacture. A few years ago the sugar planters of the islands formed themselves into an association, and established a laboratory and experimental station, from which much benefit has been derived. The director of this association makes periodical visits to the plantations, and prescribes such fertilisers as may be required, after a careful analysis of the soil has been made by the chemist of the association. The analyses of all fertilisers used by the planters have to be guaranteed by the manufacturers, and on their delivery they are sampled and analysed at the association laboratory at the expense of the manufacturers, who are not paid until their goods pass the tests made for the buyers. In this way fraud and careless manufacture are checked, and the planters are protected. At the experimental station of the association, sugar cane of different varieties is grown and tested, and various experiments of value are continually being made in fertilisers, irrigation, and other matters connected with the successful raising of sugar cane.

The favourable weather of 1896, and the unusually large area of cane harvested, brought the 1896–97 crop to the highest point yet reached, namely, 251,126 tons of 2,000 lb. The 1897–98 crop will probably be under this, owing to the exceedingly dry weather last year.

After much experimenting, it has been finally decided that the most effective machine for the extraction of the juice from the cane is the "nine-roll mill," which is made up of three three-roll mills, so that the cane is pressed six times, and on its passage from one mill to the next it is subjected to "maceration," by means of a stream of hot water, which assists in forcing the cane to yield its juice. The rollers of these mills vary in size, but the usual measurement is 30 inches in diameter and 60 inches in length, though some mills in use and now being erected have slightly larger rollers. The pressure of all these new mills is controlled by hydraulic means. The Krajewski crushers and the National shredders are used in several factories in front of the mills, and they are both said to do good work in the preparation of the cane for the rollers.

Clarification of juice by the Deming apparatus is gradually extending, and crystallisation in motion is now attracting the attention of those interested in the introduction of what is best amongst the novelties of sugar-making. Amongst the advantages claimed for the Deming system by those acquainted with its working, are, that it economises from 25 to 33 per cent. over the open system and secures better results; that it is nearly automatic, one man easily operating it; and that the saving in labour often exceeds 10 per cent. on a sum twice greater than the total cost of the system. Chemical control in the sugar houses is still somewhat neglected, although there are marked indications of a desire on the part of mill managers to have resident chemists to check all losses.

The number of sugar plantations on the islands is 56. Of these the output in 1897 was as follows:—

| | Number of Plantations. |
|---|---|
| Over 15,000 tons | 1 |
| From 10,000 to 12,000 tons | 5 |
| " 8,000 " 10,000 " | 6 |
| " 6,000 " 8,000 " | 7 |
| " 4,000 " 6,000 " | 4 |
| " 2,000 " 4,000 " | 11 |
| " 1,000 " 2,000 " | 15 |
| Under 1,000 tons | 7 |

Of these 56 plantations, 44 are fully equipped with factories, the remaining 12 estates sending their cane to the adjacent factories, where it is manufactured into sugar.

### SULPHUR IN JAPAN.

*Eng. and Mining J.*, 1898, **66**, [16] 454.

The sulphur of Japan is nearly all obtained from surface volcanic deposits. The output is gradually decreasing. The exports amounted to 6,723 "long tons" in 1897, of which 4,523 tons went to the United States. The consumption in Japan, chiefly in the manufacture of sulphuric acid, is estimated at 2,500 tons.—A. S.

### MINERAL PRODUCTION OF NEW ZEALAND.

*Bd. of Trade J.*, *Nov.* 1898.

The *New Zealand Trade Review* furnishes the subjoined particulars of the value of the mineral productions of New Zealand for the years ending 31st December 1897 and 1896, taken from the annual statement of the Minister of Mines:—

| | 1897. | | 1896. | |
|---|---|---|---|---|
| | Quantity. | Value. | Quantity. | Value. |
| | Oz. | £ | Oz. | £ |
| Gold | 251,645 | 980,204 | 263,694 | 1,041,428 |
| Silver | 183,892 | 20,872 | 94,307 | 10,589 |
| | Tons. | | Tons. | |
| Antimony ore | 10 | 157 | 21 | 450 |
| Manganese ore | 180 | 541 | 65 | 205 |
| Mixed minerals | 1,561 | 5,892 | 37 | 1,335 |
| Coal | 840,713 | 451,915 | 792,851 | 428,648 |
| Kauri gum | 6,641 | 398,010 | 7,126 | 431,323 |

The decrease is in gold and kauri gum, silver and coal showing an increase. Although not yet of important dimensions, the value of mixed minerals also shows a substantial increase. The total for 1896, though better than that for 1897, was in its turn below that for 1895 by 91,736*l.*, the value of gold alone being less by over 120,000*l.*

### CHECKING THE ADULTERATION OF OLIVE OIL.

*Pharm. J.*, *Nov.* 19, 1898, 512.

To check the adulteration of olive oil with cotton-seed oil, the Council of Government of Malta has passed a new Ordinance (No. VI.), which regulates the duty on oil. The duty now established is (*a*) 6*d.* per caffiso ($4\frac{1}{2}$ imperial gallons) on oil of every description) with the exception of linseed oil, of other oils used as medicine, and of any oils which, being unfit for food, are intended for industrial purposes; and (*b*) 1*s.* per caffiso on cotton seed oil or any oil containing any mixture of cotton-seed oil. This measure originated in a report made by the Chamber of Commerce to the effect that cotton-seed oil was being mixed with olive oil and sold as olive oil. The Collector of Customs, in supporting the report, called attention to the loss that the revenue suffered by such mixture in connection with the duty on olive oil. To prevent importers of cotton-seed oil deceiving the public and defrauding the Treasury, it was considered that a 1*s.* duty on cotton-seed oil or any mixture thereof, compared with a 6*d.* duty on olive oil, would be sufficient to check fraud.

### FOREIGN CHEMICAL PATENTS IN THIS COUNTRY.

*Pharm. J.*, *Nov.* 19, 1898, 534c.

Chemical processes are frequently protected by patents in Great Britain for several years after the foreign patents have expired. The Manchester Chamber of Commerce has now decided to raise the question in the next session of Parliament. It is thought that a short Bill will be introduced for the simultaneous expiry of home and foreign patents.

### PLATINUM IN RUSSIA.

*Eng. and Mining J.*, *Nov.* 12, 1898, 574.

United States Consul-General W. R. Holloway sends from St. Petersburg the following article, translated from the *Novoe Vremia*: "The Ourals have been until now the only source from which platinum has been procured for the international market. Its part formed over 95 per cent. in the universal production of that precious metal. A small group of platinum mines, concentrated in the Verkhotinsk district of the government of Perm, includes about 70 separate mines, of which only 40 are being worked and the remaining 30 are either inactive or under investigation. During 1897 the production of platinum amounted to about 6 tons, of which separate enterprises produced the following amounts: Mines of Count P. P. Shuvaloff, 1 ton 396 lb.; mines of

Count Demidov San Donato, 1 ton 141 lb.; mines of J. Burdakov and Sons, 1,512 lb.; mines of Kolly, 1,404 lb.; mines of Andreieff heirs, 1,188 lb.; mines of Koenigsberger, 1,008 lb.; mines of P. A. Konukhoff, 792 lb.; mines of 19 small proprietors, 1,620 lb. Two of the above-named mines passed, in 1897, into the hands of foreigners. In the beginning of September other enterprises were sold to a Syndicate from Paris, with a capital of several million francs. The price was fixed according to the productiveness of the mines. There are now only seven great platinum enterprises in the Ourals, of which five have been purchased by foreigners. By the sale of these mines, Russia has lost the advantages which she could acquire from platinum industry."

### General Summary of the Mineral Produce of the United Kingdom and of the Isle of Man.

*Chem. Tr. J., Nov.* 19, 1898, 324.

| Description of Mineral. | 1896. | 1897. | |
|---|---|---|---|
| | Quantity. | Quantity. | Value. |
| | Tons. | Tons. | £ |
| Alum clay (bauxite) | 7,249 | 13,327 | 2,823 |
| Alum shale | Nil | 611 | 76 |
| Arsenic | 3,616 | 4,165 | 74,795 |
| Arsenical pyrites | 8,808 | 13,137 | 10,734 |
| Barytes | 23,737 | 22,723 | 24,117 |
| Basalt, diorite, &c. | 2,286,209 | 2,335,554 | 441,391 |
| Bog ore | 6,652 | 7,124 | 1,781 |
| Calc spar | .. | 4,066 | .. |
| Chalk | 3,559,229 | 3,858,148 | 163,595 |
| Chert and flint | 107,267 | 93,710 | 16,495 |
| Clays | 11,341,782 | 12,705,196 | 1,453,128 |
| Coal | 195,361,260 | 202,129,931 | 59,740,009 |
| Copper ore | 8,970 | 7,132 | 18,706 |
| Copper precipitate | 198 | 220 | 2,320 |
| Fluor spar | 394 | 297 | 397 |
| Gold ore | 2,765 | 4,517 | 6,282 |
| Granite | 1,756,816 | 1,847,323 | 552,604 |
| Gravel and sand | 1,268,310 | 1,350,787 | 111,332 |
| Gypsum | 193,311 | 181,385 | 66,978 |
| Iron ore | 13,700,764 | 13,787,878 | 3,217,795 |
| Iron pyrites | 10,017 | 10,583 | 4,525 |
| Jet | 294 | 84 | 8 |
| Lead ore | 41,089 | 35,338 | 275,409 |
| Limestone (other than chalk). | 11,011,350 | 11,003,521 | 1,155,993 |
| Manganese ore | 1,080 | 599 | 351 |
| Mica | Nil | 4,983 | 1,727 |
| Nickel ore (or cobalt) | .. | 300 | 300 |
| Ochre, umber, &c. | 9,891 | 14,422 | 12,997 |
| Oil shale | 2,419,525 | 2,223,715 | 555,936 |
| Petroleum | 12 | 12 | 29 |
| Phosphate of lime | 3,000 | 2,000 | 3,500 |
| Plumbago | Nil | Nil | .. |
| Quartz | 519 | 452 | 339 |
| Salt | 2,022,357 | 1,903,493 | 620,898 |
| Sandstone | 4,507,745 | 4,964,109 | 1,524,700 |
| Slag | 582,298 | .. | .. |
| Slate and slabs | 586,233 | 609,194 | 1,649,576 |
| Soapstone | Nil | 28 | 98 |
| Strontium sulphate | 18,042 | 14,987 | 4,310 |
| Tin ore | 7,093 | 7,120 | 251,218 |
| Uranium ore | 35 | 30 | 1,367 |
| Wolfram | 43 | 125 | 2,008 |
| Zinc ore | 19,319 | 19,278 | 69,154 |
| Total value | .. | .. | 72,043,801 |

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 31st October. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,853,430 | 1,657,870 |
| Chemicals and dyestuffs | 329,470 | 312,889 |
| Oils | 735,969 | 643,011 |
| Raw materials for non-textile industries. | 5,211,185 | 5,445,358 |
| Total value of all imports | 39,011,763 | 38,601,673 |

### Summary of Exports.

| Articles. | Month ending 31st October. | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 3,053,570 | 2,775,407 |
| Chemicals and medicines | 708,696 | 673,680 |
| Miscellaneous articles | 2,909,626 | 2,802,223 |
| Total value of all exports | 19,283,052 | 19,863,019 |

### Imports of Metals for Month ending 31st October.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Copper:— | | | | |
| Ore .......... Tons | 10,264 | 8,114 | 80,404 | 66,986 |
| Regulus .......... „ | 9,016 | 5,573 | 235,560 | 128,520 |
| Unwrought .......... „ | 4,862 | 5,000 | 247,260 | 259,563 |
| Iron:— | | | | |
| Pig and puddled bar. „ | 14,994 | 13,478 | 58,605 | 54,501 |
| Ore .......... „ | 410,797 | 317,345 | 301,223 | 232,842 |
| Bolt, bar, &c. .......... „ | 7,521 | 8,553 | 58,859 | 74,496 |
| Steel, unwrought .......... „ | 1,844 | 3,780 | 20,049 | 23,820 |
| Lead, pig and sheet „ | 14,124 | 17,245 | 185,209 | 221,443 |
| Pyrites .......... „ | 53,330 | 41,361 | 90,516 | 75,267 |
| Quicksilver .......... Lb. | 25,510 | 102,710 | 2,350 | 10,051 |
| Silver ore .......... Value £ | .. | .. | 155,508 | 97,378 |
| Tin .......... Cwt. | 58,372 | 33,716 | 181,093 | 126,423 |
| Zinc .......... Tons | 5,165 | 5,922 | 89,346 | 122,636 |
| Other articles ... Value £ | .. | .. | 147,448 | 164,140 |
| Total value of metals | .. | .. | 1,853,430 | 1,657,870 |

### Imports of Chemicals and Dyestuffs for Month ending 31st October.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali .......... Cwt. | 14,416 | 23,424 | 7,161 | 13,308 |
| Bark (tanners', &c.) „ | 17,639 | 29,991 | 5,400 | 11,743 |
| Borax .......... „ | .. | 46 | .. | 46 |
| Brimstone .......... „ | 34,003 | 33,324 | 7,593 | 7,939 |
| Chemicals ...... Value £ | .. | .. | 114,414 | 92,921 |
| Cochineal .......... Cwt. | 350 | 222 | 2,063 | 1,347 |
| Cutch and gambier Tons | 1,344 | 767 | 22,705 | 14,234 |
| Dyes:— | | | | |
| Alizarin ....... Value £ | .. | .. | 17,107 | 16,872 |
| Anilin and other „ | .. | .. | 42,189 | 40,546 |
| Indigo .......... Cwt. | 353 | 369 | 4,623 | 6,068 |
| Nitrate of potash . „ | 15,079 | 19,631 | 12,115 | 13,283 |
| Valonia .......... Tons | 1,732 | 1,389 | 17,141 | 14,553 |
| Other articles ... Value £ | .. | .. | 76,939 | 80,028 |
| Total value of chemicals | .. | .. | 329,470 | 312,889 |

### Imports of Oils for Month ending 31st October.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Cocoa-nut .......... Cwt. | 33,593 | 35,006 | 35,218 | 39,759 |
| Olive .......... Tuns | 1,286 | 731 | 46,951 | 26,994 |
| Palm .......... Cwt. | 102,037 | 65,953 | 108,272 | 71,728 |
| Petroleum .......... Gall. | 19,795,715 | 16,597,237 | 347,869 | 292,649 |
| Seed .......... Tons | 2,284 | 2,922 | 52,419 | 61,888 |
| Train, &c. .......... Tuns | 3,314 | 1,942 | 48,980 | 30,040 |
| Turpentine .......... Cwt. | 14,552 | 34,207 | 12,724 | 40,095 |
| Other articles .. Value £ | .. | .. | 83,536 | 79,858 |
| Total value of oils... | .. | .. | 735,969 | 643,011 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 31ST OCTOBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 2,543 | 1,882 | 4,724 | 2,589 |
| Bristles.......... Lb. | 389,298 | 283,286 | 37,410 | 27,288 |
| Caoutchouc...... Cwt. | 35,830 | 41,600 | 388,375 | 526,172 |
| Gum:— | | | | |
| Arabic......... „ | 2,755 | 5,892 | 5,840 | 14,019 |
| Lac, &c. ....... „ | 10,590 | 2,605 | 35,062 | 8,431 |
| Gutta-percha .... „ | 5,660 | 4,699 | 61,490 | 51,659 |
| Hides, raw:— | | | | |
| Dry............ „ | 31,889 | 26,283 | 72,729 | 68,669 |
| Wet........... „ | 59,298 | 68,904 | 126,961 | 147,447 |
| Ivory............ „ | 949 | 809 | 39,289 | 36,596 |
| Manure:— | | | | |
| Guano......... Tons | 1,312 | 5,448 | 5,248 | 28,288 |
| Bones ......... „ | 5,121 | 7,009 | 14,780 | 28,611 |
| Nitrate of soda... „ | 3,245 | 6,588 | .. | .. |
| Phosphate of lime „ | 23,052 | 24,669 | .. | .. |
| Paraffin.......... Cwt. | 67,483 | 79,693 | 57,880 | 70,461 |
| Linen rags....... Tons | 1,809 | 1,754 | 18,359 | 14,934 |
| Esparto.......... „ | 12,690 | 11,319 | 49,314 | 47,792 |
| Pulp of wood:— | | | | |
| Chemical ...... „ | 33,474 | 15,916 | 172,839 | 102,121 |
| Mechanical .... „ | | 18,198 | | 52,052 |
| Rosin............ Cwt. | 56,403 | 68,014 | 14,240 | 15,060 |
| Tallow and stearin „ | 106,543 | 233,754 | 102,180 | 239,834 |
| Tar ..............Barrels | 3,093 | 17,145 | 2,163 | 14,152 |
| Wood:— | | | | |
| Hewn ......... Loads | 301,939 | 270,910 | 525,603 | 533,897 |
| Sawn .......... „ | 924,127 | 946,701 | 2,139,004 | 2,138,870 |
| Staves......... „ | 15,924 | 15,428 | 49,037 | 53,268 |
| Mahogany ..... Tons | 7,921 | 9,154 | 67,898 | 86,911 |
| Other articles....Value £ | .. | .. | 1,190,026 | 1,052,859 |
| Total value ....... | .. | .. | 5,241,185 | 5,445,353 |

Besides the above, drugs to the value of 68,486*l*. were imported, as against 79,459*l*. in October 1897.

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 31ST OCTOBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 8,682 | 9,084 | 40,714 | 41,074 |
| Copper:— | | | | |
| Unwrought.... „ | 44,559 | 52,564 | 115,576 | 139,590 |
| Wrought ...... „ | 31,583 | 27,984 | 95,371 | 88,683 |
| Mixed metal... „ | 26,060 | 18,246 | 61,466 | 45,463 |
| Cutlery .........Value £ | .. | .. | 189,576 | 173,443 |
| Hardware ....... „ | | | | |
| Implements ..... „ | .. | .. | 115,587 | 115,637 |
| Iron and steel ... Tons | 340,915 | 261,340 | 2,161,693 | 1,837,328 |
| Lead ............ „ | 3,521 | 4,323 | 49,524 | 58,636 |
| Plated wares ... Value £ | .. | .. | 47,328 | 48,496 |
| Telegraph wires . „ | .. | .. | 47,746 | 89,313 |
| Tin ............. Cwt. | 9 243 | 8,342 | 29,976 | 32,183 |
| Zinc............. „ | 13,721 | 14,336 | 10,293 | 13,431 |
| Other articles .. Value £ | .. | .. | 88,720 | 92,130 |
| Total value ....... | .. | .. | 3,053,570 | 2,775,407 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 31ST OCTOBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali........... Cwt. | 483,891 | 375,239 | 120,487 | 102,657 |
| Bleaching materials „ | 126,310 | 103,198 | 39,653 | 28,069 |
| Chemical manures Tons | 33,431 | 82,154 | 145,828 | 169,306 |
| Copper sulphate .. „ | 1,070 | 576 | 18,338 | 9,523 |
| Medicines....... Value £ | .. | .. | 106,436 | 100,481 |
| Other articles... „ | .. | .. | 277,954 | 263,644 |
| Total value ......... | .. | .. | 708,696 | 673,680 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 31ST OCTOBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 556,900 | 517,400 | 14,022 | 10,850 |
| Military stores.. Value £ | .. | .. | 115,754 | 146,718 |
| Candles.......... Lb. | 2,138,100 | 2,052,900 | 27,931 | 28,535 |
| Caoutchouc ..... Value £ | .. | .. | 116,959 | 118,975 |
| Cement.......... Tons | 35,512 | 31,432 | 59,440 | 62,197 |
| Products of coal Value £ | .. | .. | 143,139 | 117,835 |
| Earthenware ... „ | .. | .. | 149,759 | 145,556 |
| Stoneware ...... „ | .. | .. | 16,372 | 14,926 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 146,243 | 186,487 | 8,542 | 10,852 |
| Flint........... Cwt. | 9,004 | 9,077 | 23,028 | 20,477 |
| Bottles......... „ | 80,445 | 64,314 | 37,979 | 30,942 |
| Other kinds.... „ | 21,803 | 19,490 | 16,801 | 16,414 |
| Leather:— | | | | |
| Unwrought .... „ | 16,451 | 13,098 | 138,077 | 113,320 |
| Wrought ..... Value £ | .. | .. | 39,390 | 43,156 |
| Seed oil.......... Tons | 4,287 | 3,287 | 73,866 | 59,421 |
| Floorcloth ..... Sq. Yds. | 1,743,700 | 1,684,100 | 75,839 | 72,700 |
| Painters' materials Val. £ | .. | .. | 136,841 | 141,551 |
| Paper ........... Cwt. | 90,189 | 72,868 | 132,511 | 111,278 |
| Rags............ Tons | 5,718 | 4 290 | 28,199 | 20,675 |
| Soap............. Cwt. | 63,295 | 60,710 | 67,253 | 71,622 |
| Total value .......... | .. | .. | 2,909,626 | 2,802,223 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

2896A. E. H. Hurry and H. J. Seaman. Improvements in process and apparatus for using pulverised fuel and in rotary roasting furnaces for use therewith. Complete Specification. Filed Oct. 26. Date claimed Feb. 4, 1898.

21,787. R. Beese and A. Perlich. Contrivance for raising liquids. Complete Specification. Oct. 17.

21,797. M. Arndt. Improvements in apparatus for measuring and indicating the pressure of gas. Oct. 17.

21,935. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. Improvements in closing devices for vessels containing volatile liquids. Oct. 18.

21,951. S. E. Ericson. Improved valve-cock. Filed Oct. 18. Date applied for March 22, 1898, being date of application in Sweden.

22,185. R. Bodmer and J, Schwarzenbach. Improvements in apparatus for the separation of liquids from fibrous materials. Complete Specification. Oct. 21.

22,204. W. L. Wise.—From A. Kitson, United States. Improvements in and relating to vapour-burning apparatus. Oct. 21.

22,221. C. W. R. Goeresch and P. J. Eisenstuck. Improvements in glory-hole furnaces. Complete Specification. Oct. 22.

22,279. F. G. Maardt. Improvements in continuously operating kilns. Oct. 22.

22,437. J. S. V. Bickford. Improvements in apparatus for effecting combustion of liquid fuel for furnaces and the like. Oct. 25.

22,819. A. S. Plews and J. W. Worsey. Improvemeans for agitating mineral ore matte or other heavy matter in a liquid. Oct. 31.

22,8[illegible]. A. J. Newton and W. H. Cocks. Improvements in apparatus for separating or removing sediment from liquid and for similar purposes. Oct. 31.

23,176. J. Foster. *See* Class IX.

23,31[illegible]. L. S. Bond. Improvements in means for carrying and storing carbide of calcium. Nov. 7.

23,490. H. W. Miller and W. Turvey. An improved furnace. Nov. 7.

23,50[illegible]. J. Presley. Improvements in digesting apparatus. Nov. 8.

23,6[illegible]8. M. [illegible]en. Apparatus for mixing difficultly miscible liquids or liquefied matters. Nov. 9.

23,687. A. James. Improved apparatus for the distribution of solutions and for the separation of clear solutions from precipitated matter. Complete Specification. Nov. 10.

23,688. C. Martini. A process of and apparatus for drying viscous and like substances. Complete Specification. Nov. 10.

23,731. G. Cockburn. Improvements in jointing or packing tubes for surface condensers, evaporators, distilling apparatus, and the like. Complete Specification. Nov. 11.

23,763. F. L. Gibbs. Improvements in the manufacture of glass-lined metallic vessels. Nov. 11.

Complete Specifications Accepted.*

1897.

25,265. G. Helps. Regenerative furnaces for the heating of gas retorts and the like. Nov. 9.

25,634. E. Theisen. Process and apparatus for absorbing, extracting, distilling, or evaporating liquids and gases or vapours. Nov. 16.

25,793. C. Delecroix. Hamper for bottles containing acids, essences, and the like. Nov. 9.

26,082. G. Watson. Furnaces for the destruction of town refuse. Nov. 19.

26,454. H. A. House, jun., and R. R. Symon. Apparatus for regulating the supply of combustible liquid or vapour to liquid fuel burners. Oct. 26.

27,061. J. P. Thornton and J. P. Len. Generators, and apparatus in connection therewith. Nov. 9.

29,500. E. F. Murphy. Apparatus for carbonating water and other liquids. Oct. 21.

1898.

1496. C. H. Cribb. Apparatus for the condensation of vapours for distilling and like purposes. Oct. 26.

2274. W. Nelson. Apparatus to be used in refrigerating or freezing purposes. Nov. 2.

2464. A. F. J. Durafort. Apparatus for containing compressed carbonic acid, and charging water or other liquid therewith. Nov. 2.

5682. A. [illegible]. Apparatus for heating or cooling liquids, gases, and the like. Nov. 16.

11,135. W. F. Berner. Means for heating furnaces. Nov. 16.

16,672. A. Lecomte and J. Loeser. Apparatus for regulating the pressure of and distributing gas. Nov. 16.

17,206. J. Metzger. Filtering presses. Nov. 9.

19,105. J. Zebra. Apparatus for the production of ice. Oct. 26.

## II.—FUEL, GAS, AND LIGHT.

Applications.

21,851. H. C. Jenkins and T. C. Lowden. Improvements in or relating to the manufacture of "producer gas," and apparatus therefor. Complete Specification. Oct. 17.

22,020. H. H. Lake.—From F. L. Slocum, United States. Improvements relating to the manufacture of gas and to apparatus therefor. Oct. 19.

22,110. J. Schneeweis. An improved acetylene generator. Complete Specification. Oct. 20.

22,272. A. D. Williamson. Improvements in or relating to means for generating, purifying, storing, and distributing acetylene gas. Oct. 22.

22,330. G. Dollner. An improved process for purifying acetylene, and apparatus for same. Oct. 24.

22,437. J. S. V. Bickford. *See* Class I.

22,572. W. Ireland. Improvements in acetylene gas generators, and in the means of charging them. Oct. 27.

22,734. W. J. A. Dossetter. Improvements in or relating to acetylene gas generators and the like. Oct. 29.

22,784. J. B. Lafond. Improvements in or relating to the manufacture of water-gas. Complete Specification. Oct. 29.

22,807. H. H. Lake.—H. G. Semadeni, Switzerland. Improvements in incandescent gas mantles. Oct. 29.

22,826. J. W. Bailey and J. Clapham. Improvements in apparatus for generating acetylene. Oct. 31.

22,922. J. E. Smith. Improvements in apparatus for the generation and storage of acetylene gas. Nov. 1.

22,982. B. Acres. Apparatus for temporarily increasing the pressure of illuminating gas. Nov. 1.

22,985. G. Coming, E. Coming, and the Briquette Coal Company. Improvements in artificial fuel. Complete Specification. Nov. 1.

23,079. E. Disdier-y-Crooke. Improvements in or relating to the utilisation of the gases issuing from blast furnaces and from coke ovens. Nov. 2.

23,126. H. Niewerth. Means and method for heating air or gases. Nov. 3.

23,142. H. J. Dowsing. Improvements in apparatus for producing and using radiant heat. Nov. 3.

23,230. G. A. Gilmer. Improvements in or relating to apparatus for generating acetylene gas. Nov. 4.

23,255. J. G. L. Bormann. Improvements in and relating to gas producers. Nov. 4.

23,287. W. E. Patterson. Improvements in generators for the manufacture of acetylene gas. Nov. 5.

23,324. J. South. *See* Class XII.

23,363. H. Kinsey. Improvements in apparatus for generating and storing acetylene gas. Nov. 7.

23,369. R. Beese and A. Perlich. Improvements in gas burners with self-igniting Auer filament. Complete Specification. Nov. 7.

23,370. O. Brünler. Improvements in burners with vaporiser for liquid fuel. Complete Specification. Nov. 7.

23,401. A. Clerk. Improvements in and relating to incandescent lighting and heating. Nov. 7.

23,576. F. W. Smith. Improvements relating to apparatus employed in the generating of acetylene gas. Nov. 9.

23,675. H. de Thiersant and W. A. Coulson. Improvements in or relating to generators for acetylene gas. Nov. 10.

23,796. N. Ostert. Improvements in coke ovens. Nov. 11.

Complete Specifications Accepted.

1897.

18,128. F. Goulding. Apparatus employed in the production of acetylene gas.

24,328. J. Wilson. Manufacture of burners for incandescent gas lighting. Nov. 2.

25,002. L. de Ravel. Apparatus for the production of acetylene gas. Nov. 2.

26,269. G. E. Arkell, J. W. Bailey, and J. Clapham. Apparatus for use in the production of acetylene. Nov. 9.

26,776. J. Kremer. Acetylene gas generators. Nov. 2.

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* See Note (*) on previous page.

26,843. E. S. Bond. Means for storing and using calcium carbide or the like for acetylene and the like gas-generating apparatus. Oct. 26.

27,016. A. C. Fraser. Methods of and apparatus for burning acetylene and other rich gases. Nov. 9.

27,288. J. Reibel. Apparatus for generating acetylene gas. Oct. 26.

28,496. H. Gardner. Manufacture of artificial fuel and fire lighters. Nov. 2.

28,526. J. R. Wigham. Method of increasing the illuminating power of gas made from coal or other material. Nov. 2.

28,706. J. Moeller. Incandescence mantles. Nov. 9.

28,824. H. Bryant. Apparatus for making illuminating gas. Oct. 26.

29,096. J. A. Drake and J. S. Marsland. Inclined gas retorts. Nov. 9.

29,201. E. V. Prevost, O. Raus, A. Cerkel. Apparatus for the production of acetylene. Nov. 2.

29,767. W. B. Smith. Mantles to be heated by gas. Nov. 16.

29,828. E. Jürgens and J. F. C. Jürgens. Manufacture of igniting pills for illuminating gas. Oct. 26.

30,324. J. C. H. Kramers and A. E. Kiderlen. Appatus for enriching generator gas. Nov. 16.

30,585. J. McConechy. Apparatus for the production and storage of acetylene gas. Nov. 2.

30,833. E. E. Yvonneau. *See* Class VII.

1898.

2863. J. S. Ferguson. Acetylene gas generator. Oct. 26.

8382. H. H. Lake.—From J. Grand, France. Apparatus for the production of acetylene gas. Oct. 26.

8439. J. Stott. Apparatus for generating and storing acetylene gas. Nov. 9.

12,491. A. J. Boult.—From Société Italiana Pel Carburo di Calcio Acetilene ed altrigas, Italy. Acetylene gas generators. Nov. 9.

14,438. S. J. Ingram. Apparatus for charging coal-gas and other retorts. Nov. 16.

17,516. J. Fielding. Apparatus for producing and carburetting hydrogen. Oct. 26.

17,612. E. W. Gillett and C. R. Gillett. Acetylene gas generators. Oct. 26.

18,063. K. G. Gustafson. Acetylene gas apparatus. Nov. 2.

18,220. E. Engasser. Apparatus for generating acetylene gas. Nov. 9.

18,591. R. Quantannens-Moens and E. Carreer-Dilger. Production of acetylene gas. Nov. 2.

18,943. R. Söderquist. Apparatus for the production of acetylene gas. Oct. 26.

19,369. J. Boss. Apparatus for generating and utilising gas, such as acetylene. Nov. 2.

20,273. A. J. Boult.—From E. Verbeke, Belgium. Bunsen burners. Nov. 9.

20,646. R. Abeles. Carbide containers, and containers for acetylene gas apparatus. Nov. 9.

## III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

### Application.

28,653. A. Gordon. Retorts for the distillation of shale and other substances. Nov. 2.

### Complete Specification Accepted.

1898.

20,086. J. Armour and F. Alison. Method of and apparatus for heating paraffin scale. Oct. 26.

## IV.—COLOURING MATTERS and DYES.

### Applications.

21,832. A. G. Green, A. Meyenberg, and The Clayton Aniline Company, Ltd. Improvements in the manufacture and production of colouring matters containing sulphur. Oct. 17.

21,839. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of dyestuffs of the diphenylnaphthylmethane series. Oct. 17.

22,106. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of amido-acylamido-carbonic acid esters. Oct. 20.

22,459. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in or connected with the manufacture of products and colouring matters related to the indigo group. Oct. 25.

22,460. A. G. Green, A. Meyenberg, and The Clayton Aniline Company, Ltd. Improvements in the manufacture and production of colouring matters containing sulphur. Oct. 25.

22,765. W. McCowan. Improvement in the manufacture of colouring matter. Oct. 29.

22,847. A. G. Green, A. Meyenberg, and The Clayton Aniline Company, Ltd. Improvements in the manufacture and production of colouring matters. Oct. 31.

23,435. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of basic phenol ethers. Nov. 7.

28,615. H. H. Lake.—From Farbwerke Mühlheim, formerly A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. Nov. 9.

23,644. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of oxyanthraquinones and oxyanthraquinone-sulphonic acids. Nov. 9.

23,712. D. C. Bendix. Improvements in the manufacture of dry alizarine colours. Nov. 10.

23,893. G. W. Johnson.—From Kalle and Co., Germany. The manufacture of new polyazo dyestuffs. Nov. 12.

### Complete Specifications Accepted.

1897.

22,121. G. G. Ellis.—From La Société Chimique des Usines de Rhône anciennement G. P. Monnet et Cartier. Production of aromatic aldehydes. Nov. 2.

28,107. S. Pitt.—From L. Cassella and Co. Manufacture of azo dyestuffs and in the production of fast dyeings on animal fibre. Nov. 2.

28,707. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning. Manufacture of *m*-oxybenzaldehyde-*o*-sulphonic acid. Oct. 26.

29,631. J. Imray.—From Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, France. Manufacture of green and blue-green colouring matters derived from triphenylmethane. Nov. 16.

30,118. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of benzaldehyde, *o*-nitrobenzaldehyde, and *p*-nitrobenzaldehyde. Nov. 16.

30,446. O. Imray.—From The Farbewerke vormals Meister, Lucius und Brüning, Germany. *See* Class VI.

1898.

676. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of new colouring matters. Nov. 16.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

22,101. W. E. Aykroyd, W. H. Aykroyd, and H. E. Aykroyd. Improvements in or relating to apparatus for the mercerisation of cotton or other fibrous materials. Oct. 20.

22,104. M. G. Olivier. An improved product and process for rendering stuffs, tissues, paper, feathers, furs, and the like impermeable. Filed Oct. 20. Date applied for April 9, 1898, being date of application in France.

22,231. J. Ashton and E. C. Käyser. An improved method and means for producing permanent gloss on cotton and other vegetable fibres and fabrics. Oct. 22.

22,244. W. P. Thompson.—From E. Claviez, Germany. *See* Class XIX.

22,439. G. H. Nussey and W. B. Leachman. A new or improved method or process of pressing and finishing cloth and other textile fabrics. Oct. 25.

22,646. H. Skelland and F. Craven. Improvements in apparatus for clearing yarns and also for testing yarns. Oct. 28.

22,813. H. Magnac and C. Newman. Novel material for clothing and other purposes. Oct. 31.

23,016. J. Ashton and E. C. Käyser. An improved method and means for producing permanent gloss on cotton and other vegetable fabrics. Nov. 2.

23,128. E. Hantsch. Improvements in treating cotton yarn, and apparatus therefor. Complete Specification. Nov. 7.

Complete Specifications Accepted.

1897.

21,468. G. Ormondroyd. Method of and apparatus for scouring hanks of yarn. Nov. 2.

27,020. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Process for mercerising vegetable fibre. Oct. 26.

28,491. C. Wetherwax. Treatment of vegetable fibres. Nov. 2.

29,613. A. Wyser. Apparatus for mercerising yarn. Oct. 26.

1898.

3100. H. R. Romney and J. Thame. Treating waste cocoa-nut dust, fibre, or refuse. Nov. 9.

18,217. E. Maertens. Process and apparatus for treating of wool and similar fibres by means of disolvents. Nov. 16.

19,177. H. Kirssmanck and F. Anderieth. Apparatus for and in connection with the mercerising of yarns. Nov. 9.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

Applications.

21,833. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in mordanting cotton or other vegetable fibre. Oct. 17.

23,656. H. C. Longsdon. Improvements in machinery or mechanism for washing or cleansing, dyeing, and drying clothes, fabrics, and the like. Nov. 10.

23,710. C. D. Abel.—From Firm of H. C. Meyer, jun., Germany. Process for dyeing and bleaching cane. Nov. 10.

23,827. G. D. Burton. *See* Class XI.

Complete Specifications Accepted.

1897.

25,564. W. H. Thorpe. Machinery for dyeing, scouring, and bleaching hanks or skeins of yarn. Oct. 26.

26,5[illegible]8. B. C. Marot and A. Bonnet. Process for mordanting cotton for aniline black. Oct. 26.

29,317. H. Haag, C. Rotter, and M. Rotter. Process for dyeing. Nov. 9.

30,446. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Production of fast brown to brown-black tints on the fibre by combining naphthol with the diazo compound of diamidocarbazol. Nov. 9.

## VII.—ACIDS, ALKALIS, and SALTS.

Applications.

22,192. W. R. Ormandy. Improvements in the treatment of kaolin, sulphate of soda, sand, and other substances containing iron, and in obtaining certain compounds of iron, and apparatus therefor. Oct. 21.

22,546. W. P. Thompson.—From G. Wischin, Russia. Improvements in or relating to the manufacture of sulphuric anhydride or sulphuric acid, and in apparatus therefor. Oct. 26.

22,868. C. Uebel and Chemische Fabrik Rhenania. Improvements in the manufacture of high-grade volatile acids, in particular nitric acid and acetic acid, and of compounds to be used therefor. Complete Specification. Oct. 31.

23,081. D. Williams. Improvements in the manufacture of sulphuric acid free from arsenic. Nov. 3.

23,876. S. Douglas. An improved process for obtaining purified silicic acid and fluosilicic acid from the residual mixture obtained in the purification of graphite by means of dilute fluoric acid. Complete Specification. Nov. 12.

23,894. E. G. Scott. Improvements in the manufacture of sulphuric acid, and in apparatus therefor. Nov. 12.

Complete Specifications Accepted.

1897.

21,178. J. Hargreaves. Treatment of solutions of carbonate or soda resulting from electrolysis of chloride of sodium, and in apparatus applicable for use therein. Oct. 26.

26,172. Method or process of preparing oxalic acid and oxalates. Nov. 9.

29,008. J. Bastick and W. A. Thornton. Manufacture of calcium carbide. Nov. 9.

30,833. C. E. Yvonneau. Treatment of calcium carburet. Nov. 10.

1898.

223. E. G. Scott. Manufacture of acetic acid. Nov. 2.

12,852. W. van den Berg and T. Haas. Process for the manufacture of water- and acid-proof non-conducting articles of clay. Oct. 26.

16,978. I. Care. Apparatus or plant for the manufacture of sulphate of ammonia from ammoniacal liquors. Nov. 16.

18,110. W. L. Wise.—From The International Patent Company, United States. Manufacture of hydrofuge metallic carbides. Nov. 16.

## VIII.—GLASS, POTTERY, and ENAMELS.

Applications.

21,934. F. Ohm. Improvements in the manufacture of porcelain. Oct. 18.

23,025. S. Gibson. Improved method or process of preparing the surface of earthenware or ceramic articles for receiving decoration. Nov. 2.

23,724. F. Mountford and The Corporate Body trading as B. Dellagana and Co., Ltd. Improved means and their manufacture for effecting the coloured decoration of pottery. Nov. 10.

Complete Specifications Accepted.

1897.

26,384. F. Hancock and C. B. Winzer. Kilns or muffles used for pottery and brick purposes. Nov. 2.

27,811. W. Owen. Kilns for calcining or fritting materials used in the production of potter's glazes. Nov. 9.

30,571. W. P. Kelly. Improvement in or in connection with articles of china, Delph glass, and such like. Nov. 16.

30,835. P. T. Sievert. Mode of and apparatus for producing slabs or sheets of glass with inlaid wire. Nov. 9.

1898.

16,550. A. J. A. Berthelier. Furnaces for baking enamels, glassware, and porcelain. Nov. 9.

17,141. Cloisonné work and mosaics. Nov. 9.

19,436. C. C. Hartung. Machines for rolling wire glass. Oct. 26.

19,437. E. Walsh, jun. Machine for and method of rolling wired glass. Oct. 26.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### Applications.

22,059. F. P. Jones. An improved method of utilising waste or other heat for drying bricks, clayware, clay, shale, cement, timber, or other wet products or manufactures. Oct. 20.

22,197. P. Davies. Improvements relating to furnaces or kilns applicable for use in the production of Portland cement and other similar materials. Oct. 21.

22,242. G. A. Keifer. Improvements in the manufacture of artificial stone. Oct. 22.

22,319. R. Y. Foley. Improvements in or relating to tiles for building purposes. Oct. 24.

22,480. J. Webster, J. T. Woodhouse, and H. F. Worrall. An improved brick for building purposes. Oct. 26.

22,882. A. J. Boult.—From P. A. J. Gasse, France. Improvements in or relating to the manufacture of artificial stone, marble, and the like. Oct. 31.

23,159. A. H. van der Vijgh, H. van der Vijgh, and G. van der Vijgh. Improvements in the manufacture of artificial stone. Complete Specification. Nov. 3.

23,176. J. Foster. An improvement in and in connection with kilns for burning limestone, cement, and similar material. Nov. 4.

23,262. H. A. H. Moore. Patent Cornish stone. Nov. 5.

23,508. T. Potter. Improvements in the construction of fire-proof buildings. Nov. 8.

23,702. J. W. Pattison. Improved apparatus for treating asphalt. Complete Specification. Nov. 10.

### Complete Specifications Accepted.

1897.

28,938. H. V. Simpson. Treating wood for the purpose of rendering it non-inflammable or for preserving it. Oct. 26.

1898.

2003. L. A. Garchey. Manufacture of ceramic ware. Nov. 9.

17,158. W. Courtenay. Artificial stone, known as reconstructed granite, and process of making the same. Oct. 26.

18,302. G. H. Grey.—From J. W. Hayward, United States. Manufacture of artificial asphalt, and in apparatus to be employed therein. Nov. 2.

19,465. V. Kjeldsen. A furnace or kiln for calcining cement materials. Nov. 2.

## X.—METALLURGY, MINING, Etc.

### Applications.

21,865. S. O. Cowper-Coles. Improvements in the manufacture of zinc oxide. Oct. 18.

21,948. F. E. Elmore. Improvements in separating metallic from rocky constituents of ores, and apparatus therefor. Oct. 18.

21,949. A. S. Elmore. Improvements in separating the metallic from the rocky constituents of ores, and apparatus for that purpose. Oct. 18.

22,140. A. Jack. Improvements in apparatus for charging materials into open-hearth steel-melting furnaces. Oct. 21.

22,182. A. Reynolds. Improvements in blast and cupola furnaces. Oct. 21.

22,265. E. Raynaud. New or improved means for facilitating extraction of alumina from aluminous ores. Complete Specification. Filed Oct. 22. Date applied for March 25, 1898, being date of application in Belgium.

22,366. G. Rigg. A process for the production of oxide of zinc and carbonate of zinc from ores or other materials containing zinc. Oct. 25.

22,405. S. H. Thurston. Improvements in coating iron, steel, and other metals with copper. Complete Specification. Oct. 25.

22,421. E. D. Hopcroft. Improvements in the treatment of ore for the extraction of metals. Oct. 25.

22,483. P. F. Browne. New or improved solvent solutions for gold, copper, silver, and other metal elements. Oct. 26.

22,731. R. W. Western. Improvements in and relating to the smelting of refractory ores. Oct. 29.

22,871. A. M. Nicholas. An improved rotating filtering apparatus, principally applicable for the separation of gold- and silver-bearing solutions from tailings, slimes, and the like. Complete Specification. Oct. 31.

23,002. J. G. Slater. Improvements in and relating to aluminium. Complete Specification. Nov. 1.

23,022. J. Baxeres de Alzugaray. Improvements in extracting metals from their oxides and in forming alloys. Nov. 2.

23,068. R. H. Thwaite. Improved method of manufacturing armour plates. Nov. 2.

23,072. J. G. Slater. Improvements in and relating to the soldering of aluminium. Complete Specification. Nov. 2.

23,221. G. D. Bechi. Improvements in the treatment of complex ores. Nov. 4.

23,227. J. E. Sutphen. An improved amalgamator. Complete Specification. Nov. 4.

23,228. J. E. Sutphen. An improved method of amalgamating ores of gold and silver. Complete Specification. Nov. 4.

23,386. G. E. Bourgoin. A new fine alloy for hardening aluminium, increasing its strength and its elasticity. Nov. 7.

23,437. G. E. Bourgoin. Brazing or soldering of objects made of aluminium or of the like. Nov. 7.

23,697. J. C. Montgomerie. Improved means applicable for use in the extraction of metals from ores. Nov. 10.

23,795. B. H. Jamison. Improvements in the manufacture or treatment of steel or nickel steel, and of armour plates and other articles made from steel or nickel steel. Nov. 11.

23,902. G. E. Bourgoin. Improvements in and relating to soldering aluminium and its alloys. Nov. 12.

### Complete Specifications Accepted.

1898.

16,735. W. Feit. Treating lead containing precious metals. Nov. 16.

18,110. W. L. Wise.—From The International Patent Company, United States. *See* Class VII.

18,127. A. J. Rossi, J. MacNaughton, and W. D. Edmonds. Alloys of titanium, and processes for producing same. Nov. 2.

18,788. C. W. Sponsel and W. A. Lorenz. Process and apparatus for hardening metals. Nov. 16.

19,012. W. L. Wise.—From J. Burns, United States. Art of producing steel. Nov. 16.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### Applications.

21,835. W. J. Still. Improvements in storage batteries. Oct. 17.

21,974. S. O. Cowper-Coles. Improvements in the electro-deposition of metals. Oct. 19.

22,070. C. Levetus. Improvements in and relating to galvanic and other batteries. Oct. 20.

22,096. H. C. Bull, F. G. Bate, and B. A. Spaull. Improvements relating to electric storage batteries. Oct. 20.

22,115. E. Baresch, R. M. A. Baresch, E. M. Tosler, and W. von Zabern. Improvements in galvanic batteries. Oct. 20.

22,171. J. H. Graeber and H. Tobler. Improvements in insulating devices for the electrodes of accumulators. Complete Specification. Oct. 21.

22,461. J. Y. Johnson.—From H. J. Dercum, United States. Improvements in primary batteries. Complete Specification. Oct. 25.

22,872. J. Loiselet. Improvements in electrolytical apparatus. Oct. 31.

23,243. P. Ribbe. Improvements in and relating to electric accumulators. Complete Specification. Nov. 4.

23,249. C. H. Preston. An improved primary battery for generating electricity. Nov. 4.

23,306. C. Levetus. Improvements in or relating to galvanic batteries. Nov. 5.

23,307. C. Levetus. Improvements in or relating to galvanic batteries and the like. Nov. 5.

23,308. E. L. Levetus. Improvements in or relating to acetylene generators. Nov. 5.

23,481. J. Bastick. Improvements in or relating to electric furnaces for the production of calcium carbide. Nov. 8.

23,545. O. P. Nauhardt. Process for obtaining an electrolytic deposit of metallic films on aluminium. Complete Specification. Nov. 8.

23,597. J. W. Smith and W. Evans. Improvements in apparatus for the electro-deposition of metals. Nov. 9.

23,716. R. Haddan.—From G. J. Miller, United States. Improvements in or relating to secondary or storage batteries. Complete Specification. Nov. 10.

23,827. G. D. Burton. Improvements in the art of electric dyeing and shrinking, and in apparatus therefor. Complete Specification. Nov. 12.

23,853. S. F. Walker. Improvements in primary galvanic batteries. Nov. 12.

### Complete Specifications Accepted.

1897.

17,161. Means applicable for use in the electrolysis of salts. Nov. 2.

25,388. L. Parvillée. Compositions for use in the construction of electrical heating apparatus and resistances, and the processes for their manufacture. Nov. 9.

25,890. G. Bell and G. W. Bell. Electrolytic cells. Nov. 16.

26,680. A. Tribelhorn. Electric accumulators or storage batteries. Nov. 2.

27,683. W. H. Lane. Electrical storage batteries. Nov. 9.

27,707. J. E. Hartley and H. E. Hartley. Rotative electro-metallic depositing apparatus. Nov. 2.

28,460. C. Payen. Electrical accumulator. Nov. 16.

30,441. C. L. R. E. Menges. Primary and secondary galvanic batteries. Nov. 16.

1898.

932. C. Pollack. Manufacture or production of electrodes for secondary batteries. Nov. 16.

12,322. E. Edwards.—From P. A. Emanuel, United States. Electric batteries. Nov. 9.

13,861. W. F. Heys.—From E. Dumoulin, France. Electro-deposition of copper and other metals on rotary cathodes. Oct. 26.

16,[illegible]59. Dr T. Stillberg. Electrolytic process for extracting metals from their halogen combinations. Oct. 26.

17,588. A. J. Boult.—From A. Busson, France. Primary electric batteries. Oct. 26.

## XII.—FATS, OILS, AND SOAP.

### Applications.

22,3[illegible]1. B. T. Saunders, trading as B. C. Taylor. Improved combination of ingredients for the manufacture of soap (hard). Oct. 25.

22,778. W. J. Engledue and B. J. Yarnold. Improvements in the bleaching and refining of vegetable and animal fats and oils, fish oils, and waxes. Oct. 29.

23,139. M. Schaaf and E. Schaaf. An improved process for introducing more or less volatile substances into finished or unfinished soaps, and apparatus therefor. Complete Specification. Nov. 3.

23,324. J. South. Improvements in purifying hydrocarbon oils and rendering them non-explosive. Complete Specification. Nov. 5.

23,557. C. Gruene. An improved process of extracting fatty matters from wool. Complete Specification. Nov. 8.

23,806. D. Gray, jun. An improved soap compound. Nov. 11.

23,848. W. L. Gadd and W. Gadd. Improvements in bleaching soap. Nov. 12.

### Complete Specifications Accepted.

1897.

22,862. F. Arledter. Manufacture of resinous soap. Nov. 16.

29,728. S. Banner. Compound applicable for making a substitute for linseed-oil body for paint, printing-ink, floorcloth making, and the like. Nov. 9.

30,662. W. A. Smith. Process of refining oil. Nov. 2.

1898.

1153. E. G. Hughes. Manufacture and composition of soap for toilet and laundry purposes. Oct. 26.

18,730. C. G. Hepburn. Apparatus for treating and cooling heated liquid fats and oils with air. Nov. 2.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

21,830. Matthews' Lancashire, Cheshire, and North Wales District White Lead Company, Ltd., S. Pope, and G. V. Barton. Improvements in apparatus employed in the production of lead oxide and the treatment of rough litharge. Oct. 17.

22,029. C. O. Weber, C. F. Cross, and I. Frankenburg. Manufacture of cellulose esters. Oct. 19.

22,888. D. F. Lucas. Improvements in composition for coating or painting roofs, vessels, and the like. Complete Specification. Oct. 31.

22,891. P. Emanuel, H. M. T. Wächter, and H. Zugt. A new or improved blacking composition. Complete Specification. Oct. 31.

23,071. A. Stoop. A process for the manufacture of pigments and the like. Complete Specification. Nov. 2.

23,703. H. Davison. Improvements in or relating to the manufacture of white lead or the like. Nov. 10.

### Complete Specifications Accepted.

1897.

26,800. C. J. Grist. Manufacture of paints, anti-corrosive compositions, and compositions for like uses. Nov. 9.

1898.

119. Blundell, Spence, and Co., Ltd., and S. Hill. Varnish especially applicable for giving a frosted appearance to glass, talc, or the like. Nov. 16.

17,497. G. X. Dime. Fireproofing compositions. Nov. 2.

## XIV.—TANNING, LEATHER, GLUE, AND SIZE.

### Applications.

21,803. H. Wade.—From F. A. T. Moor, United States. Improvements in the manufacture of leather. Complete Specification. Oct. 17.

21,984. J. F. Jones. Improvements in tanning pits. Oct. 19.

22,318. J. F. Jones and E. S. Clegg. Improvements in apparatus for treating the tanning of hides and skins. Oct. 24.

22,443. J. Sprachocinski and C. Stoldt. Improved impermeable leather. Complete Specification. Oct. 25.

22,995. A. O. Eevenbye. A dressing composition for leather. Complete Specification. Nov. 1.

### Complete Specifications Accepted.

1897.

25,871. H. H. Lake.—From C. Dreher, Germany. Process for waterproofing leather and rendering the same impervious to aqueous solutions. Oct. 26.

1898.

6638. U. de Günzberg. Method of preserving and tawing skins. Nov. 2.

12,274. F. Grognet. Process for the preparation of skins. Oct. 26.

20,444. P. Emperauger and A. Huillard. Rotary barrel for use in rapid tanning. Nov. 9.

## XV.—AGRICULTURE AND MANURES.

### Application.

23,542. A. Wenck and E. R. Besemfelder. A process for obtaining the nitrogen of nitrogenous vegetable and organic waste and similar substances in the form of ammonia, and the treatment and utilisation of the resulting products. Complete Specification. Nov. 8.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Application.

22,788. G. B. Ellis. From F. Hornig, Germany. Improvements in the treatment of spent beetroot, and production of a useful body therefrom. Oct. 29.

### Complete Specifications Accepted.

1898.

19,021. A. J. Boult. From C. M. Higgins, United States. Adhesive compounds. Oct. 26.

20,900. W. P. Thompson. From K. Koch, Russia. Manufacture of cube or loaf sugar. Nov. 16.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

21,993. J. R. Garroway. Improvements in and relating to the treatment of distillers' waste liquor. Oct. 19.

22,058. J. H. Fullarton. Improvements in or connected with the disposal of distillery effluents. Oct. 20.

22,167. J. A. Brackman. A liquid for fermentation of beers, to be called "Brackman's liquid." Oct. 21.

22,208. J. Correnti and A. A. Amidei. A device for filtering, purifying, and sterilising the air before its entry into casks or other vessels containing fermented liquors, and for other like use. Oct. 21.

22,247. H. Finlay. Improvements in the manufacture of yeast or barm. Oct. 22.

22,355. P. Kropf. Process of manufacturing beer. Complete Specification. Oct. 24.

22,607. J. T. Hewitt. Improvements in the purification of alcoholic liquids. Oct. 27.

22,857. H. W. Joyce. Improved means for drying hops and other materials in kilns. Oct. 31.

29,892. C. Schwager. Improvements in malting apparatus. Complete Specification. Oct. 31.

23,260. T. C. Andrew. Improvements in utensils used for brewing. Nov. 5.

23,318. H. Finlay. Improvements in the manufacture of yeast or barm, and in apparatus connected therewith. Nov. 5.

23,402. G. F. Zimmer. Improvements in and connected with apparatus used in the manufacture of flaked maize malts and the like substances. Nov. 7.

23,482. E. A. Pettit. Improvements in gelatinisers for gelatinisation of grain for brewing and other purposes. Nov. 8.

23,571. J. Mitchell. Extracting the insoluble ingredients from the wash of distilleries, and the means employed therefor. Nov. 9.

### Complete Specifications Accepted.

1897.

29,157. H. M. Kettelwell. Method and apparatus for the prevention and extinction of fires in breweries, distilleries, and other like premises. Oct. 26.

1898.

4943. A. F. Watson. Process for recovering beer, ale, and porter from vat bottoms, vat sweepings, or vat deposit generally. Nov. 9.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

*A.—Foods.*

21,971. E. Frichot. An improved process for sterilising and blanching cereals and alimentary leguminous substances, and the meal produced therefrom. Oct. 18.

22,023. J. Wetter.—From the firm of R. d'Ambrières et Cie., France. Improvements in or connected with the preservation of milk. Oct. 19.

22,184. H. J. Dunn. A process for preserving animal food. Oct. 21.

22,989. W. H. Myers. Improved means for preserving eggs, and the employment of an ingredient for that purpose. Nov. 1.

23,440. J. B. A. Grouchy. Improved composition for dressing of fabrics of all kinds employed in connection with chemical, alimentary, and other products. Complete Specification. Nov. 7.

23,631. H. C. Plaut. Process and apparatus for removing dirt from milk. Complete Specification. Nov. 9.

23,637. M. Julien. Process and apparatus for treating butter for purposes of conservation and subsequent restoration to the fresh condition. Nov. 9.

*B.—Sanitation.*

21,777. W. J. Baker. Improvements in and relating to apparatus for softening and clarifying water. Oct. 17.

21,791. H. Ellison. Improvements in chemical drain testers. Oct. 17.

21,954. C. Wegener. Improvements in or relating to furnaces for burning refuse, and in treating the slag therefrom. Oct. 18.

22,000. F. W. Wentworth-Shields. Improvements in the construction of bacterial tanks and filters for the purification of sewage. Complete Specification. Oct. 19.

22,233. G. Heaton. Improvements in apparatus for use in the treatment of sewage and other waters requiring purification. Oct. 22.

22,520. H. Desrumaux. Improvements in apparatus for purifying water and other liquids. Oct. 26.

23,562. Wm. Ross Hutton, jun. Improvements in the utilisation of precipitated sewage sludge. Nov. 9.

*C.—Disinfectants.*

22,032. J. Long. Improved antiseptic lamp. Oct. 19.

23,057. A. Zimmermann.—From The Chemische Fabrik auf Actien vormals E. Schering, Germany. Improvements in disinfecting by means of formic aldehyde. Nov. 2.

### Complete Specifications Accepted.

*A.—Foods.*

1897.

27,617. W. Lanwer. Process for preserving meat and other alimentary substances. Nov. 9.

28,379. H. H. Lake.—From A. Moyet, France. Apparatus for sterilising meat in the raw state. Nov. 2.

30,842. C. Kobbe. Process for the manufacture of alimentary products containing vegetable albumen. Nov. 16.

1898.

16,195. A. Rehse. Preserving food. Oct. 26.

### *B.—Sanitation.*

1897.

23,913. J. Rudman and H. J. G. Rudman. Process for the effective removal of noxious and other vapours, and appliances for use in connection therewith. Oct. 26.

26,969. G. E. Gale. System of apparatus for treating garbage. Oct. 26.

27,026. H. McPhail. Heating, softening, and purifying water, and apparatus therefor. Nov. 2.

28,164. G. Krell and M. Elb. Disinfecting process by means of methylaldehyde or polymeric methylaldehydes, and method of employing same. Nov. 2.

### *C.—Disinfectants.*

1898.

3923. E. Copeland. Composition for preserving milk and its products. Nov. 16.

## XIX.—PAPER, PASTEBOARD, Etc.

### Applications.

22,244. W. P. Thompson.—From E. Claviez, Germany. An improved manufacture of yarn from paper, and apparatus therefor. Complete Specification. Oct. 22.

22,588. E. R. Richardson and W. Dickson. Improvement in the process of paper making. Oct. 27.

22,671. E. Bush. Improvements in or connected with machinery for varnishing and otherwise coating continuous paper or other material. Oct. 28.

### Complete Specifications Accepted.

1897.

30,337. T. Conn and Mardon, Son, and Hall, Ltd. Imitation leather, cloth, parchment, and the like. Nov. 16.

1898.

3633. F. A. Rouse. Blotting paper. Nov. 9.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

### Applications.

22,268. R. W. James.—From Société Française de Disinfection, France. An improved product for the sterilising of germs, micro-organisms, and the like. Complete Specification. Oct. 22.

23,254. A. Strebel. Manufacture of ionone from citral and acetone, and of cyclocitral as intermediate product. Nov. 4.

23,425. R. Fickeisen. Process for the manufacture of morphine compounds capable of being easily absorbed, like mekonate. Complete Specification. Nov. 7.

23,611. J. Wilson. A diluted or reduced standardised saccharin. Nov. 9.

### Complete Specifications Accepted.

1897.

28,467. O. Imray.—From La Société Fabriques de Produits Chimiques de Thann et de Mulhouse, Germany. Manufacture of artificial musk. Oct. 26.

29,061. S. Pitt.—From The Pharmaceutische Institut Ludwig W. Gans, Germany. Production of the operative substance from the thyroid glands and other organs in an unaltered uncoagulable state. Nov. 16.

[illegible]08. A. Goldberg, W. Supermann and H. Flemming. Process for the preparation of guanidine salts. Nov. 2.

## XXI.—PHOTOGRAPHY.

### Applications.

22,361. R. R. Beard. Improvements in photography. Oct. 25.

23,001. E. Hunt. Improvements in apparatus for photographing and reproducing moving or stationary objects without or with colours. Nov. 2.

23,277. E. Debenham. An improved method and apparatus for igniting flash powder as used in photography. Nov. 5.

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28,920. J. W. Bennetto. Production of coloured photographs, and apparatus for that purpose. Nov. 9.

1898.

18,799. W. Selke. Process and apparatus for the plastic representation of solid bodies by the aid of photography. Nov. 16.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

21,815. C. A. Seddon.—From C. Killing, Germany. Improvements in the means for igniting combustible or explosive gas mixtures. Oct. 17.

22,069. J. Williamson. Improvements in the manufacture of gunpowder and blasting powder, and in the treatment or preparation of carbonaceous materials therefor. Oct. 20.

22,269. R. C. Parsons and D. W. Nightingale. An improvement in explosives. Oct. 22.

22,317. J. B. Lee. Improvements in the composition and construction of match heads. Oct. 24.

22,398. H. S. Wilson and W. G. Wilson. Improvements in fuses and in apparatus for setting the same. Oct. 25.

22,114. J. T. O'Callaghan. A submarine explosive mine, and apparatus therefor. Oct. 25.

22,138. A. T. Dawson and J. Karstairs. An improvement in explosive charges for ordnance. Oct. 25.

22,525. A. Luck. An improvement in the manufacture of explosives. Oct. 26.

22,535. Sir W. G. Armstrong, Whitworth, and Co., Ltd., Sir A. Noble, and G. Stuart. Improvements in time fuses. Oct. 26.

22,955. S. L. Fog and A. G. Kirschner. An improved process for the manufacture of matches. Complete Specification. Nov. 1.

23,410. The Electric Fog Signal Syndicate, Ltd., and W. R. Sykes. Improvements in explosive signalling apparatus for railways. Nov. 7.

23,857. G. Hookham. Improvements in lucifer matches. Nov. 12.

### Complete Specifications Accepted.

1897.

26,896. C. A. McEvoy. Time and percussion fuses. Oct. 26.

30,112. H. Boyd. Explosives. Oct. 26.

30,546. C. Kastner and A. Swoboda. Manufacture of matches. Nov. 9.

1898.

18,570. W. P. Ferguson. Fulminate caps for dynamite cartridges. Nov. 15.

## PATENT UNCLASSIFIABLE.

### Complete Specification Accepted.

1897.

27,877. J. Rodgers. Petrification or incrustation of porous or other substances. Nov. 2.

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THE JOURNAL

OF THE

# Society of Chemical Industry.

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Dr. B. W. Gerland. "On the Lower Sulphonic Acid of Indigotine."
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### Collective Index.

In reply to numerous inquiries, the printing of the Collective Index is in active progress, the whole of the work being in the printers' hands. The labour of revision and correction must, however, of necessity, take time; and it is hoped that those members who have already subscribed will extend their indulgence to the Indexer and Committee who have the work of correction in hand.

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## Deaths.

Moore, Thos., Riversdale, Northwich. Nov. 11.
Obach, Dr. Eug. F., of Siemens Bros. and Co., Ltd., Woolwich, at Graz. Dec. 27.
Wylde, J. R., United Alkali Co., Ltd., St. Helens. Nov. 28.

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## Liverpool Section.

*Meeting held on Wednesday, November 30th, 1898.*

MR. ALFRED SMETHAM IN THE CHAIR.

### SOME EXPERIMENTS UPON HIGH-STRENGTH HYPOCHLORITE SOLUTIONS.

BY MAX MUSPRATT AND E. SHRAPNELL SMITH.

The hypochlorites are a class of bodies upon which comparatively little work has been done, though a large amount of work exists where their solutions have been used for reactions upon other, more especially organic, bodies. Their most important member—hypochlorite of lime—has also engaged a considerable degree of attention, but the need of more definite information upon the others, notably sodium hypochlorite, led to the experiments which we are now placing before this Section.

Reviewing the past work, we find in Watts' Dictionary a large assortment of methods of preparation and general reactions of hypochlorous acid and hypochlorites, including, of course, the standard method of passing chlorine gas into caustic soda solution; but, beyond the statement of the fact of their formation, nothing is to be found with regard to strength of solutions, decomposition at various strengths, or information which would place the hypochlorites upon the plane of well-known and fully understood bodies.

Subsequent work, as already stated, has tended more to study the uses to which hypochlorites may be put as oxidising agents, especially upon organic bodies, than to study their specific properties and strength; but that which has been done upon them may be briefly summarised.

An extract from the Bulletin de la Société Industrielle, of Rouen, given in this Journal, 1890, 611, points out that sodium hypochlorite has become a not unimportant commercial product, and reviews the methods of its manufacture. The earliest method depended upon the simple reaction already mentioned, but this was improved upon by Planeau in 1876, who decomposed bleaching powder with carbonate or sulphate of soda in solution with the formation of hypochlorite of soda solution. Under the name of "Eau de Javelle," this liquid had a considerable sale as a disinfectant upon the Continent.

This brief extract is really all that is to be found about hypochlorite of soda up to 1890.

In that year a patent was taken out by Count de Brochocki for what he asserted was a wonderful improvement upon "Eau de Javelle." We need not explain this patent, as it was both chemically and practically an absurdity, though there was no question as to its novelty. The next year, however, the same inventor took out a patent which, though scarcely practicable for commercial purposes, is, from a chemical standpoint, ideal. Under this patent, dry chlorine and air are subjected to an electrical discharge, the resultant gases being passed into caustic liquor. By this means the inventor claims to have enormously increased the energy of the chlorine, but certainly does not seem to understand the chemistry of the alteration that he has made, which has simply resulted in the conversion of chlorine gas into $Cl_2O$. The advantage of this method is best shown by the equations $Na_2O + Cl_2O = 2NaOCl$, that is, *two* available oxygen atoms for two of chlorine, instead of the usual reaction $Na_2O + Cl_2 = NaOCl + NaCl$, that is, *one* available oxygen atom for two of chlorine.

The application of an electrical discharge for "energizing" chlorine, however, scarcely comes into the range of practical politics.

In 1895, F. Oettel investigated the question of electrolytically produced hypochlorite, for which processes had sprung up. This work is full of interest; but his statement that 1·25 of available chlorine per 100 c.c. is the highest strength obtainable by this means, even if, as we have been informed from an at present private source, the strength is somewhat under-estimated, places his experiments outside the scope of this paper.

The same remark applies to other work which has been done dealing with hypochlorites under 1 per cent.

In the Zeits. für anorg. Chem. for 1897, however, a long series of investigations as made by T. Bhaduri are given, and these are the first systematic attempts to make and examine hypochlorite of soda solutions approaching the strength of those already on the market.

Bhaduri passed chlorine into caustic solutions containing from 1 to 20 grms. of $Na_2O$ per 100 c.c. The highest hypochlorite, however, which he made corresponded to only about 8 grms. of available chlorine per 100 c.c., so that he had a large excess of alkali in his solutions.

He noticed that even when pure caustic soda was used, the colour of the solution after formation of hypochlorite was purple; this he attributed to formation of permanganate, but did not pay further attention to the point.

He also investigated in great detail the decomposition of the hypochlorite on standing, and gives as his results that a hypochlorite of about 5 to 8 per cent. by volume decomposes rapidly, even in the dark, with formation of chlorate and a certain evolution of oxygen.

This paper did not come to our hands till most of our work was completed, and it will be seen that in our results considerable divergence from his occurs. To this we will return.

There is no further work, as far as we have been able to ascertain, upon this subject, and what we have mentioned is obviously far from being exhaustive. Now, hypochlorite of soda is becoming more and more a recognised commercial product, and for several years there have been different makes upon the market, chiefly under trade names. The usual strength was 5 to 8 per cent. by volume of available chlorine, and this was not exceeded until the United Alkali Co. came upon the market with a 12½ to 15 per cent. article. This has, as yet, not been approached by other makers; but the demand for a high-strength hypochlorite is increasing, and in order to obtain definite information upon such hypochlorites these investigations were undertaken.

In choosing a method for these experiments, which are purely laboratory researches, we reverted to the old textbook method of passing chlorine into caustic soda solution as the simplest to follow in every phase.

Simple as the process is, we must ask permission to recall it, as several apparent anomalies arise which are only comprehensible if the reactions are quite clear in the mind.

$$\begin{matrix}NaOH\\NaOH\end{matrix} + \left\{\begin{matrix}Cl\\Cl\end{matrix}\right. = \begin{matrix}NaOCl\\NaCl\end{matrix} + H_2O.$$

For every two atoms of free chlorine, one has become fixed as inert sodium chloride, the other has combined with oxygen to form hypochlorite; the one molecule of hypochlorite having, so to speak, absorbed the activity of the two atoms of chlorine. Quantitatively, 80 parts of caustic soda have combined with chlorine to produce 133 parts of solid and 18 parts of water.

The problem that we now set ourselves was to determine the highest strength of hypochlorite that could be obtained from caustic soda.

A preliminary trial demonstrated the impossibility of making hypochlorite direct from solid caustic, for the simple reason that absolutely no reaction takes place.

The first limiting factor, then, for a high-strength hypochlorite is determined by the highest strength of caustic liquor obtainable in the cold; this we may take as 50 grms. $Na_2O$ per 100 c.c., which, allowing for the increase in volume due to the reaction, would give a hypochlorite corresponding to 50 grms. available chlorine per 100 c.c. There is a very big difference between this and the highest strength known hitherto, and it was taken provisionally as the upper limit to be aimed at.

The second limiting factor is the extent to which 80 parts of caustic can be replaced by mixed hypochlorite and salt so that these latter shall remain in solution, and this consideration may be materially modified by the possibility of the sodium chloride being thrown out of solution, leaving the hypochlorite, which may be regarded as a highly soluble body, by itself.

Our problem, therefore, was the fixing of these points and the general properties at these points. The apparatus consisted merely of a fairly large bottle containing caustic liquor, into which dried chlorine gas was passed. The caustic was in the form of commercial caustic liquor, containing, in addition to sodium hydrate, small quantities of chloride, sulphate, and carbonate; this solution was diluted down to the required strength for each experiment.

Table I.

| Experiment. | Caustic Soda Solution used. |  | Hypochlorite. |  |  |  |  | Remarks. |
|---|---|---|---|---|---|---|---|---|
|  | ° Tw. | $Na_2O$. | Av. Cl. | Cl. as Chlorate. | Total Cl. | $Na_2O$. | ° Tw. |  |
| A | 60 | 28·5 | 22·2 | .. | .. | 1·8 | 60 | Free alkali all carbonate. |
| B | 65 | 30·4 | 24·1 | 0·6 | .. | 0·7 | 66 |  |
| C | 70 | 31·7 | 25·8 | 0·1 | 20·9 | 2·0 | 70 |  |
| D | 72 | 33·8 | 28·6 | 2·4 | 23·3 | 0·4 | 62 | 18·6 Av. Cl. after 4 weeks. |
| E | 89 | 43·6 | 31·1 | 6·6 | 22·0 | 1·7 | 70 | 24·0 „ 3 „<br>9·1 „ 16 „ |
| F | 98 | 49·8 | 31·5 | 9·3 | 21·9 | 5·9 | 70 |  |
| H (1) | 70 | 32·9 | 28·2 | 0·6 | .. | 0·7 | 63 |  |
| H (2) | 70 | 7·4 | 26·2<br>33·2 | 0·6<br>0·9 | 18·0<br>.. | 0·5 | 68 |  |
| H (3) | 73 | 6·5 | 32·3<br>35·5 | 0·9<br>0·9 | 20·3<br>.. | 1·7 | 73 |  |
| H (4) | 78 | 8·4 | 32·8<br>39·8 | 2·8<br>2·8 | 18·5<br>.. | 0·6 | 75 |  |
| H (5) | 84 | 11·5 | 37·8<br>44·6 | 2·8<br>4·0 | 20·0<br>.. | 1·4 | 80 |  |
| H (6) | 85 | 6·2 | 43·0<br>49·2 | 4·0<br>9·4 | 23·1<br>27·6 | 0·5 | 84 | 28·6 Av. Cl. after 2 weeks.<br>5·8 „ 14 „ |
| I (final) | .. | .. | 44·5 | 2·9 | 20·6 | 7·8 | 84 | 18·4 „ 6 „ |
| K (1) | 70 | 29·8 | 26·6 | 1·1 | 21·0 | 0·9 | 63 |  |
| K (2) | 71 | 7·3 | 32·5 | 0·7 | 22·2 | 0·7 | 66 |  |
| K (3) | 71 | 4·3 | 34·0 | 0·4 | 22·0 | 1·0 | 68 | 33·7 Av. Cl. after 1 week. |

All results are in grammes per 100 c.c. of the solution, which for shortness we shall call "per cent."

The earlier experiments offered no difficulties, and hypochlorites of 12 to 15 per cent. were obtained, the fundamental conditions to be observed being a continuous excess of free alkali, and further a temperature below 90° F.; with these precautions the formation of chlorate could be entirely avoided.

The change of colour noted by Bhaduri also occurred with us, and in our opinion this trifling fact is of such great importance in its influence upon the hypochlorites that we will deal more fully with it in the course of our paper.

Proceeding now to higher strengths (see Table I.), the deposition of salt, as already anticipated, set in. The point at which this commenced varied with the strength of the solution and the progress of the absorption, but for the conditions under which the bulk of our experiments were carried out—namely, with a 70° Tw. caustic liquor at normal temperature—was when the solution had absorbed chlorine to the extent of 12 per cent. by volume. The deposit at this stage was not very large, and settled rapidly at the bottom, leaving the hypochlorite solution intact; but as we proceeded further to higher solutions the deposition was so copious that serious experimental difficulties arose, which made direct formation impossible. Two of our earlier experiments were as follows (A and B in Table I.):—

A solution of 60° Tw. yielded 22·2 % of available chlorine.
„ 65° „ 24·1 % „

But in these experiments, though an excess of alkali was left in at the finish, rapid decomposition took place. It was found on investigation that the free alkali had been entirely carbonated. This we had not realised as a disadvantage, but it was proved by special experiments that carbonate did not act as free alkali in maintaining the stability of hypochlorites; these special experiments are given in Table II.

Several changes in the solution at and just before complete saturation were interesting. The first sign was an alteration in the colour, the purple or dark red colour, which the solution had, becoming fainter; this reached its culmination in the throwing down of iron hydroxide, leaving a clear golden liquid. This liquid became rapidly hot, and a considerable decomposition into chlorate had taken place, the strengths, which at the moment of saturation had been 22·2 per cent. and 24·1 per cent. available chlorine, having on the following day fallen to 14 per cent. and 16 per cent. Our source of chlorine had not been unimpeachable as regards carbonic acid, but after remedying this defect and regarding only caustic alkali as free alkali, we were able to proceed.

Table I.

*Experiments D, E, F.*

Solution at 72° Tw. gave...... 28·6 % available chlorine.
„ 89° Tw. „ ...... 31·1 % „
„ 98° Tw. „ ...... 31·5 % „

But the two last contained, respectively, 6·6 per cent. and 9·3 per cent. Cl. as chlorate.

This would seem to indicate the attainment of a natural limit for the formation of hypochlorite, but as several occurrences had been noticed which might have had a disturbing influence, we sought to eliminate these before drawing too rapid conclusions. Thus in D the excess of alkali was extremely low, while in E and F the deposit of salt, owing to the high gravity of the solutions, was so heavy and settled so badly that the circulation of the liquor by the gas was greatly impeded; this could easily lead to local over-saturation and overheating, both of which might lead to abnormal results.

That the concentration had fallen to 70° Tw. in both cases indicated some limiting conditions of concentration which would not be ignored.

Now it had been noticed that, if a 70° Tw. caustic is treated with chlorine, its end concentration, owing to expulsion of salt, is several degrees lower than at the start. There was therefore a probability that the solution would be capable of dissolving up solid caustic, which could then be saturated with chlorine. Experiments based upon this assumption proved thoroughly satisfactory.

A 70° Tw. caustic solution, on saturation with chlorine, yielded a solution (Table I., H (1)) of only 63° Tw. with 28·2 per cent. available chlorine. Caustic soda was next dissolved up to the extent of 7·4 per cent. $Na_2O$; this led to further deposition of salt, the liquor was decanted off and the caustic saturated with chlorine.

This was repeated five times, the full results being given on Table I., Experiment H., the top line in each operation representing the state of the solution after addition of caustic and decantation, the second line being the state at the end of the chlorination.

The final solution was as follows:—

Tw. 84°, Av. Cl. 49·2 per cent., Cl. as chlorate 9·4 per cent. Total chlorine 27·6 per cent., with a large excess of alkali.

A second operation resulted in 44·5 per cent. Av. Cl. with 3 per cent. of chlorine as chlorate; but no special features belong to this experiment, which was merely confirmatory of the previous one.

The large quantity of chlorate was, however, unsatisfactory, and we still believed that it was an incidental rather than an essential of the operation. We were supported in this belief by a consideration of the following points in the process: up to 33 per cent. the chlorine as chlorate had been kept below 1 per cent.; the next operation saw it rise to 2·8 per cent., but here we were conscious that the temperature had been allowed to rise too high. In the further operations the increase was rapid, but during these the gravity of the solution had so risen that the salt, instead of depositing, rose as a foam-like scum to the top of the liquid. And this scum would carry up liquor which would then be subject to either evaporation or carbonation, with resultant conversion of hypochlorite into chlorate. On stirring, this found its way back into the solution, which consequently showed a high percentage of chlorate.

It would have been possible to repeat these experiments in an improved form, but, having obtained a practically 50 per cent. solution, we considered that we had sufficient information upon this phase of the subject, and preferred to investigate the speed of decomposition of these various solutions.

Before leaving this first stage of our work we will summarise the conditions for the formation of high-strength solutions of hypochlorite of soda:—

(1.) Always have an excess of alkali present;

(2.) Keep the temperature as low as possible—say not above 80° F.;

(3.) Commence at a gravity at which the salt thrown out of solution deposits readily;

(4.) Avoid the formation of scum, but, if found, remove as effectually as possible;

(5.) To obtain higher strengths than those corresponding to the alkali contained in solutions fulfilling the conditions named, solid caustic soda must be introduced into the solutions from time to time.

Proceeding now to the investigation of the decomposition of these solutions, the severest of tests was applied, namely, keeping in glass cylinders in full daylight throughout last summer.

Taking this into consideration the medium strengths show a rate of decomposition not prohibitive from a commercial standpoint, but the decomposition of the very high strength hypochlorites is so rapid that they need not be further considered.

| | | | | | | |
|---|---|---|---|---|---|---|
| The solution | D, 28·6 % Av. Cl. | had fallen in | 4 | weeks | to | 18·6 % |
| „ | E, 31·1 % Av. Cl. | „ | 3 | „ | | 24·1 % |
| „ | H, 49·2 % Av. Cl. | „ | 2 | „ | | 28·6 % |
| „ | I, 44·5 % Av. Cl. | „ | 6 | „ | | 18·4 % |

that is, the very high solutions had fallen in equal periods to much the same point as the 30 per cent. solutions; or, in other words, at some point not much above 30 per cent., hypochlorite becomes absolutely unstable.

Further tests after still longer periods gave:—

| | | | | |
|---|---|---|---|---|
| E. after | 4 | months | 10 per cent. | Av. Cl. |
| H. „ | 3½ | „ | 6 „ | „ |
| I. „ | 2 | „ | 15·6 „ | „ |

the gravities in all cases being between 62° and 66° Tw. We felt that we now had sufficient data to propound an explanation of the various observations made, especially with a view to defining and finding the conditions under which a high-strength hypochlorite should have stability.

Reviewing Table I. we find that, with rising percentage of chlorine and concentration of solution, the total chlorine has been steadily diminished, owing to the expulsion of salt.

Now the free salt contains half the chlorine involved in the reaction—

$$2NaOH + Cl_2 = NaOCl + NaCl + H_2O.$$

When, therefore, a solution has been made which is of such a concentration that all free salt has been thrown out of that solution, the *total* chlorine will only be *half* the available chlorine—an apparent paradox, which a consideration of the equation explains.

The reaction for the decomposition of a hypochlorite into chlorate is expressed by the equation—

$$3NaOCl = NaClO_3 + 2NaCl;$$

but when we have reached the point indicated above, where total chlorine = half available chlorine, we have a solution in which salt is absolutely insoluble.

The law of mass states that if, in any system of chemical bodies in solution, a rearrangement of the molecules is possible which can result in the formation of an insoluble molecule, that rearrangement will take place. Apply the law to the latter equation under the conditions premised, and we have, as the stage beyond which no stable hypochlorite can exist, that stage where all free salt has been thrown out of solution. To fix this possible degree of stable concentration is, therefore, a matter of experiment.

The consideration is complicated by the presence of chlorate in most of our early experiments, but even they enable an approximation to be made, as well as give a confirmation of the theory.

Consider the following:—

### *Experiment D.*

| | |
|---|---|
| Active chlorine, 30·7 per cent.; total chlorine | 23·3 per cent. |
| Now the NaCl corresponding to the active chlorine = | 15·3 „ |
| Of which there is now only | 8·0 „ |

that is, a deposition of 47·7 per cent. Here the critical point has not been reached. This solution, after four weeks, was 18·6 per cent. Av. Cl.

### *Experiment E.*

| | |
|---|---|
| Total chlorine | 22·0 per cent. |
| Free NaCl corresponding to active chlorine | 18·85 „ |
| Of which there is now only | 3·15 „ |

Here the deposition of salt is 82·7 per cent., and the point has not yet been reached.

After three weeks the strength was 24·1 per cent., and after four months 9·1 per cent.

On the other hand—

*Experiment H.*

Total chlorine........................ 27·6 per cent.
Free NaCl, corresponding to active chlorine.......................... 29·3 „

Here the point has been exceeded by 6 per cent.

This solution, after two weeks, only tested 28·6 per cent. Av. Cl, and after $3\frac{1}{2}$ months, 5·8 per cent. Av. Cl.

In other words, the whole excess of strength above the stage where all free salt has been deposited is not only valueless with respect to stability, but even adversely affects the keeping powers when the strength has fallen to that of the solutions below the critical point.

To exactly fix this point of comparative stability is difficult, as it will vary within certain limits; but Experiment H (3), under the conditions then existing, would be very near the limit set by our hypothesis. In it active chlorine = 35·6; total chlorine, 18·5; free NaCl, corresponding to active chlorine, 17·8; that is, deposition of free salt, 96 per cent.

We concluded from these experiments, therefore, that it was useless to attempt to manufacture a hypochlorite solution of over 35 per cent. available chlorine. A review of the various concentrations, both before and after keeping, led us to the further conclusion that stability could not be maintained in a solution of a concentration appreciably above 68° Tw.; in other words, in a system of hypochlorite, chlorate, caustic, and chloride of sodium, in the proportions existing in our various experiments, chloride of sodium is soluble at a concentration of about 68° Tw.

With these two conditions added to those already referred to, we again attempted to make a really stable high-strength hypochlorite.

Here, as in the earlier experiments, difficulties of manipulation occurred, which for some time baulked our efforts. We found that the most satisfactory way of working was in a tall glass cylinder, introducing the chlorine by means of a narrow glass tube, and thus preventing local saturation. Frequent stirring was also found advisable, and the concentration was never allowed to exceed 71° Tw. at the commencement, nor 68° Tw. at the end, of each operation.

The details of this experiment are given in Table I. (K.)

The final result was: Av. Cl., 34·0; Cl. as chlorate, 0·4 per cent.; total chlorine, 22·0 per cent., which represents a deposition of salt of only 72 per cent. It would therefore be possible to obtain a somewhat higher strength, which should be stable upon our hypothesis, but the one in question was of sufficient strength to be a fair criterion of the stability of a really high strength hypochlorite. After keeping for a week in a dark but open vessel, the strength had only fallen by 0·3 per cent., containing still 33·7 per cent. available chlorine.

Compared with the statements made concerning hypochlorite of soda of greater strength than 5 per cent., we contend that this may be called a stable solution.

One modifying condition we will in conclusion discuss, but this condition strengthens our contention that hypochlorities even of very high strength are stable. Reverting to Bhaduri's investigations, we must especially criticise his statement that hypochlorites of 8 per cent. decompose fairly rapidly, with evolution to a certain extent of oxygen in the cold.

This decomposition he places as high as 0·3 per cent. of Av. Cl. in four hours.

That he obtained these results we quite believe, but we contend that he has assumed too lightly that the coloration of the liquid already mentioned was due to permanganate, and has ignored its possible effect upon the decomposition.

Our experience from both practical and special experiment is that hypochlorite of soda is stable for considerable periods in the absence of iron, and Bhaduri's results only agree with ours on the assumption that the colour of his solution was due, as in ours, to ferrate of soda. We have had unpleasant evidence of the evolution of oxygen in the presence of iron as the following anecdote will show:—In the early days of this business we were negotiating for the shipment of hypochlorite of soda to a coastwise port that shall be nameless. A sample had been forwarded to the shipowner's office in a tightly-stoppered stoneware bottle. Unfortunately the bottle burst at the office, giving the office floor and desk a much needed but, locally, too violent cleansing. That line of steamers has placed hypochlorite in the class of "dangerous explosives," and will not carry it at any price.

TABLE II.

| Original Test. | | Reduction of available Chlorine. | | | | | | | Remarks. |
|---|---|---|---|---|---|---|---|---|---|
| Av. Cl. | $Na_2O$. | 1 Week. | 2 Weeks. | 3 Weeks. | 4 Weeks. | 5 Weeks. | 6 Weeks. | 7 Weeks. | |
| (A.) 11·2 | 0·2 | 11·1 | 11·0 | 10·7 | 7·6* | .. | .. | .. | * Alkali all carbonate. |
| 10·7 | 0·4 | 10·7 | 10·7 | 10·5 | 8·7 | 7·0* | .. | .. | |
| 8·5 | 2·2 | 8·5 | 8·5 | 8·5 | 8·5 | 8·5 | 8·3 | 6·2* | |
| (B.) 11·2 | 0·2 | 11·1(*a*) | 7·5 | 2·5(*b*) | .. | .. | .. | .. | (*a*) $Fe_2(HO)_6$ introduced.<br>(*b*) Caustic alkali still present. |
| 6·7(*a*) | 3·2 | 5·9 | 5·2 | 4·0 | 3·0(*b*) | .. | .. | .. | |
| (C.) 27·6(*c*) | 2·8 | .. | 26·6 | .. | .. | .. | .. | .. | (*c*) Ferrate present.<br>(*d*) Ferrate removed. |
| 20·0(*d*) | 2·0 | .. | 20·0 | .. | .. | .. | .. | .. | |
| 29·6(*c*) | 2·5 | .. | 28·6 | .. | .. | .. | .. | .. | |
| 23·0(*d*) | 1·9 | .. | 23·0 | .. | .. | .. | .. | .. | |
| 12·8(*d*) | 1·4 | .. | .. | .. | 12·8 | .. | .. | .. | |

The same occurrence was experienced in less violent form on many occasions, a hypochlorite containing iron being always found under pressure if kept in a closed bottle. Upon the other hand, hypochlorite of soda freed from iron, of which there is also a commercial form, could be kept in closed bottles or sent by post without appreciable rise in the pressure.

These are rough tests, but are fully borne out in every case coming under our notice, and, though we do not go so far as to say that there is no evolution of oxygen, a consideration of the experiments in Table II. show that in the case of a really iron free hypochlorite the evolution cannot be measurable in a month because during that time no decomposition has occurred.

*Group A.* were conducted to show the action of carbonic acid in the air upon hypochlorites; the decomposition is very low, until all the alkali is converted into carbonate after which the decomposition proceeds rapidly.

These solutions are all free from iron.

*Group B.* Ferric hydrate was introduced in the first case after one week, in the second at the commencement. The rapid decomposition of these when compared with Group A. demonstrates the effect of iron even in the cold.

*Group C.* are high-strength hypochlorites. By removal of the sodium ferrate the decomposition is completely arrested for two weeks and probably much longer.

The last sample is a chance commercial sample free from iron.

We therefore feel perfect confidence in stating that the presence of iron in a hypochlorite has a serious influence upon its stability.

The evolution of oxygen is easily explained by the mutual reaction of hypochlorite upon ferrate, but why this should largely increase the decomposition into chlorate is not clear.

That ferrate is actually present in our liquors has been proved with the spectroscope by Mr. C. Longuet Higgins.

In all our earlier experiments upon the high-strength hypochlorites this ferrate was present, and hence the results are relative rather than absolute. In the last experiment, owing to a different kind of glass being used, it was very small in quantity but it did still exist.

Upon these various facts we draw the following conclusions as to the nature of hypochlorite of soda. Up to the point when salt commences to deposit hypochlorite in the absence of iron is stable. From that stage to a point where all the free salt is deposited hypochlorite in the absence of iron is not quite so stable but its stability is considerable. Beyond the stage where all free salt is deposited absolute instability exists.

Under favourable circumstances a hypochlorite can be made containing 35 per cent. by volume of available chlorine, which is, except for hydrogen peroxide of unmarketable strengths, the strongest oxidising solution in the cold.

*Crystals of Hypochlorite of Soda.*—During the course of our investigations we succeeded in producing crystals of hypochlorite of soda.

We dried these by drawing off the liquor by means of a vacuum filter as rapidly as possible, dissolved in weighed water and after reweighing analysed the solution. The results showed—

| | Per Cent. |
|---|---|
| NaOCl | 37·6 |
| $NaClO_3$ | Nil |
| NaCl | 3·7 |
| Water (by diff.) | 58·7 |
| | 100·0 |

A small amount of caustic was also present.

The salt is probably present as a deposit on the crystals. The crystals are fine needles, and on the above composition would have a formula, $NaOCl \times H_2O$ with × not more than 6. According to all the literature of the last 50 years hypochlorite of soda has been known only in solution; but in 1827 an investigator, whose name we have been unable to discover, on *boiling* a solution of hypochlorite and carbonate of soda obtained needle-like crystals which bleached indigo. Whether this was anticipation we are not prepared to say, but we believe that we are the first to isolate and submit samples of these crystals, at any rate to this Society.

In conclusion, we must express our acknowledgment to Mr. Arthur Carey for the initiation of the work upon Hypochlorite of Soda to which this paper is merely the logical sequel.

## NOTE ON THE SOLUBILITY OF CHLORINE IN BRINE.

BY CHARLES A. KOHN, PH.D., B.SC., AND F. O'BRIEN.

The solubility of chlorine in saturated sodium chloride solution is a factor of some interest and importance in connection with electrolytic alkali processes.

Determinations of the solubility of chlorine in sodium chloride solutions have been made by Goodwin (Ber. **15**, 3039) and the Kumpf (Ann. Phys. u. Chem. Beibl. 1882, Vol. **6**, p. 276), but the experiments are practically all restricted to unsaturated solutions. Those of Kumpf appear to be the most reliable, he gives the following interpolation formulæ for the absorption coefficients for solutions containing 9·97 per cent., 16·01 per cent., and 19·66 per cent. of sodium chloride respectively:—

$x = 2{\cdot}2317 - 0{\cdot}05505\,t + 0{\cdot}000025\,t^2$ for 9·97 per cent. NaCl.

$x = 2{\cdot}1923 - 0{\cdot}11281\,t + 0{\cdot}0032806\,t^2 - 0{\cdot}00004218\,t^3$ for 16·01 per cent. NaCl.

$x = 1{\cdot}7440 - 0{\cdot}06717\,t + 0{\cdot}00117\,t^2 - 0{\cdot}0000097\,t^3$ for 19·66 per cent. NaCl.

In addition, Kumpf states that a marked separation of sodium chloride occurs when chlorine is passed into a saturated solution of the salt, and that the co-efficient of solubility of the solution is 0·6179 at 19°·9 C.

The method adopted in the present determinations was to pass dry chlorine into a flask fitted with an inlet and exit tube, thermometer and syphon tube, the last for drawing the samples. The chlorine was passed in until the solution was saturated, a 100 c.c. sample drawn, an excess of potassium iodide added, and the liberated iodine titrated with sodium thiosulphate. The solubility was determined at 14°·5, 29°, 60°, and 82°; in each case a saturated brine solution of sp. gr. 1·205 at 15° corresponding to 26·39 per cent. of sodium chloride was employed, and the temperature kept constant by means of a water-bath provided with an agitator.

The following results represent the average of two or more experiments:—

| Temperature. | Co-efficient of Solubility at 0° C. and 760 mm. | Solubility at t° and 760 mm. |
|---|---|---|
| 14°·5 | 0·3607 | 0·3898 |
| 29°·0 | 0·3125 | 0·3458 |
| 60°·0 | 0·1332 | 0·1625 |
| 82°·0 | 0·0586 | 0·0763 |

Chlorine is, therefore, about one-sixth as soluble in saturated brine as in water. According to Kumpf no oxygenated chlorine compounds are formed when chlorine is passed into sodium chloride solutions, but we are unable to confirm his statement. When the chlorine is passed for more than two hours, the solution begins to become acid, and the solubility gradually rises without, however, reaching a maximum. This is in accord with Berthelot's observations (Compt. Rend. 1880, **91**, 191) on the solubility of chlorine in water and in chlorides of the alkaline earths. He found that the solubility increased on prolonged treatment with chlorine and varied with the intensity of the light. These variations are due to the decomposition of water with the formation of oxygenated chlorine compounds. The high coefficient of solubility found by Kumpf for saturated brine is probably due to such decompositions.

The following experiments illustrate this action. In Experiment I. a slow current of chlorine was passed; in Experiment II. a more rapid current was employed, and consequently the decomposition began sooner:—

EXPERIMENT I.

| Time. | Solubility at 0° C. and 760 mm. | Temperature. |
|---|---|---|
| H. M. | | |
| 2 15 | 0·3188 | 14° |
| 2 45 | 0·4339 | 14°·5 |
| 3 15 | 0·5243 | 15° |
| 3 45 | 0·5522 | 16° |

EXPERIMENT II.

| Time. | Solubility at 0° C. and 760 mm. | Temperature. |
|---|---|---|
| H. M. | | |
| 1 40 | 0·4312 | 14° |
| 2 5 | 0·5180 | 15° |
| 2 50 | 0·6001 | 15° |
| 3 25 | 0·6382 | 16° |

No deposition of salt was observed even after passing the chlorine for 3½ hours.

# London Section.

*Meeting held on Monday, December 5th, 1898.*

MR. BOVERTON REDWOOD IN THE CHAIR.

## OZONE AND ITS COMMERCIAL APPLICATIONS.

BY COL. W. J. ENGLEDUE, (LATE) R.E.

The purposes to which ozone or ozonised air can be applied are many and various, but it has been found that the effectual application is by no means an easy matter. Much time has been expended and mechanical methods tried before results could be attained, and much money has, I fear, been wasted; but still certain satisfactory results have been arrived at, which it is hoped will benefit all concerned.

The experiments made with ozone by the Syndicate, of which I am the chairman, make it possible to apply this powerful oxidising agent in a practical manner to a great variety of purposes, some of which I will shortly explain.

Dealing first with the production of the ozonised air, I do not propose to draw invidious comparisons between the few ozone generators known to the public; suffice it to say that we are quite satisfied with the Yarnold ozoniser, we find it simple and cheap as to construction, effective as an ozone producer, whether from oxygen or from atmospheric air, easily and quickly repaired, and economical as to electric current consumed. It is calculated that the maximum yield of ozone is 175 grms. per E.H.P. hour, which is considered satisfactory. The experiments to determine this having been carried out by Mr. S. G. Rosenblum.

The method of producing the ozonised air is simple. A wooden box is furnished with a series of shallow trays, on which fresh quick lime is packed. Atmospheric air is drawn through this box by means of a Roots blower, and, thus deprived of its moisture, is then forced through the ozone generators into a compressor, which compresses the ozonised air to about 15 lbs. per square inch in an iron cylinder. Thence the ozonised air is led to the various operations for which it is used. In processes requiring a large supply of ozonised air, it would be advisable to dry the air by means of refrigerators, but this would increase the cost of plant and production. The generators now in use at the works have been continuously in operation for a considerable period, and are still working well. All the repair necessary to renew their efficiency is the changing of the tin foil between the plates, which is simple and inexpensive.

In regard to the practical applications of ozonised air, I will in the first instance confine myself to those which are of proved utility and successful in working on a large scale. These are:—

Cleaning brewers foul casks, known in the trade as "stinkers"; refining and bleaching of linseed oil and palm oil; the production of siccative oils and blown oils for lubricating purposes; the manufacture of oxidised oil (cement) for linoleum and seasoning of linoleum.

As regards the cleansing of brewers' foul casks, it is well known that the loss to the brewing trade arising from "stinkers" is enormous. In Burton alone, it is calculated that 100,000*l.* per annum is thus sunk. This can easily be realised, when it is remembered that every 36 gallon barrel costs about 25*s.*; and that directly it becomes foul, as often happens within a very short period of its life, its value is only 5*s.* or less. This foulness or "stink" appears to be caused by a fungus, which often penetrates to a considerable depth in the wood, and can only be effectually reached by gases under pressure. Very large amounts have been spent in vain by brewers in trying to find some cure for these casks, but it has remained for the intensely oxydising action of ozone to accomplish the hitherto impossible.

The apparatus required is simple and inexpensive; a Westinghouse brake pump is used to force the ozonised air into the casks, which are supported in iron cradles to enable them to stand the pressure of about 50 lb. to the square inch. Before turning on the ozone, steam is led in to moisten the interior of the barrels, and the process consists of alternately steaming and ozonising under pressure for about two hours, this time depending on the condition of the cask. Previous to treatment it is sometimes necessary to lightly shave the inside of the casks, if very foul; and it is also of advantage to soak in water for two or three days. In the worst cases an air-tight cylinder is used, in which pressure is brought to bear on both the inside and outside of the cask, the steaming alternating with the ozonising as before.

At a brewery possessing surplus steam power and an alternating current of electricity, it is calculated that a plant to deal with say 30 "stinkers" per day could be erected for about 500*l.*; such installation could also ozonise and sterilize daily 100 casks in the ordinary course of cleansing, in order to destroy any germs which might taint the beer. This process of washing out with steam and then ozonising has been advocated by the professional adviser of one of the largest firms at Burton, who at present lose a considerable proportion of their export ale owing to casks which have gone off, notwithstanding the greatest care in examining them. As 35 millions of barrels of beer are brewed yearly in the United Kingdom, there is a promising outlook for ozone if only a small proportion of the brewers were to adopt the system.

The refining of linseed oil is accomplished by merely passing ozonised air through it. The oil is freed from moisture, and those impurities called "mucilage" are eliminated, which by the ordinary treatment could only be removed by heating the raw oil to a high temperature at the expense of its colour, or by settling in tanks for a long period. By the ozone process all the valuable properties of the linseed oil as a drying oil are retained, whilst the consistency is slightly increased. The process occupies only two to three hours, and entails no loss of material, while the comparatively low temperature at which the operation is conducted, minimises the risk of fire and explosion, effecting consequential saving in premiums on insurance.

Irrespective of the ozone plant, steel jacketed steam pans for holding the oil and for keeping it at the required temperature are all that are necessary, with pure tin perforated coils for conveying the ozonised air to bubble it up through the oil. The ozonised air should be conveyed from the generators by glazed earthenware pipes. As the question of economical working on a large scale resolves itself chiefly into the cost of producing ozonised air, Dr. Lewkowitsch has carefully checked all the electrical and air measurements, and found that, based on a production of 15,000 cub. ft. per hour, the cost of blowing air through the generators would amount to 12*d.* The indications of a Siemens torsion electrodynamometer showed that for a voltage of 100, there were required for the then produced 1,800 cub. ft., 1·12 ampères, giving a wattage of 112. This would give for 15,000 cub. ft. 940 watts, or, say, one B.T.U. One B.T.U. is supplied at the cost of 4*d.* by the Electric Supply Company, but could be produced by dynamos at the works for 3*d.*

Adding for depreciation of ozonising plant, contingencies and air-drying plant, 2*d.*, the following summary was arrived at by Dr. Lewkowitsch:—

| | *d.* |
|---|---|
| Cost of blowing air | 12 |
| Electrical energy | 3 |
| Depreciation, &c. | 2 |
| Cost of 15,000 cub. ft. ozonised air | 17 |

As the refining of one ton of linseed oil would require about 11,000 cub. ft. of ozonised air, costing, say, 1*s.* 1*d.*, it would, therefore, appear that the cost of the ozonised air is extremely moderate.

Based on a production of 30 tons of refined linseed oil per week, and allowing for extra staff, depreciation of the special plant, and interest on capital, it is calculated that at an existing refinery the cost of refined oil by the ozone process would be about 7*s.* per ton, exclusive of royalty. As regards the difference in value between the raw and refined oil, this was fixed recently by a firm of oil blowers and refiners to be 30s.; there is, therefore, a fair margin of profit, which would be increased when dealing with larger quantities.

I have here two small bottles, one holding the raw oil as supplied by crushers, and the other containing the refined article.

In the case of palm oil, the advantages of the ozone treatment are very marked. It is throughout a "dry" process. The usual treatment with chemicals, and the repeated washing the oil to remove the last traces of the chemicals, is entirely dispensed with, and the bleaching of a batch of oil is accomplished in the short time of three to four hours, while both the "titre" and melting point are increased. Messrs. Norman Tate and Co., of Liverpool, found that the "titre" increased from 15·5 per cent. to 46 per cent., and the melting point from 38·1 per cent to 43·5 per cent. The plant required for the refining and bleaching of palm oil is much the same as for the linseed oil, and the expenses are not much larger. I have here samples of the unbleached and bleached product, the dark specimen being a very impure "Benin" oil, which is very difficult to bleach by any ordinary process. Considerable quantities of both Benin and Lagos oils have been submitted to brokers for quotations, which have shown that the values have been enhanced to the extent of from 2*l.* to 3*l.* 10*s.* per ton. A cake of soap made from ozone refined Benin palm oil is remarkable for the delicate fragrance of its odour and its good colour.

I now come to the manufacture of quick-drying (siccative) oils. These oils are prepared by dissolving varying quantities of "driers" in ordinary raw oil; they are then sprayed in conjunction with ozone in a specially designed "atomizer" at a certain temperature for different lengths of time, up to three hours. According to the quantity of drier employed, oils of varying degrees of drying power are obtained, and according to the length of time the oils are sprayed, siccative oils of varying consistency (body) are produced. It is found that oils treated by the ozone process possess in the highest degree those properties, which should be looked for in an ideal siccative oil, viz., drying power, consistency, hardness, smoothness, elasticity and glossiness of coat, combined with paleness of colour. The most essential point in the process is that no high temperature is required; furthermore, there is no loss of material, whereas in the usual processes a considerable percentage of oil is lost. There is a further saving in the entire absence of "foots." The oil can be turned out rapidly in a marketable condition, and does not require preliminary settling over a prolonged period. The specimens I have here show the difference in colour between the ozone treated drying oil and the ordinary boiled oil of commerce, and it will at once be seen that the use of a light oil must favourably affect the colour when mixed with delicate pigments such as white lead, zinc white, and primrose chromes.

As regards the production of blown oils for lubricating purposes, these can be prepared in the same description of apparatus and by the same method as for dealing with refined linseed oil, so that an oil refiner having a plant constructed for the linseed, could also manufacture blown oils from rape or cotton seed oils.

I have here a specimen of a blown cotton seed oil prepared from American raw oil, also a bottle of the original oil: you will notice the vastly improved colour, and the specific gravity has been increased from ·92 to ·98. This oil was prepared by ozone in 4 hours, whereas the usual process occupies 18 hours.

Closely allied to the preparation of drying oil, is the manufacture by ozone of oxidised oil for "cement" for linoleum. Starting with commercial raw linseed oil and employing a small proportion of driers, a solid oxidised oil of a pale colour can be prepared in the short time of from 10 to 13 hours, without the aid of any mechanical appliances for stirring or heating. The very short period required for making the solid material will no doubt commend this process to the manufacturers of linoleum and similar products. The older method of making the solid oxidised oil by the "Scrim" process should also be favourably affected by the use of ozonised air, as a more rapid result would be arrived at, and also a lighter coloured product.

I have here a specimen of the oxidised oil for "cement" prepared in 13 hours by the ozone process.

Ozonised air has been found to have a very favourable influence in the seasoning of linoleum. A long series of experiments were executed by Dr. S. Rideal, at the works of the Commercial Ozone Syndicate, with a view of ascertaining the suitability of ozone produced by the Yarnold method for the seasoning of linoleum. For this purpose samples of fresh unseasoned linoleum were procured from various large manufacturers, and included plain, inlaid, and painted varieties. In all the experiments care was taken to adhere as far as possible to the conditions maintained in the existing method of seasoning, substituting, however, ozonised air for ordinary air. It was found that in the case of most of the linoleum seasoned by means of ozone, the results were superior to those produced by the present methods as regards time of seasoning, and the quality of the seasoned article. Whilst the seasoning by the present processes occupies it is said from five to eight weeks, the seasoning by means of ozone could be accomplished in from five to ten days, according to the quality of the unseasoned linoleum. It was noted that, in all the samples seasoned by means of ozone, the colours were very markedly and advantageously improved. It is thought, having regard to the cheap rate at which ozonised air can be produced, that the extra expenditure involved in the introduction of ozonised air instead of atmospheric air for the seasoning of linoleum will be more than compensated by the saving effected in time of seasoning, and consequently in labour and interest on capital invested in the unseasoned material, as the process would, on a given plant, allow of a quicker and larger turnover.

These are brief descriptions of applications of ozone to commercial purposes and of processes, which have been exhaustively investigated by experts, and for which there are no difficulties in designing works suitable for extensive commercial businesses.

Whilst these processes have been in course of development, experiments have been made as to the value of ozone in other industrial branches. The bleaching of cotton yarn has had more particular attention. I have here a skein of the bleached and one of the untreated cotton, you will notice the difference in colour, which has been arrived at entirely by the action of ozone in a very short space of time, without the use of any chemical bleaching medium. We expect soon to be able to introduce this process into Lancashire on a commercial scale.

Considerable attention has been devoted to the refining of dark brown sugars, and as these specimens will show you, we have been fairly successful on a laboratory scale, but it still remains to be seen how the ozone can be practically and economically applied, when dealing with large quantities.

Experiments have been made in dealing with sewage, and the most maledorous effluents can be quite deodorised in a few minutes by the action of ozone, but the practical use of such deodorisation has yet to be determined.

In conclusion, gentlemen, if any of you would like to see the processes I have described in actual operation I shall be happy to afford you facilities for doing so, when, if you so wish it, your own samples can be operated on.

### Discussion.

The Chairman observed that before inviting discussion it might be convenient for him to call attention to the fact that Colonel Engledue had in his paper expressly disclaimed any intention of drawing invidious comparisons between the Yarnold ozoniser, which his company employed, and other apparatus for producing ozone. At the same time, he ventured to think that it would add materially to the value of the paper if there were included in it a statement as to the grounds upon which the company based their preference for that particular form of apparatus. Through the kindness of Mr. Gordon Salamon, who had devoted much attention to the subject of the application of ozone in chemical industries, he had recently had an opportunity of inspecting an important installation in Paris for the employment of ozone in certain industrial processes, and he had been greatly impressed not only with what had actually been accomplished at that factory, but even more with what he might call the great potentialities of ozone as so employed. He was satisfied that there was in the employment of ozone an enormous field for future development. At the same time it was essential that they should know, as far as possible, what were the good and the bad points in the various forms of apparatus employed in the production of the ozonised air. He thought that the ideal to be aimed at in the discussion which he hoped would follow on this very suggestive paper would be the elucidation of certain points which for convenience he would venture to summarise thus:

(1.) The cost per pound of active oxygen, without allowing for labour, establishment charges and amortisation, stating the scale of working upon which the estimate is based.

(2.) The cost of removing moisture from the saturated air. In connection with that he might point out that whilst Colonel Engledue attached considerable importance to the drying of the air, an operation which would be expensive to carry out on a large scale, some authorities considered that the presence of moisture did not much interfere with the ozonising action.

(3.) Whether the Yarnold ozoniser did not become heated by constant working; and whether heating did not diminish to a considerable extent the production of ozone. He knew that some experts considered it desirable that the air to be ozonised should be cool, and that in one installation steps had been taken to largely reduce its temperature.

(4.) Whether it was not necessary to remove oxidised nitrogen products before employing the ozonised air for some of the various purposes specified.

(5.) What was the percentage of oxygen contained in air passed through the Yarnold ozoniser converted into ozone; and

(6.) Generally, what were, in the opinion of Colonel Engledue and his professional associates, the advantages of the Yarnold ozoniser. He thought that if they kept those points in view they might elicit information which would largely add to the value of the paper.

Mr. W. F. Reid considered that there was one point upon which he was sure the meeting would be unanimous, and that was, the enormous field that existed for the use of ozone. All sorts of applications had been already proposed, and a great number had been actually tried, and their gratitude was due to Colonel Engledue for producing a paper of so practical a character, and for classifying his methods in so honest and straightforward a statement with respect to the things which had been found to be successful and those which were not yet successful. He had never heard any paper in which the practical applications of a body had been so carefully sifted from those which were only theoretical. He had had an opportunity of personally seeing some processes referred to, and was satisfied that they were practical and were being carried out on a commercial scale. The palm oil, for instance, was in the first instance a most disagreeable-smelling substance; but after treatment by ozone for a short time one got an almost white substance with an agreeable odour. With regard to the Chairman's remarks about the temperature of the generator, it had, he thought, been stated by the well-known German investigator, O. Brunck, that ozone would stand a temperature of 300° C. without decomposition, and that the limit of temperature was not so narrow as might be imagined from the literature on the subject. Then there was the question whether the ozone might not have been confused with the oxidation products of nitrogen. In his opinion some of the uses of ozone were due to these nitrogen oxidation products; and in many cases the chief effects might be due to them. In this view, any chemical application of ozone should be accompanied by some careful analytical method of distinguishing between these bodies and the ozone itself. The sense of smell was not sufficient; for even experts could not always distinguish between them in that way. Another difficulty was the want of some practical method for the estimation of the amount produced, ozone being so difficult to estimate quantitatively. With regard to the use of ozone for bleaching yarn, it was interesting to find that it could be done without the use of chlorine, because at the establishment in Silesia they had found it necessary always to use chlorine as a finishing, though of course much less was used. It was curious that ozone had a distinctly selective action. It would act upon certain substances in a mixture without touching others. For instance, in the case of linseed oil, first of all the colouring matter would be acted on, and then that mysterious body known as mucilage; a general term for all the impurities that settled out of oil when stored. The ozone having acted upon these impurities, then followed the oxidation of the oil itself. He feared the Author would consider his remarks a disparagement of his success in oxidising oils, but he could not help saying that the present methods were so cheap that there did not appear to him a good opening for the use of ozone in this direction, the present cost was only a few shillings per ton, and it was difficult to see any margin for the cost of the electrical energy required to convert the oxygen into its more active form. In connection with the particular establishment he had mentioned some figures were lately given with regard to the h.p. required. A 3-h.p. engine would bleach about 1,100 kilos. daily of grey yarn, or say, about 3-h.p. per ton. As to the production of ozone it had been stated and never controverted, that it was not due to a dark discharge of electricity but to a light discharge, though there was no sparking. Wherever ozone was present there seemed to be a certain definite electric discharge which produced a maximum quantity of that body. Therefore those who were interested in ozone production would be well advised not to rush at it with any preconceived notions; for the laws which governed its production were up to the present practically uninvestigated and presented a great field for research. One application of ozone put before them in a very practical manner, and which would probably have a great future, was its bactericidal action. It was known that these "stinkers" were due to certain organisms that penetrated the wood, and which could not be got rid of except by destroying them within the wood itself. Apparently ozone dealt with these bacteria extremely well. In treating wine and other liquids, the ozone first attacked those substances in them which were easily oxidisable and then the germs were destroyed. That was one reason why it was very difficult to apply ozone on a large scale unless its production was cheapened, and that again was the reason why it would be extremely difficult to use it for the treatment of sewage. The liquid for which ozone was mostly used on the Continent was brandy, upon which it produced an effect very similar to ageing. Ozone had also been used on a considerable scale in Hamburg for purifying coffee. In the ordinary process of drying coffee a number of beans were crushed, some of which were unripe, and these beans would subsequently decay, and after packing would communicate a disagreeable taste to the bulk of the coffee, and render it unsaleable. By passing ozone through the beans this unpleasant flavour was destroyed, and the value of the coffee was considerably increased. That was an eminently practical use of ozone. Of theoretical uses the most interesting, perhaps, was the production of nitric acid from the air. It was well-known that that could be done by the arc discharge, but it was possible that by the discharge which produced ozone one might be able to produce a larger number of units of nitrogen compounds, and if so, that would constitute an important improvement. The use of an ozone carrier was often referred to, and he believed Col. Engledue had himself produced a solution containing turpentine and a large percentage of ozone. When ozone passed through turpentine white vapours were given off of an intensely active character. For ages turpentine had been used as an oxygen carrier for drying oils, and it was not surprising, therefore, that it was now found to be a useful carrier for ozone. Another eminently practical use for this body was the bleaching of starch. The impurities therein were oxidised much more rapidly than the starch granule itself, and the starch was thereby greatly improved in value. It would be seen that most of the uses of ozone depended upon the oxidation or destruction of a very small portion of material which interfered with the commercial value of the article treated. One peculiarity of ozone was that one could never tell exactly what it would do in any particular case; which of the decomposable substances in any given material it would first seize upon. Col. Engledue had treated a sample, he (the speaker) had sent him, and from which he had anticipated a certain result. The result obtained was totally different, though in itself very remarkable and likely to prove of some value. He would strongly advise any member who wished to take up a new line of research to try ozone. Among other minor applications he might mention that ozone would in a few seconds rid plants of parasites, without having an ill effect on the plants themselves. Another feature worth attention was the fact that in ozone one was using an entirely volatile substance, and that was a great advantage in dealing with food products.

Dr. S. Rideal regretted that he had not been present to hear the paper read. His own work had been specially directed to the action of ozone on drying oils, and to the formation of hydrogen peroxide. He would like to obtain some definite information with regard to the actual cost of ozone as an oxidizing agent. He had received a letter only yesterday from a member in the midlands, asking for information as to whether ozone could be obtained in large quantities sufficiently cheaply to use as an oxidising agent in the colour industry. He would therefore ask, firstly, what was the actual cost per pound of available oxygen, and how did that cost compare with other oxidising agents. On the blackboard it was stated that 15,000 feet of ozonised air cost seventeen pence, but it was necessary to ascertain what weight of ozone was present per cubic foot of such air before one could compare the cost of ozone with that of other oxidising agents. He believed, likewise, that it would be found to be very dear. Secondly, he would like to know what would be the first cost of an apparatus to produce, say, fifty pounds of ozone per hour. Thirdly, he would like to know whether the Ozone Syndicate were prepared to supply a convenient small ozonator for laboratory purposes.

Mr. Otto Hehner quite agreed with Mr. Reid's view as to the advisability of limiting the application of this process to the improvement of those chemical bodies which were small in bulk and high in value. When it came to dealing with matters in large bulk, the question assumed quite a different aspect, especially proposals for treating such matters as London sewage. In very few cases had they the means of measuring the quantity of ozone necessary for effecting the results of which they had some illustrations that evening. But in one case, viz., the production of blown oils, they had a very accurate way of ascertaining the necessary quantity. For example, in the case of linseed oil to be treated by ozone and to be oxidised it was the oxidising action which was wanted and nothing else. They started with a body having an iodine absorption of 180. That figure in the blowing operation went down to about 80. It was easy to convert those figures into the oxygen equivalents; 127 of iodine being equal to 8 of oxygen, or 24 of ozone, consequently a diminution of 100 in iodine absorption corresponded to about 20 per cent. of ozone consumed; hence to convert one ton of linseed oil into solid oil one would require 4 cwt. of ozone. Taking the author's figures of 175 grammes of ozone per horse power per hour, costing, say, one penny, one could easily calculate what 4 cwt. of ozone would cost, namely nearly 5*l.*, without including cost of blowing the large bulk of air necessary. Considering that the price of linseed oil was only 16*l.* per ton and the price of the blown oil a pound or two more, he did not see how this process could possibly be a commercial success. He had no wish to disparage the uses of ozone indicated by Col. Engledue, but to indicate that it would be unwise to go out of the track in which ozone had obviously a useful application, first, for the destruction of colouring, &c. odoriferous matters, and secondly, for the production of valuable materials, such as certain organic compounds, in which the cost of production was comparatively immaterial. But when it came to dealing with bodies so valueless as sewage he considered the process quite out of place.

Mr. G. H. Hurst had been much interested in the preparation of siccative oils, and had come to the opinion that ozone was a very useful material for that purpose. With regard to the question of cost he would like to point out that when dealing with oils, which required only a small amount of oxidation, it was sometimes cheaper to use an expensive oxidiser, whose action could be regulated, than one like bichromate of potassium which was not so amenable to control. For instance, in bleaching palm oil with bichromate and acid, sometimes the operation would succeed and sometimes it would fail, in which case the total cost might be greater than in using ozone, which yielded a better product, and was always successful. In preparing siccative oils from linseed oil one wanted to bring about a certain degree of oxidation and no more. If one employed powerful oxidising agents they often failed because they carried the oxidation too far, but with ozone one could regulate the action with a great degree of nicety. In the case of siccative oils, too, it was important to get a pale tint. That was almost impossible with the older processes; but by using ozone one could get an almost colourless product.

Mr. E. Grant Hooper said that boiled or oxidised linseed oil exhibited increased density and refractive power with a lowered iodine absorption, and he would like to know if Col. Engledue had given or could give the figures illustrating the changes in the oils of which he had exhibited samples. One could easily appreciate the advantages in colour resulting from the ozone treatment; but it struck him on mere inspection that the treated sample of linseed oil seemed to be thinner than most samples of boiled oil, and might not exhibit the drying character of the latter. Perhaps the author would be able to state whether in actual working his treated linseed oil had proved as satisfactory as ordinary boiled oil.

Mr. H. S. Elworthy desired to know whether the ozone treatment had any effect upon sugar beyond that of decolorisation. If it had not, the process might be of considerable value in India, where the natives had a strong prejudice against sugar purified by means of animal charcoal. With regard to the question of the cost of the process, he would point out that the value of ozone would depend, not entirely on the cost of its production and application, but upon its comparison with the bodies it substituted, both as regards the cost of production and the value of the work done. The same argument applied to its bacteriological action. One speaker had stated that the first action of the ozonised air was upon the colouring matter in the casks only, and the final action only on the bacteria, which caused those casks to become "stinkers." He would like to know if that was absolutely certain, because it seemed to him that the action of ozonised air might be, first, to select the bacteria to be acted upon, and in such case there would be a large field for its use in bacteriological applications, such as the disinfection of plague houses. He had been in Bombay during the recent visitation of plague, and the municipal authorities there were prepared to go to any expense in obtaining liquefied chlorine for the treatment of houses. But it seemed at least possible that this ozonised air, being so powerful a bactericide, might have been used with great advantage.

Dr. D. Martini had not understood Col. Engledue to state clearly what percentage of ozone was present in the ozonised air, but only that he was able to pass about a thousand cubic feet of air per minute through the apparatus. The best chemical text-books stated that ozone was an allotropic modification of oxygen, and that only about four per cent. of it was contained in atmospheric air. If, therefore, ozone were merely converted oxygen why could one not get in the condition of ozone the whole of the 21 per cent. of oxygen present in atmospheric air? With regard to the exhibits before the meeting, had Col. Engledue found that the alternate treatment by steam and ozone caused any leakage in the casks. He had reason to believe that it would have some such effect upon the material of which they were made, seeing that ozone had a very beneficial effect in seasoning woods. He would also like to know what was the cost of bleaching a ton of yarn, such as had been shown them. Mr. Gartside, one of the largest bleachers in Great Britain, had told him that he estimated the cost of bleaching at about a halfpenny per pound; therefore the bleaching of cotton would cost roughly *4l.* 15*s.* per ton. Mr. Gartside was also of opinion that ozone would be practically useless for the purpose of bleaching cotton unless it could be guaranteed to give a perfectly even whiteness throughout the whole length of cloth.

Mr. S. G. **Rosenblum** expressed the hope that, in considering the merits of this paper, the fact would not be lost sight of that Mr. Yarnold's apparatus had been a very important element in achieving the results arrived at.

Prof. E. H. **Rennie** observed that some of the previous speakers had expressed a doubt as to whether some of the effects attributed to ozone might not be due to the action of compounds of nitrogen and oxygen present in the ozonised air. If Col. Engledue had made any experiments, with a view to distinguishing between the action of such compounds and that of ozone itself, it would be very valuable to publish the results, as they would have a most important bearing on the question of the application of ozonised air for chemical purposes.

Mr. **Otto Hehner** observed that he had been much struck by the curious fact that the figures put down by the author for the melting points of the fat, before and after ozone treatment, showed a difference of seven degrees in favour of the treated sample, a fact which pointed to the production of elaidine, due to nitrous gases in the ozone used.

Mr. C. **Sevin** said that he had himself been struck by that fact, and would be glad if the author could state whether the melting points of the bodies treated by ozone remained constant at the figures given, or whether after melting they would go back to the usual figure.

Mr. W. F. **Reid** said that one gentleman having put a question arising out of his remark as to the selective action of ozone, he would like to point out that a very interesting paper had been written by Fröhlich on that matter, of which an abstract appeared in the Society's Journal for 1895, p. 164.

Mr. E. **Ackermann** said, with regard to the heat developed in the apparatus, it was well known that ozone could not exist at even 300° C. If, therefore, that temperature was reached, there must be a loss of electricity, which would increase the cost of the ozone actually produced.

Col. **Engledue**, having been called upon to reply, asked to be excused from doing so at the meeting, but promised to add a reply on all the points raised in the discussion on receiving a report thereof. He might say at once, however, on the question of the economical application of the process to oils, that all the results given in the paper were those produced by the treatment of oils on a large scale, and that so applied the ozone process had been found more economical than any other process.

The **Chairman** thought it was evident, from the interesting discussion which had followed the reading of this suggestive paper, that there was considerable room for investigation, not only in the directions indicated by Mr. Reid, with respect to the application of ozone, but also in regard to the conditions governing the cost of producing it, and it appeared to him, without in any way desiring to minimise the importance of the paper, which had been very properly characterised by Mr. Reid as exceptionally practical, that the suggestions he had made with a view to guiding the discussion were entirely justified. Those suggestions had also, he thought, anticipated the points raised by Dr. Rideal. He was quite in accord with the views put forward by Mr. Hehner, that Col. Engledue and those who were working with him would be wise to confine the application of ozone in the first instance to those branches of industrial chemistry in which either small quantities of certain bodies constituting impurities had to be eliminated, or in which the products obtained commanded a high price.

Col. **Engledue** sends the following reply:—

In replying to the various points raised in the very friendly discussion which followed the reading of my paper, I think that the questions put by the Chairman practically cover most of the remarks made by those who followed him. I therefore reply in the order of his queries:—

1. The cost per lb. of active oxygen. As 1 E.H.P. costing 1*d.* yields 175 grms., the cost of 1 lb. of ozone would be $2\frac{4}{7}$*d.*, therefore 1 lb. active oxygen would cost $7\frac{3}{7}$*d.*, assuming one-third of the ozone molecule only to be capable of combustion; but as in some cases the entire molecule of ozone is absorbed, it follows that the cost would be $2\frac{4}{7}$*d.* per lb. in those cases.

2. As regards the cost of removing moisture from the saturated air, it has been found that the cost for 11,000 cub. ft., required to refine 1 ton of oil, has not exceeded 1*d.* When generating ozonised air on a large scale, it would be advisable to use refrigeration for air drying. It is found that unless the air is dried before passing it through the ozone generator, small electrical arcs are produced, which it is believed would contribute to the production of oxides of nitrogen; it has, therefore, always been the aim to keep a steady violet glow between the plates of the generator, and to avoid all "sparking."

3. Heating of apparatus. Slight heating takes place, but never to above 110° F., which does not cause intermittent working, nor is the yield of ozone affected by this temperature.

4. No attempt has ever been made to remove oxidised nitrogen products before employing the ozonised air, it being believed that with the Yarnold generator no such products are generated. Experiments have been made by an able and experienced chemist, which bears out this belief.

5. As regards the percentage of oxygen in air converted into ozone, the installation is so arranged as to produce a variable percentage, as may be desired. It is possible to work at anything from $\frac{1}{2}$ to 5 per cent. At the Commercial Ozone Works it is calculated that 1 per cent. of the oxygen is converted into ozone.

6. The advantages of the Yarnold ozone generator are considered to be—

1. Best efficiency in conversion of oxygen into ozone, in ratio to the electric current used.
2. Utility, as running continuously and with minimum attention.
3. Low wear and tear.
4. Cheapness of construction.

With reference to the remarks of Mr. W. F. Reid and of Mr. Otto Hehner, as to the prohibitive cost of ozonised air for blown or oxidised oils, it seems to have been considered that the oxidation is effected solely by ozone, whereas it is, what might be termed, "aerio-ozonic" oxidation. The unozonised oxygen contained in the air passed through the generator would also combine with the oil in the usual manner, and assist largely in the oxidation. As, however, it has been certified by such experts as Dr. Lewkowitsch, Mr. Alfred Allen, and Mr. G. H. Hurst, that results are obtained very quickly and much more speedily than by existing methods, it was concluded that the use of ozonised air would be of commercial advantage. The ozone contained in the ozonised air would appear to produce changes in the oil in a very short time, so modifying it as to make it capable of more rapidly absorbing ordinary oxygen; thus a final result is quickly arrived at. It may here be remarked that, with reference to the cost of ozone from the application point of view, it is not fair to judge it generally from the weight of active oxygen, as, in some cases, the whole molecule is absorbed. Again, the remarks of Mr. G. H. Hurst apply very strongly in many cases, and in this connection it should not be forgotten that *no after processes are required*, as is the case with nearly all known oxidants, and which are heavy elements of cost.

In reply to Mr. C. Sevin relative to palm oil, it has been found that the melting points remained constant on remelting, at the figures given.

In reply to Mr. Grant Hooper, it is found that the linseed oil treated by ozone is increased in density at a lower iodine absorption. The investigations made by Dr. Lewkowitsch, Mr. Allen, and Mr. Hurst show that the drying qualities are in no way impaired.

In reply to Dr. Martini as to the effect on "stinkers," no leakage is found to result. As regards the cost of bleaching yarns, it was stated that this process is only as yet in an experimental stage, and although very promising results have been obtained the cost cannot at present be definitely given.

I can quite understand that a certain amount of scepticism exists amongst even very experienced chemists as to the results of the practical use of ozone such as have been described by me, for probably no one in a laboratory has been able to produce ozone in such large volumes. Mr. R. T. Yarnold, who very ably carries out the practical work on which my syndicate is engaged, and who is a good practical chemist, will be happy to show our processes to any gentleman introduced by the Chairman of the London Section of the Society. I may here say that I am largely indebted to Mr. R. T. Yarnold for materials on which my paper was based, and who has assisted me in my replies to the points raised by the discussion, for I regret I am neither a chemist or an electrician.

*See under* XX., *page* 1173.

## AN IMPROVED APPARATUS FOR THE ESTIMATION OF CARBONIC ACID IN MINERALS, &c.

BY ARTHUR MARSHALL.

As gravimetric methods for the estimation of carbonic acid are not suitable for technical purposes, it is usual to measure the volume of carbon dioxide evolved in a suitable apparatus. Scheibler's apparatus is the one most employed. A description of it may be found in most text-books on quantitative analysis (Fresenius, Quant. Anal., 7th ed., pp. 343—346). In it the substance is decomposed inside a glass bottle by the action of 10 c.c. of hydrochloric acid of 1·12 sp. gr. (24 per cent. HCl). The gas generated expands a rubber bladder placed inside a second closed bottle, and this in turn forces air into a graduated tube, where it is measured over water. This volume must then be corrected for temperature and pressure, and a further correction made for the carbon dioxide, about 3·2 c.c., which remains dissolved in the 10 c.c. of acid. The apparatus works quickly and easily, but is open to the following objections:—

1. A considerable and unknown volume of gas remains dissolved in the acid.

2. The corrections for temperature and pressure involve troublesome calculations.

3. Readings have to be taken of the thermometer and barometer.

4. The total volume of the generating and measuring apparatus is considerable—about 600 c.c. Consequently, if the atmospheric temperature or pressure vary during the experiment, a large error may be introduced; *e.g.*, if the temperature rise 1° C. (which may easily happen) the reading of the volume will be about 2 c.c. too high.

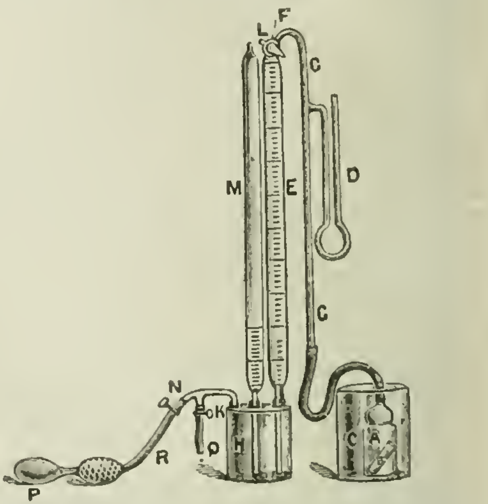
Wigner (Analyst, 1876, p. 158; 1879, p. 228) advocated displacing the last traces of carbon dioxide by boiling the liquid.

Lunge (this Journal, 1891, 658; Zeits. f. angew. Chem. 1891, 229—235) proposed a method in which the last traces of carbon dioxide are swept into the measuring tube by generating hydrogen at the same time. The volume of gas generated is measured, the carbon dioxide is absorbed by potash, and the diminution of volume ascertained.

Gantter (Zeits. Anal. Chem. 1893, p. 300) recommended the determination of the volume of the carbon dioxide by measuring it in his gas volumeter over concentrated calcium chloride solution.

For further papers on the subject of previous forms of calcimeter, see Zeits. fur analyt Chem. **6**, 398; **23**, 371; **27**, 721: **26**, 337; J. fur prakt. Chem., new series, **35**, 86.

My apparatus consists of a gas reduction tube M, and a measuring tube E, which both pass though corks to the bottom of the Woulff's bottle H, which is so fitted that the pressure of air in it can be accurately adjusted. It contains some refined petroleum oil of high boiling point, which can be forced into the tubes M and E. M contains such a quantity of air that, if it were reduced to 0° C. and 760 mm. pressure, it would just fill the tube down to a certain mark on it. The tube E is graduated in cubic centimetres, and is fitted at the top with a three-way cock of special design, by means of which it can be brought into communication either with the atmosphere or with the tube G, which leads to the generating vessel A. Branching out of G is the mercury manometer D, which enables one to adjust the pressure inside A, G, and E till it is equal to the atmospheric pressure. The generating vessel A is fitted with a well-ground tubulated stopper, and contains a small glass tube B to hold the acid. It is inserted in the glass water-bath C, which should contain cold water.



Briefly stated, an estimation is conducted as follows:—The carbon dioxide is generated by the action of a small volume, 1 c.c., of concentrated hydrochloric acid on a weighed quantity of the substances to be tested; 0·1 to 0·8 or more grms. should be taken, according to the percentage of carbonic acid it contains. A mixture of air and carbon dioxide passes over into the measuring tube E. When the action is complete, the pressure is adjusted, till the manometer D shows that it is equal to that of the atmosphere. The cock is then turned off and the pressure is again adjusted till the liquid stands in M at the highest graduation. The volume in E is then read off. This, without any correction whatever, is the volume of carbon dioxide contained in the substance taken. The whole operation does not take more than 10 to 15 minutes.

Whilst one determination is proceeding, a fresh quantity of material may be weighed out. As soon as the first determination is complete, E is brought into communication with the atmosphere and the petroleum is forced to the top of B. The cock is then turned to bring G into communication with the atmosphere. If the first pressure adjustment was properly carried out, the mercury in D will not move when this is done. A and B are then removed and washed out and wiped. The second quantity of substance weighed is introduced into A, and 1 c.c. of concentrated hydrochloric acid is measured into B. The stopper is inserted into A, and the cock is turned to bring G and E into communication. The bottle A is then tipped up till the acid runs into the substance, A is immersed in the water-bath, and the process is completed as described above.

The gas reduction tube E acts on the same principle as that in Lunge's well-known "gas volumeter." (This Journal, 1890, 547; Ber. 1890, No. 4; Zeits. Ang. Chem. 1890.) To give absolutely accurate results the level of liquid in M and E should be the same. The density of the petroleum is, however, so small that the error from this cause never amounts to more than 0·3 c.c. with an apparatus having the dimensions which I have selected.

Carbon dioxide is slightly soluble even in heavy petroleum oil, but the solution proceeds very slowly. In the case of my apparatus, if my instructions be followed, only a dilute mixture of carbon dioxide can come in contact with the petroleum. The error due to this cause therefore falls well within the limits of experimental error due to other causes.

The error due to the solubility of carbon dioxide in hydrochloric acid is reduced to a minimum by employing a small quantity of concentrated acid; using 1 c.c. of acid of 1·17 sp. gr. it does not amount to more than 0·5 c.c. This error is in an opposite direction to that due to the inequality of the levels in the tubes M and E. Consequently it is to a great extent neutralised by the latter. Concentrated hydrochloric acid dissolves less carbon dioxide than the same volume of dilute acid.

I find that if the generating vessel A be not kept cool a notable quantity of hydrogen chloride is expelled from it, and is slowly reabsorbed as the apparatus cools down again. This, of course, would interfere with the accuracy of the process. During the action the vessel should, therefore, be kept immersed in cold water. The cold water-bath also tends to prevent the temperature of the generating apparatus varying to any perceptible extent. Any error due to the latter cause is, in addition, greatly reduced by the small volume of the generating apparatus, which is not more than 100 c.c.

It is said that india-rubber and gutta-percha dissolve considerable quantities of carbon dioxide. (Fresenius, Zeit. Anal. Chem. 1889, p. 78.) I have therefore dispensed with these substances as far as possible. Instead of a gutter-percha tube, I use a small glass tube to contain the acid.

The following are the chief advantages of the apparatus described:—

1. The quantity of carbon dioxide dissolved in the acid is reduced to a minimum by using a small quantity of concentrated acid.

2. No corrections have to be made for temperature and pressure; consequently no reading of thermometer or barometer need be taken.

3. The total volume of the generating and measuring apparatus being less than 100 c.c., and the generating vessel being immersed in a considerable quantity of cold water, the volume of the air inside it cannot change during a determination to an extent sufficient to introduce a perceptible error.

4. The apparatus is quite simple, and although no barometer or thermometer is required the results are considerably more accurate than those obtained with Scheibler's.

I must, in conclusion, thank Mr. Arthur Webster for his assistance in connection with the elaboration of the instrument.

### Discussion.

Mr. W. F. Reid inquired whether the percentage of carbon dioxide absorbed by the petroleum had been actually tested.

Mr. A. Marshall replied that the body was of course somewhat soluble. The solution proceeded slowly, and with the apparatus he had designed only very dilute carbon dioxide came into contact with the petroleum, and it could not have more than a very imperceptible action, as the experiment did not take more than 10 to 15 minutes.

## ACTION OF AN ACETYLENE GAS FLAME ON PLATINUM.

BY ILTYD J. REDWOOD.

As the laboratory was lighted by acetylene gas the writer designed a Bunsen burner for use with this gas, with a view to doing away with the use of "spirit" burners.

After the acetylene burner had been in use a short time it was discovered that the platinum crucible had gained slightly in weight, and that its bottom very much resembled the appearance of new galvanised iron. The crucible was now carefully watched, but did not change in either weight or appearance until one day, after a long (about two hours) heating, it was noticed to give off a vapour from the bottom when removed from the flame. An examination showed that the bottom had entirely changed, and, instead of a galvanised appearance, it now had a somewhat spongy appearance, and a close inspection revealed the presence of a number of small cracks that extended right through the metal, while two or three small beads hung on the outside, and a little ridge had formed on the inside and extended some distance across the bottom.

The crucible had not been heated in a muffle, and at no time had its temperature been higher than that indicated by a cherry-red heat. The precipitate placed in the crucible this last time was oxalate of lime.

These few facts are given with the hope that they may result in preventing other platinum apparatus meeting with a similar fate.

### Erratum.

## THE CHEMICAL AND PHYSICAL CHARACTERS OF SOME INDIAN EDIBLE OILS.

BY ARTHUR W. CROSSLEY, M.SC., PH.D., AND
HENRY R. LE SUEUR, B.SC.

(*This Journal*, 1898, 989—991.)

On p. 990, col. I, par. 6, line 4, the number of the page is incorrect. For 82 read 988.

# Manchester Section.

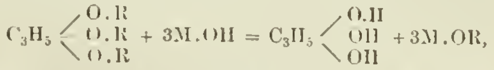
*Meeting held on Friday, December 2nd*, 1898.

DR. WATSON IN THE CHAIR.

## THE THEORY OF SAPONIFICATION.

BY DR. J. LEWKOWITSCH.

The reaction taking place when a triglyceride (*i.e.*, a fat or a fatty oil) is saponified is generally expressed by the following equation:—



$$C_3H_5 \begin{cases} O.R \\ O.R \\ O.R \end{cases} + 3M.OH = C_3H_5 \begin{cases} OH \\ OH \\ OH \end{cases} + 3M.OR,$$

where R denotes the radicle of a fatty acid, and M a monovalent metal or hydrogen.

This equation represents, however, only the final conditions and does not answer the question as to the manner in which the chemical change actually takes place, *i.e.*, whether the triglyceride is broken up directly into three molecules of free fatty acids and one molecule of glycerol, or whether there is a gradual splitting off of the acid radicles, the triglyceride passing, first, through the stage of the diglyceride, then of the monoglyceride, until finally the formation of glycerol is reached.

In the former case the chemical change would be an example of a tetramolecular reaction (of the 4th order) whereas, in the second case, two products only being obtained in each stage, a bimolecular reaction (of the 2nd order) would take place.

Until recently, the former opinion was held, but last year Geitel (Jour. f. prakt. Chemie, 1897, [55], 429) has shown that the latter explanation would be the correct one. For if the reaction is a bimolecular one, then the law of mass action would demand that the differential equation, applicable to chemical changes of this kind, viz.:

$$\frac{dx}{dt} = K(A - x)(B - x)$$

(where K denotes a constant) should hold good for each stage. And, indeed, Geitel has proved in the case of triacetin, when treating it with dilute acid, and, further, in the case of cotton seed oil, when digesting it in the cold with an equimolecular amount of alcoholic potash under suitable conditions, that the experimental values obtained for K were in practically satisfactory agreement with those postulated by theory.

I have explained elsewhere (Jahrbuch der Chemie, VII., 367 ff.) the importance of this theory for the chemistry of fats and oils and its bearings on their analysis; and I likewise pointed out, that I had instituted some experiments on partially saponified fats which, at that time, appeared to lead to conflicting results.

The difficult mathematical calculations given by Geitel will hardly appeal to every practical soap- or candlemaker, who wishes to gain a scientific insight into the principles underlying his technical operations, and he would certainly prefer to have demonstrated the, however transitory, existence of diglyceride and monoglyceride, and to have them shown up, as it were, in flesh and blood.

For the purposes of this communication, I wish to place myself in the position of the practical man who saponifies the fats on a large scale, either in a soap pan by means of aqueous caustic alkalis, or in an open vat by means of lime. Therefore, experiments with alcoholic solutions of saponifying agents or others, where an amount of alkali insufficient for complete saponification is used, are meanwhile left out of consideration.

If the recent views are correct, then samples taken at intervals from the pan or vat, whilst saponification is progressing, would represent mixtures of (1) unsaponified triglyceride; (2) diglyceride; (3) monoglyceride; (4) glycerol; (5) soap (either soda or lime soap); (6) free alkali; and (7) water. The removal of the last four substances would be comparatively easy, but the resolving of the mixture of the first three would obviously be a difficult task—although their different solubilities in alcohol might afford a method for approximate separation, as suggested by Geitel.

Fortunately the quantitative methods practised in fat analysis furnish a clue which leads to the satisfactory solution of the problem, especially the determination of the acetyl value (this Journal, 1897, 503) lends itself admirably to this purpose.

My experiments were carried out in each case on several pounds of fat (tallow, cottonseed oil). This was saponified either by caustic soda or caustic lime in a manner simulating completely the operations on a large scale. At certain intervals samples were withdrawn, whilst the remaining mass underwent further saponification. The samples were treated with mineral acids, &c., so as to obtain a fatty mixture free from glycerol, alkali, and water.

The progress of saponification was ascertained by the acid values of these samples (proportion of free fatty acids).

Now, supposing we had to deal with pure tristearin, then, according to the old views, all the samples should be mixtures of tristearin and stearic acid and their acetyl value should be = *nil*, whereas, according to the new theory the samples would contain varying amounts of distearin and monostearin as well. A measure of their proportion would be furnished by the acetyl values of the samples, as will be readily seen from the following table:—

| Substance. | Formula. | Acetyl Value. |
|---|---|---|
| Tristearin.......... | $C_3H_5(OC_{18}H_{35}O)_3$ ............ | 0·0 |
| Distearin .......... | $C_3H_5(OH)(OC_{18}H_{35}O)_2$ ...... | 84·2 |
| Monostearin ....... | $C_3H_5(OH)(OH)(OC_{18}H_{35}O)$.. | 253·8 |
| Stearic acid ........ | $C_{18}H_{36}O_2$........................ | 0·0 |

If, as in my experiments, the tallow and the cottonseed oil had certain acetyl values to begin with, then according to the old theory all the samples should have the same acetyl value, whereas the new theory would demand varying acetyl values, rising at first to a maximum then to fall again to the acetyl value of the original fat. In other words, if we plot the acid values as abscissae and the acetyl values as ordinates we would obtain in the former case a straight line, and in the latter a curve.

In nearly all my experiments curves were obtained, corresponding in a general way to the postulates of the new theory, notably so in the case of cottonseed oil saponified with lime—the highest acetyl value being 58,—and in the case of tallow saponified with caustic soda—the highest number being 65—whereas the acetyl values of the original fats were 12 and 6 respectively.

These results speak unmistakably in favour of the new theory.

Additional proof was obtained by determining the amount of insoluble fatty matter—Hehner value—of the *acetylated* samples, after complete saponification, as also the amounts of alkali required for their complete saponification, *i.e.*, their saponification values.

The following table, taking again tristearin as an example, shows that although the indications furnished by the Hehner and saponification values have not so decisive a character as the acetyl values, still, according to the old theory, the Hehner values could only vary between 95·73 and 100 (Nos. 1 and 6) and the corresponding saponification values between 189·1 and 197·5, whereas in the light of the new theory much greater variations must be expected (Nos. 4 and 5 of the table). Now, in the case of cottonseed oil, whose values would not differ considerably from those calculated for tristearin, the experimental numbers led to curves which were parallel to the acetyl value curves for the saponification values, and ran in the opposite directions for the Hehner values. It is evident that it could not be otherwise, if the samples taken from the pan did contain diglycerides and monoglycerides.

| No. | Substance. | Formula. | Hehner Value. | Saponification Value. |
|---|---|---|---|---|
| 1 | Tristearin ........................ | $C_3H_5(O.C_{18}H_{35}O)_3$ .......................... | 95·73 | 189·1 |
| 2 | Distearin ......................... | $C_3H_5(OH)(O.C_{18}H_{35}O)_2$........................ | 91·02 | 179·8 |
| 3 | Monostearin ...................... | $C_3H_5(OH)(OH)(O.C_{18}H_{35}O)$ ............ | 79·33 | 156·7 |
| 4 | Monoacetyldistearin ............... | $C_3H_5(O.C_2H_3O)(O.C_{18}H_{35}O)_2$........... | 85·3 | 252·7 |
| 5 | Diacetylmonostearin................ | $C_3H_5(O.C_2H_3O)_2(O.C_{18}H_{35}O)$ ........... | 64·2 | 380·8 |
| 6 | Stearic acid....................... | $C_{18}H_{36}O_2$............................................ | 100·0 | 197·5 |

The experiments briefly outlined here will be given fully in another paper, but incomplete as they are they leave no doubt that the saponification of fats takes place in stages.

### DISCUSSION

Dr. T. MARCHLEWSKI thought it rather surprising that the saponification of glycerides should take place in accordance with the simple differential equation of the second degree, especially in the face of the fact that, according to the ingenious researches of the lecturer, the saponification of the triglycerides took place gradually in three different stages. He thought the saponification of glycerides might be compared to a certain degree with the hydrolysis of complicated hydrocarbons, in which latter case, according to his own experience, the simple equation was not quite satisfactory, a circumstance foreseen by theory. As to the isolation of the various saponification products, he would suggest the use of benzene—or toluene sulphochlorides which reacted remarkably easily with hydroxylated compounds and yielded as a rule condensation products which crystallised exceptionally well.

Mr. ALLEN remarked that the author had not referred to an abstract which had appeared in the Journal by a German chemist, in which it was shown that supposing only a fractional part instead of the full amount of caustic soda was used, it was possible in that case to obtain complete hydrolysis. By using only about 10 to 15 per cent. it would of course preclude the possibility of using alcoholic solutions. He wished to know if Dr. Lewkowitsch's curves had been obtained from one sample only, or more than one, inasmuch as his curves agreed with each other. He took it that Dr. Lewkowitsch had dealt with one sample only, otherwise he would not have obtained such agreement. It was well known that it was almost impossible to get two soap pans in complete agreement, so that he thought it would be hardly possible for Dr. Lewkowitsch to have obtained similar results with different samples.

Dr. **Lewkowitsch** thought the remarks which Dr. Marchlewski had made were very pertinent indeed, as the reaction which he mentioned was very similar to the saponification of fats, both reactions being nothing else but hydrolysis, such hydrolysis taking place in stages. In order to have the mathematically expressed law proved, homogeneous solutions and molecular proportions should be taken which would allow the course of hydrolysis to be studied closely, but as these ideas were entirely new to a practical man he had placed himself in the position of a practical man and renounced altogether at the outset theoretical views and proceeded in the usual way of saponifying on a large scale, taking samples from time to time and examining them as to the progress of hydrolysis. With reference to Mr. Allen's remarks, no doubt he referred to Dr. Henriques' paper abstracted in the Journal. To begin with, there was nothing new in that paper, as the same observation had been fully described by Mr. Duffy in the year 1852, before the Chemical Society in London; but the facts explained in these papers had nothing whatever to do with the question, because in that case hydrolysis also proceeded in stages just as in the case dealt with in the paper. There was simply an exchange of the acid radicle with the $C_3H_5$ radicle, and the presence of water was absolutely necessary in order to let that reaction take place which was described in the papers of Duffy and Henriques. The curves were of course obtained on one and the same sample as samples taken at different times would represent different stages.

He had just touched the great difficulty which presented itself in an attempt to isolate the mono and diglycerides. Perhaps one might succeed by treating the mixture of triglycerides, diglycerides, and monoglycerides with alcohol and base a process of separation on their different solubilities. Another way might be to apply the re-agent which Dr. Marchlewski had proposed, but he could not conceal from himself the great difficulties one would find, and in view thereof he had proceeded in the way he had explained by examining the mixtures and seeing whether such values could be obtained as would favour the view that saponification took place in stages. That such values were obtained was clearly shown by the curves. Altogether the subject was a very difficult one and might be recommended to anyone who wished to study a complicated problem of chemical dynamics.

## HYDROSULPHITE OF SODA, AND ITS USE IN INDIGO DYEING.

BY J. GROSSMANN, PH.D.

The discovery of hydrosulphites may be dated back as far as 1718, when Stahl noticed that iron dissolved in sulphurous acid. This was confirmed by Berthollet in 1789, and a similar reaction was obtained with zinc and tin by Fourcroy and Vauquelin. But we may take it that until Schützenberger published his researches in 1869 the nature of the products thus obtained was not recognised, although Schönbein had found previously that they were possessed of strong reducing properties. Schützenberger (C. r. 69. p. 196) assumed that the reactions took place as follows:—

(1.) $Zn + 2SO_2 + H_2O = ZnSO_3 + H_2SO_2$

(2.) $Zn + 3NaHSO_3 = Na_2Zn(SO_3)_2 + NaHSO_2 + H_2O$.

Bernthsen (Ber. 14. 438; Annalen 208; 161) proposes the equation:

(3.) $Zn + 4NaHSO_3 =$
$ZnSO_3 + Na_2SO_3 + Na_2S_2O_4 + 2H_2O$.

Later on (Ann. 211; 300) Bernthsen gives another equation—

(4.) $3\ Zn + 10NaHSO_3 =$
$Zn_3Na_2S_8O_{10} + Na_2SO_3 + 3Na_2S_2O_4 + 5H_2O$.

As far as the left side of the equations 2 and 4 is concerned, there is so little difference between them that they are almost identical, but the formula for the resulting hydrosulphite which these two chemists assume respectively is very different, the one taking it as $NaHSO_2$, the other as $Na_2S_2O_4$. Leaving, however, theoretical speculations for the present on one side, we find that as regards practical results there is very little difference between these two chemists. For we have:—

(5.) $NaHSO_2 + O = NaHSO_3$

(6.) $Na_2S_2O_4 + O = Na_2S_2O_5$

Therefore—

According to Schützenberger's equation (2) $3SO_2 = O$.

According to Bernthsen's equation (4) $3\frac{1}{3}SO_2 = O$,

showing only a difference of 10 per cent. in the practical reducing results, whichever of these equations we may adopt. At present I incline to Schützenberger's formula and equation. According to Bernthsen's equation the product resulting would be a neutral salt $Na_2S_2O_4$; this is against all experience. The compound which results from the action of zinc on bisulphite is an acid salt and I have found that the acidity corresponds to that of an acid salt of a two-basic acid. I arrived at this result in the following manner:—The liquor which resulted from the action of zinc on bisulphite was on the one hand tested for reducing power, calculated on $NaHSO_2$; on the other hand it was ascertained how much $Na_2O$ it required to become neutral, *i.e.*, just to show a precipitation of zinc; and the following figures were obtained:—

| | |
|---|---|
| $NaHSO_2$ | 13·4 grms. per litre. |
| $Na_2O$ | 5·27 grms. per litre. |

If we divide these figures by the respective equivalents, *viz.*, 88 and 31, we obtain the ratio 0·15 to 0·17, which is near enough to prove that we have a two-basic acid in which one half of the hydrogen is replaced by sodium.

Bernthsen himself found the ratio between Na and S. by analysis as represented by his formula; if we take it that he had the acid salt and overlooked the two atoms of H, his formula would be actually $Na_2H_2S_2O_4 = 2NaHSO_2$, identical with Schützenberger's formula. And if the ratio between Na and S be as found by Bernthsen, which I do not doubt, it would be proof that the formula for hydrosulphurous acid must be $H_2SO_2$, for it could not be $H_2SO$, for this formula would not agree with the practical reducing results of the hydrosulphites, in that it would require double the amount of oxygen. Neither could it be $H_2SO_3$ or $H_2SO_4$, which would be the only other possible formulae for it.

If we then adopt Schützenberger's formula and equation, according to equation 2, 3 molecules of sodium hydrogen sulphite should produce one molecule of acid sodium hydrosulphite; or expressed differently three molecules of sulphurous acid should, when converted into hydrosulphite, be capable of reducing one molecule of indigotine. Taking commercial bisulphite of soda of 60° Twaddle to contain about 20 per cent. total $SO_2$, *i.e.*, to contain 2·6 lbs. total SO per gallon, and assuming all this to be in the state of sodium hydrogen sulphite, one gallon of bisulphite should reduce 3·53 lbs. indigotine. The actual yield is far short of this viz.—from 1 lb. to $1\frac{1}{3}$ lbs. of indigotine; *i.e.* only from 30 to 40 per cent. of the quantity which should be obtained according to eq. (2.) This may be due to partial oxidation, or to the fact that some more complicated reactions take place at the same time. But assuming equation (2) to be correct there is another loss inherent to the reaction as shown there; *i.e.*, the loss caused by the quantity of neutral sulphites formed; for $\frac{2}{3}$ of the total sulphurous acid is converted into the sodium zinc sulphite. Bernthsen already tried to recover these inactive compounds and reduce this loss by adding hydrochloric or acetic acid, but failed in his object, nor have I succeeded with these acids. But I found that in using sulphuric acid in not too great a quantity and by adding it gradually and slowly a yield could be obtained which, though not near the quantitative result, would be three times as great as the actual yield obtained from the old process, using the same proportions of zinc and bisulphite. This agrees very well with the equation :—

$$(7.)\ 3Zn + 3NaHSO_3 + 3H_2SO_4 = 3ZnSO_4 + 3NaHSO_2 + 3H_2O.$$

As there appears to be a good deal of misunderstanding regarding my process, it may be useful to follow more closely this equation It is clear that according to our present views no free sulphurous acid could practically exist along with neutral sodium sulphite in excess. Thus by adding a fractional equivalent of $H_2SO_4$ to one equivalent of $Na_2SO_3$ sodium hydrogen sulphite would be formed as follows :—

$$Na_2SO_3 + \frac{1}{m}H_2SO_4 = \frac{1}{m}Na_2SO_4 + \frac{2}{m}NaHSO_3 + \left(1 - \frac{2}{m}\right)Na_2SO_3.$$

Suppose now in making hydrosulphite the reaction had taken place according to equation (2), we should have as the resulting product besides hydrosulphite and water, one equivalent of $Na_2Zn(SO_3)_2$. If we add acid to this we may take it that the following reaction takes place :

$$Na_2Zn(SO_3)_2 + H_2SO_4 = ZnSO_4 + 2NaHSO_3$$

So that we have again bisulphite regenerated which may be acted on by zinc and in this case would take $\frac{2}{3}$ of an equivalent of zinc. In this case again $Na_2Zn(SO_3)_2$ would form, but only $\frac{2}{3}$ of an equivalent ; following up what will happen on adding Zn and sulphuric acid gradually and successively to the original quantity of zinc sodium sulphite formed, we have—

$$\left\{\begin{array}{llll} & Na_2Zn(SO_3)_2 + & H_2SO_4 & + \frac{2}{3}Zn = \\ \frac{2}{3} & A & + \frac{2}{3}\ B & + (\frac{2}{3})^2C = \\ (\frac{2}{3})^2 & A & + (\frac{2}{3})^2B & + (\frac{2}{3})^3C = \\ \cdots & \cdots & \cdots & \cdots \\ (\frac{2}{3})^{n-1} & A & + (\frac{2}{3})^{n-1}B & + (\frac{2}{3})^nC = \end{array}\right.$$

$$\left.\begin{array}{lllll} \frac{2}{3} & Na_2Zn(SO_3)_2 + & \frac{2}{3}\ NaHSO_2 + & \frac{2}{3}\ H_2O + & ZnSO_4 \\ (\frac{2}{3})^2 & A & + (\frac{2}{3})^2\ D & + (\frac{2}{3})^2\ F & + \frac{2}{3}\ F \\ (\frac{2}{3})^3 & A & + (\frac{2}{3})^3\ D & + (\frac{2}{3})^3\ E & + (\frac{2}{3})^2F \\ \cdots & \cdots & \cdots & \cdots & \cdots \\ (\frac{2}{3})^n & A & + (\frac{2}{3})^n\ D & + (\frac{2}{3})^n\ E & + (\frac{2}{3})^{n-1}F \end{array}\right\}$$

If we add this series up, we find the sum of $Na_2Zn(SO_3)_2$ which we may call $M = \left\{1 + \frac{2}{3} + (\frac{2}{3})^2 + \ldots + (\frac{2}{3})^{n-1}\right\}$

We have then as the summation of these equations

$$M.\ Na_2Zn(SO_3)_2 + M.\ H_2SO_4 + (M - 1)\ Zn =$$
$$(M - 1)Na_2Zn(SO_3)_2 + (M - 1)NaHSO_2 + (M - 1)\ H_2O + M.\ ZnSO_4.$$

As M is equal to 3, the equation is—

$$3Na_2Zn(SO_3)_2 + 3H_2SO_4 + 2Zn =$$
$$2Na_2Zn(SO_3)_2 + 2NaHSO_2 + 2H_2O + 3ZnSO_4.$$

or—

$$Na_2Zn(SO_3)_2 + 3H_2SO_4 + 2Zn =$$
$$2NaHSO_2 + 2H_2O + 3ZnSO_4$$

add to this

$$Zn + 3NaHSO_3 = Na_2Zn(SO_3)_2 + NaHSO_2 + H_2O$$

and the result is, as before.

$$3NaHSO_3 + 3Zn + 3H_2SO_4 = 3NaHSO_2 + 3H_2O + 3ZnSO_4.$$

This equation seems to me to confirm Schutzenberger's views, for if we were to accept Bernthsen's formula, and apply our principle to his equation we should have—

$$2Zn + 4NaHSO_3 + 2H_2SO_4 = 2ZnSO_4 + 2Na_2S_2O_4 + 4H_2O,$$

*i.e.*, the reducing power could only be doubled by the addition of sulphuric acid, and not trebled as I have found in agreement with Schutzenberger's formula.

In order to test this thoroughly a large number of experiments were carried out on a large scale, side by side, the same amount of zinc and bisulphite was used in each case with this difference only, in the one series of experiments only zinc and bisulphite were used and in the other series of experiments the same proportions of zinc and bisulphite were used with a gradual addition of $H_2SO_4$. It was then found that taking the reducing power of 1 gallon of bisulphite of soda in the old way as 1, that produced by my process would be equal to 2·7, which is near enough for all practical purposes as 1 to 3. A series of experiments were carried on, taking $1\frac{1}{2}$ lbs. zinc to 1 gallon of bisulphite, which is a favourite proportion for making hydrosulphite by the old process. The results were as follows :—

Reducing power per gallon of bisulphite calculated on indigotine, 1·16 lbs. indigotine old process, 3·13 lbs. indigotine, new process.

As we see from Schützenberger's equation and from my own the hydrosulphite produced is in the state of an acid salt which is very unstable, much more so than the neutral salt ; moreover, it does not reduce indigo in the acid state. For these reasons Schutzenberger proposed to convert it into the neutral salt by adding lime.

I have been trying to find further particulars as to how the reaction of converting the acid salt into the neutral one by means of lime was supposed to take place, but I have been unable to find any statements on that point. Now if we put down the equation

$$2NaHSO_2 + CaO = Na_2Ca\ (SO_2)_2 + H_2O,$$

we find that the only way in which the reaction could take place would be by forming a sodium calcium compound, but as the filtered liquor never contains any calcium it is clear that the reaction cannot take place in that way. The explanation which I give for this reaction is as follows, there is always a large quantity of sulphite of soda present which is easily converted into caustic soda by lime so that the reaction which really takes place is as follows :

$$Na_2SO_3 + CaO + H_2O = CaSO_3 + 2NaHO$$
$$2NaHSO_2 + 2NaOH = 2Na_2SO_2 + 2H_2O.$$

I shall have to refer again to this later on.

We may now leave the hydrosulphite as far as its manufacture and chemical properties are concerned and give our attention to its use in indigo dying. At the time when Schutzenberger proposed the hydrosulphite vat, there was in use besides the fermentation vats which do not enter into our speculations, two other vats. That is the copperas lime vat and the zinc lime vat, both these vats were well known to have certain disadvantages. For instance there is a large quantity of sediment in the copperas vat, about 8 lbs for every pound of indigotine reduced and a considerable amount of indigo was rendered inactive by being carried into the precipitate as an insoluble compound. The zinc vat was found to be not easily managed and there is no doubt that a considerable amount of indigotine is destroyed by the action between zinc and lime or caustic soda. It may be generally stated that there is no means not even by hydrosulphite of reducing indigo to indigo white quantitatively, there is always a loss, and I am at present engaged in a series of experiments determining the relative losses by different reducing agents.

As far as my results in the laboratory go I find there is certainly 10 per cent. more loss using zinc as a reducing agent than in using hydrosulphite, and my experience in practical work on a large scale quite confirms this. Both the copperas and the zinc vat require considerable time for reducing and settling, whereas the hydrosulphite vat is quick in its action, the consequence is that whereas only a limited amount of work can be got out of a copperas or zinc vat, a hydrosulphite vat can be used continuously, almost without stopping, and if it were used night and day, it would produce quite four times the quantity of dyed goods which either of the other vats could yield. It would therefore require less plant and the general charges incidental would be only $\frac{1}{4}$th of those of any other vat.

As the vat can be easily regulated the work will be more even, and the apparatus as shown before will take up less room for a given quantity of work. There will be less indigo lost either through over reduction or in the shape of insoluble compounds, and this vat, properly managed, should therefore be the cheapest vat for dyeing indigo.

Such must have been Schützenberger's views when he proposed this vat, and if his directions had been followed, there is no doubt that this vat would have replaced every other vat for cotton dyeing in this country. Unfortunately there is hardly a hydrosulphite vat in this country, though no doubt most dyers who make hydrosulphite from Bisulphite and zinc imagine they have a hydrosulphite vat.

The usual way to prepare what is now considered a hydrosulphite vat is to mix bisulphite of soda and zinc in a bucket or tub, and when the mixture becomes warm, to add it to the indigo with either lime or caustic soda.

The proportions used are from 1 to 3 lbs. zinc dust to the gallon of bisulphite; as, however, at its best, only one ½ lb. of zinc can go into solution, it follows that from ½ to 2½ lbs. of free zinc dust will go into the vat for every gallon of bisulphite which is used, so that we really have not a hydrosulphite vat, but what we might call a hybrid zinc hydrosulphite vat.

The loss by over reduction is, therefore, not much less than in a zinc vat, and the amount of sediment greater. It speaks well for the character of the hydrosulphite vat, that even under these circumstances, it should have maintained its place amongst other vats in this country. I may here also point out another fallacy which prevails with regard to the hydrosulphite vat; it is generally considered that by using soda in reducing the indigo a soda vat is formed and by using lime a lime vat. But there is no such thing as a lime hydrosulphite vat. I have shown on a previous occasion that the large amount of sulphite which is always present with hydrosulphite becomes decomposed by lime and that caustic soda is formed. It is therefore clear that every lime hydrosulphite vat contains free caustic soda as well, and is in reality a mixed soda lime vat.

We may take it then that the hydrosulphite vat has not received a fair trial in this country, as it has not been carried out according to Schützenberger's ideas, and that its application could advantageously be modified in many ways. Take for instance the way in which yarn is dyed at present in a series of vats of different strengths, and which vats have to be emptied pretty frequently. There is no reason why with a clear hydrosulphite such vats should not be worked continuously in such a manner that as much indigo as taken out by the yarn, should be replaced before the next lot of yarn was dyed up in it. There would be a considerable saving in labour, in general charges, and in the room required for the plant; this is only one of a number of instances which might be given of the advantage of using a clear hydrosulphite for cotton and yarn dying. In a future paper I will deal more fully with wool dyeing and other points relating to hydrosulphite.

# Newcastle Section.

*Meeting held on Thursday, November 17th, 1898.*

MR. JOHN PATTINSON IN THE CHAIR.

## MICROSTRUCTURE OF ALLOYS.—PART III.

BY J. E. STEAD, F.I.C., F.C.S.

### ANTIMONY AND TIN.

In the previous papers of the writer, attention was drawn to the fact that the eutectic of antimony and tin, containing about 7½ per cent. of antimony, was different from the eutectic of lead and antimony, inasmuch as it was composed of a compound which did not consist of juxtaposed plates of the two constituents.

On treating this eutectic with dilute hydrochloric acid a black amorphous powder of exceeding fineness was left insoluble, and this, it was shown, contained tin, and was therefore not entirely antimony.

Since that time the black amorphous powder has been further studied, and on separating a sufficient quantity by the long-continued action of dilute acid, an analysis was made of it. The results obtained were as follows, *viz.*:—

| | Per Cent. |
|---|---|
| Tin | 48·56 |
| Antimony | 51·44 |

If the metals had been present in equal atomic proportions the antimony would have been 50·21 per cent. The excess of antimony, over and above that what calculation shows it should have been if it was a definite chemical compound, is accounted for by the assumption that a portion of it was decomposed, the antimony remaining behind, thus increasing the total proportion in the residue.

It was shown that when the antimony was increased above 7½ per cent. a definite compound in well-formed crystals separated out, having the composition of SbSn.

Mons. Charpy describes these crystals as cubes.

The faces are not bright enough to enable one to measure them by the goniometer, but on measuring them on the revolving stage of the microscope he, the writer, had proved that all the angles were 90°, and the six faces were equal to one another; they might conclude therefore they were true cubes.

Both tin and antimony crystallise in the rhombic system. The fact that the separated crystals above referred to are cubic and not rhombic would tend to support the view that they were not mixtures, but true combinations of the two metals.

On comparing this eutectic with the pearlite, or eutectic of iron and carbon, containing about 0·9 per cent. carbon, in which the mass is composed of laminæ of iron and a definite carbide of the formula $Fe_3C$, it has been shown that if the carbon is present in larger quantity than 0·9 per cent. the excess separates out as $Fe_3C$ in meshes, surrounding the grains of pearlite, and the quantity of this independent carbide steadily increases with the increase of carbon.

There is a certain resemblance between the compounds of iron and carbon and those of antimony and tin. The eutectic in both cases consists of a metal and a definite compound, and when the proportion of the latter is raised beyond a fixed definite point it separates in isolated masses, and is found in the cold alloy embedded in the ground mass of the eutectic.

There is this important difference, however, between the eutectics, for in steel properly annealed the iron and carbide are quite separate, and lie in more or less curved plates in juxtaposition, whereas the eutectic of antimony and tin appears to consist of a solidified solution or isomorphous compound of SbSn in an excess of tin; the crystalline structure of the eutectic appears to be the same as that of tin itself.

At the joining of the crystallite forms in the eutectic, however, there appears to be a little more antimonide than in the central portions, for on polishing they appear to be harder, and remain brighter, and on slightly etching with acid they become darker.

All the compounds of tin with less antimony than 7½ per cent. crystallise in the same forms, and behave in the same way under the treatment of polishing and etching, and so far as his own pyrometric investigations indicate, have only one solidifying point—a fact which points to the conclusion that the tin and the eutectic do not separate into two parts, as happens in the case of many binary alloys. It would appear, then, that all the alloys containing from 7½ per cent. antimony to practically none, consist of almost perfectly structurally homogeneous solidified solutions of isomorphous mixtures of tin and antimonide of tin.

An attempt was made to separate and analyse the crystals from the alloys containing between 25 per cent. and 50 per cent. antimony, but it was found that when the antimony exceeded 30 per cent. it is almost impossible to remove the eutectic from the closely crowded grains of the antimonide.

The crystals from alloys containing between 9 per cent. and 30 per cent. Sb, on analysis closely approximated to the composition of SbSn. The forms of the crystals begin to change in the neighbourhood of 40 per cent. Sb and 60 per cent. tin, and it is probable that near that point they begin also to change in composition, and contain more than the atomic proportion of antimony. The crystalline form of the hard, bright portions is similar in all the alloys containing between 45 per cent. and 55 per cent. antimony, and instead of being well-defined cubes, consist of thick plates.

On etching the alloys containing 45 per cent. antimony with strong nitric acid the external portion of these plates are relatively more rapidly corroded than the internal parts. This difference is very slight in this alloy, but it increases and becomes more pronounced with each addition of antimony, and eventually, when 51 per cent. is present in the very slowly cooled alloy, a hard central plate appears on polishing, which is left in bold relief, and is evidently either antimony or antimony with some tin. When, however, this alloy is cooled rapidly from fusion this constituent apparently does not separate.

In the slowly cooled alloys containing from 51 per cent. to 57 per cent. antimony this hard central constituent increases with the antimony. At first it appears in section as a hard band without any definite crystalline form, so far as can be seen in the sections; but with more antimony these forms increase in size and branch out through the central parts of the hard crystalline plates. Photo No. 1 represents the appearance of one of these developments.

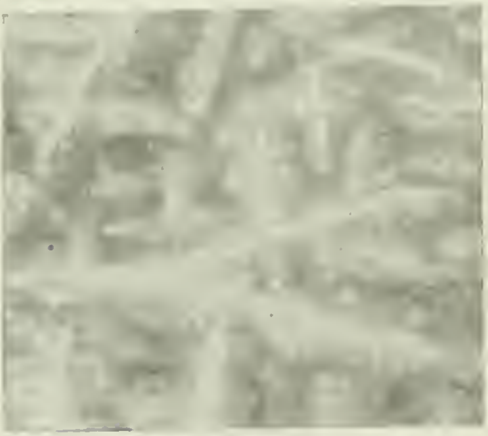


Fig. 1.
Sb 57 Sn 43
× 50 V

In the alloys containing between 57 per cent. and 80 per cent. the trains of these crystallites gradually change to more or less symmetrically shaped crystals, and resemble the trains of crystals of antimony which crystallise out of lead-antimony alloys containing 20 to 50 per cent. antimony.

When 90 per cent. antimony is reached, the crystals, owing to mutual interference during their growth, have not such symmetrical forms as they have in the alloys containing less antimony, but there are still to be observed many of the straight faces of true crystals.

As 100 per cent. of antimony is approached the crystals of that body come closer together, and very little of what may be called the second eutectic intervenes between them. When tin is absent none of the eutectic can be detected.

The presence of the first eutectic can be detected even in the alloy containing 57 per cent. antimony, where it is found in interstices between the harder crystalline masses. When the antimony exceeds 57 per cent this eutectic apparently disappears, and the central crystallites begin to assume crystalline form, and are embedded in what may be called a second eutectic.

It is difficult to determine whether this second eutectic is a solidified solution of antimony in the definite compound SbSn, or whether the two definite bodies form isomorphous compounds with each other, and when the antimony reaches a certain point it crystallises out of it in a separate state.

It is probable that the crystallites which first fall out of the second eutectic in the alloys containing between 51 and 57 per cent. antimony, may not be pure antimony, but an amorphous, imperfectly crystallised mixture of antimony with a small portion of SbSn. Success has not attended the efforts to separate them, and we therefore have not had an opportunity of analysing them.

### ANTIMONY AND COPPER.

The alloys of antimony and copper are of no industrial value, and, from the practical point of view of the Society of Chemical Industry, it would not at first appear that results obtained from the study of such alloys would be of use to its members.

Although it may be that directly little practical use can be obtained by such a study, nevertheless indirectly the knowledge obtained will undoubtedly assist us in understanding the constitution of the more valuable alloys in constant use.

Mons. Charpy, in the Bulletin de la Société d'Encouragement pour l'Industrie Nationale, March 1897, describes the alloys of antimony and copper, and in many particulars his observations agree with those of the writer, but in others they are somewhat different, as will be observed in the results which follow.

In the selection of materials the best star antimony and electro-copper were obtained. The antimony contained 99·5 per cent. antimony, 0·3 per cent. arsenic, 0·10 per cent. iron, and 0·10 per cent. of other impurities. The copper, after fusing under charcoal, contained 99·91 per cent. copper.

The only element which was likely to interfere with the micrographic structure was the arsenic, but as there is a close relationship between the structure of antimony-copper and arsenic-copper alloys, it is probable the arsenic present in the antimony would not materially alter the result. They cannot, however, be considered as more than a close approximation to the truth. It would have been better if pure antimony could have been obtained, but it was difficult to prepare the quantity required for the complete series.

*Classification of the Alloys.*

The alloys of antimony and copper can be arranged into several classes, based on their microstructure, containing between, *viz.*:—

| | | | | | |
|---|---|---|---|---|---|
| 1st. | 100 | per cent. and | 75·8 | per cent. | antimony. |
| 2nd. | 75·8 | ,, | 48·5 | ,, | ,, |
| 3rd. | 48·5 | ,, | 38·57 | ,, | ,, |
| 4th. | 38·57 | ,, | 31·00 | ,, | ,, |
| 5th. | 31·00 | ,, | 0·00 | ,, | ,, |

*Class 1.—Antimony,* 100 *per cent. to* 75·8 *per cent.*

The antimony itself, on polishing and etching or colouring by heating, indicated a homogeneous structure.

On the addition of 0·5 per cent. copper, the presence of a second constituent enveloping the grains of antimony could be clearly seen after polishing alone. This constituent in the slowly cooled alloys had not the characteristic banded appearance of a eutectic, but consisted of simple envelopes, more or less continuous, of the purple compound of antimony and copper of the composition $SbCu_2$. It is easily darkened by exposure to the fumes of hydric sulphide or by ammonium sulphide, or by oxidation produced on heating, and after such treatment the sections were easily photographed.

When 5 per cent. copper was present there was an increased quantity of the purple compound enveloping the grains of antimony.

When the copper was increased to 10 per cent. the characteristic structure of eutectics was clearly developed, and alternate bands of antimony and the purple constituent surrounded the crystalline grains of antimony. Photo No. 3 is an illustration of the structure of this alloy.

Each further increase of copper gave a corresponding increase in the proportion of the fusible eutectic, and as this became greater, the antimony in crystallising out had more room and space in which to develop its crystalline form, and the structure consisted of more or less well-developed trains of crystals cutting through the eutectic. No. 4 photo represents this structure with 80 per cent. antimony and 20 per cent. of copper.

The alloy containing about 75·8 per cent. of commercially pure antimony and 21·2 per cent. copper was composed

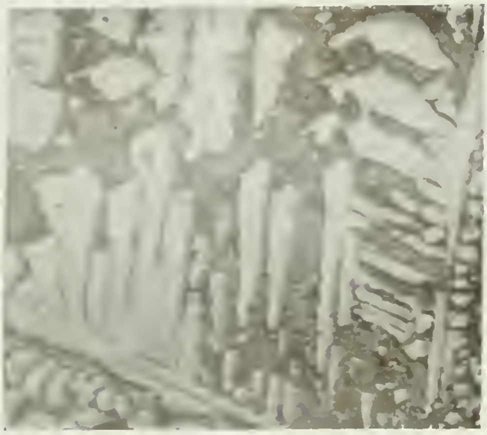


Fig. II

Sb 80 %, Sn 20

X 50 V

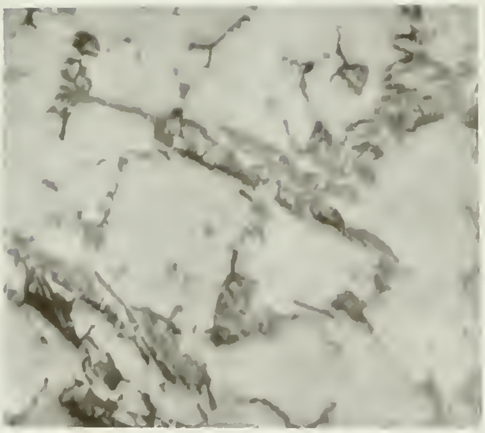


Fig. III.

Sb 90 % Cu 10 %

X 100 V

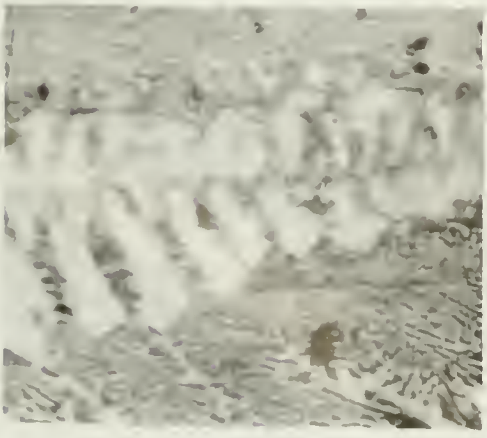


Fig. IV.

Sb 80 % Cu 20 %

X 100 V

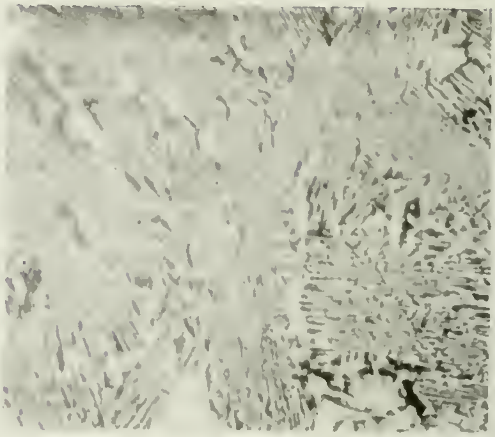


Fig. V.

Sb — 75·8 % Cu — 24·2 % X 100 V

First Eutectic.

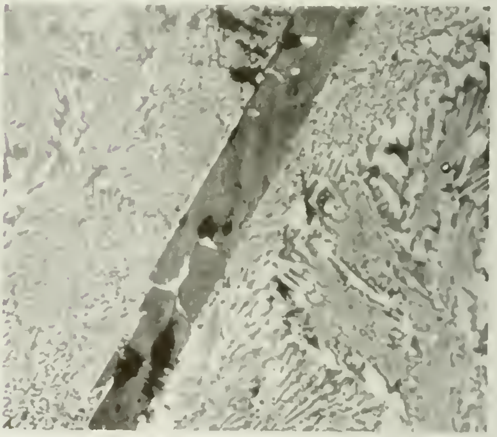


Fig. VI.

Sb 70 %, Cu 30 %

X 100 V

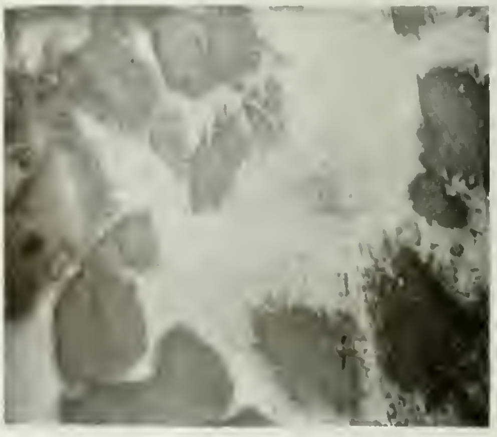


Fig. VII.

Sb 46 %, Cu 54 %

X 130 V

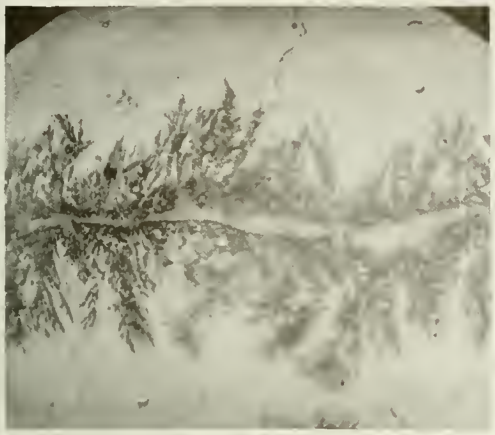


Fig. VIII.

Sb 41 %  Cu 59 %
X 130 V

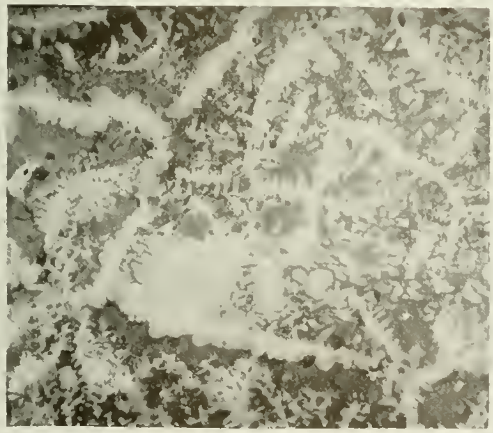


Fig. IX.

Sb - 31 %  Cu - 69 %  X 130 V
Second Eutectic, Cooled Slowly.

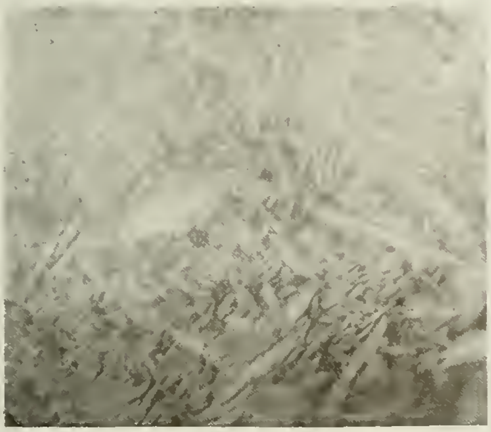


Fig. X.

Sb = 31 %  Cu 69 %  X 130 V
Second Eutectic, Cooled Rapidly.

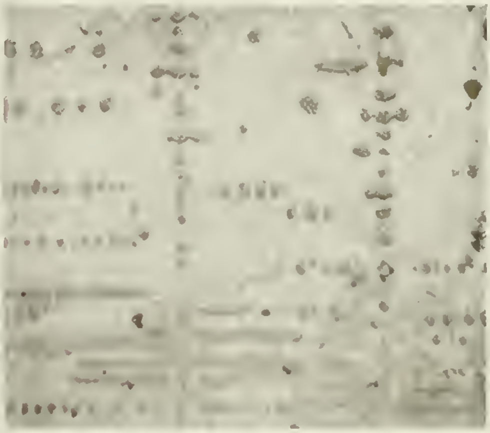


Fig. XI.

Sb 30 %  Cu 70 %
X 130 V

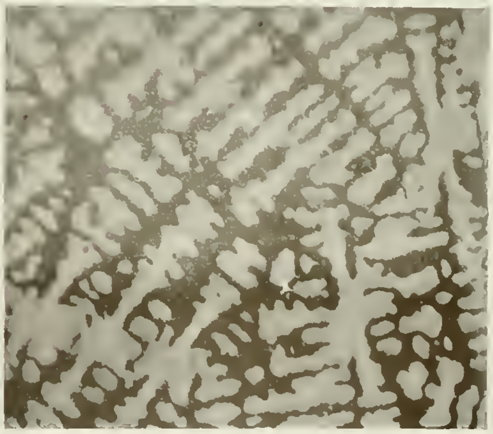


Fig. XII.

Sb 20 .  Cu 80 %
X 130 V

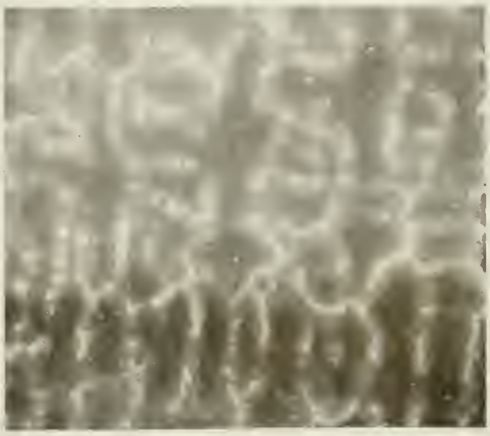


Fig. XIII.

Sb 1 %  Cu 99 %
X 50 V

entirely of the eutectic of the structure seen on each side of the crystal trains of antimony in Photo No. 4, and also in Photos No. 5 and No. 6. The structure of this eutectic was much coarser than that of many other alloys, the alternate layers being relatively much more massive.

*Class 2.—Antimony, 75·8 per cent. to 48·5 per cent.*

In this class, as the copper was steadily increased above 24·2 per cent. the crystallites of the purple compound appeared in increasing quantity cutting through the mass of the eutectic (No. 6 photo), and the eutectic, as 51·5 per cent. copper was approached, steadily decreased.

With 50 per cent. copper only separate, isolated, broken, irregular bands of antimony were observed between the grains of the purple ground mass. When the alloy contained 51·5 per cent. copper the whole mass appeared purple and homogeneous. It had approximately the composition of $SbCu_2$.

*Class 3.—Antimony, 48·5 per cent. to 38·47 per cent.*

As the copper was increased above 51·5 per cent. the alloys gradually decreased in purple colour* until about 39 per cent. antimony was approached, and they all gave, on simply polishing, sections which were quite flat, but in which could be seen under the microscope two distinct constituents, one purple and the other white.

On heating on a hot metal plate they were coloured by oxidation, taking the colours progressively of straw-yellow, orange, yellow, crimson, purple, blue, and steel-grey. If the heating was continued too long, both constituents passed through the complete range of colour and arrived at the steel grey colour, but in passing to this final point the constituents containing the highest amount of copper changed relatively more rapidly than those containing less of that element.

The purple compound contained less copper than the white constituent, and if the changes in colour on heating were carefully watched, it was possible to obtain the former as orange-yellow on a ground of the finest blue.

In the alloy containing about 54 per cent. copper and 46 per cent. antimony the purple compound appeared more or less irregular in shape, almost always rounded and free from anything like geometrical angles. From these bodies there were to be seen needle-like projections penetrating the ground mass. Photo No. 7 is a representation of this alloy, the structure having been developed by heating. The dark areas are the purple constituent.

As the copper is increased the purple constituent decreases, and when the antimony was reduced to 41 per cent. the purple constituent was found as veins cutting the ground mass, and from these veins a moss-like growth of what was possibly the same constituent, was attached. Photo No. 8 is an example of this.

When the alloy contained copper and antimony in the proportion of $SbCu_3$, the structure was similar to the ground mass shown in No. 7, and consisted of crystalline irregular-shaped grains apparently homogeneous.

The homogeneous character would lead us to believe that we had here a second definite compound of the composition $SbCu_3$.

According to Le Chatelier the alloy containing about 60 per cent. copper has a high melting point, and the curve of fusibility falls away on each side of it, with increased antimony on one side and with increased copper on the other.

M. Charpy, in his communication before referred to, says that the compound containing 60 per cent. copper and 40 per cent. antimony is entirely composed of the purple compound, and that the microscope indicates the presence of a definite compound of $SbCu_2$. There is no doubt about the statement, but as it is so well known that it is the alloy containing about 50 per cent. of each element which gives the most pronounced purple colour, it would appear that there is confusion somewhere. According to M. Charpy the definite compound is $SbCu_2$, but this contains 51·5 per cent. copper, not 60 per cent.

Again, as a matter of fact, each increment of copper above 51·5 per cent. diminishes the colour until about 61 per cent. is reached, when, instead of being composed of the purple compound, it has scarcely a tinge of purple. The high critical fusion point of Le Chatelier and the homogeneous microscopic structure would lead us to believe that the alloy was either a definite compound of the composition represented by $SbCu_3$, or some combination of two or more definite compounds, which crystallise together and do not separate into two parts.

When 51·5 per cent. copper is slightly exceeded, a small quantity of the compound, probably having the composition of $SbCu_3$, is formed, and the amount increases with the copper, till, when 61·43 per cent. is present, the alloy is entirely composed of it.

In the intermediate alloys containing between 51·5 per cent. and 61·4 per cent. the solidifying point increases with the copper, but, as would be expected from the microstructure, there are not two marked arrests in the cooling curve, but a more or less prolonged arrest through several degrees; in other words, the second arrest follows the first so closely as to make it difficult to tell when one begins and the other ends.

Probably the exceedingly delicate apparatus of Professor Roberts-Austen would give better definition than that at the command of the writer.

*Class 4.—Copper, 61·43 per cent. to 69·00 per cent.*

The correct interpretation of the microstructure of the alloys in this class is difficult; therefore, beyond describing what was actually seen and proved, and pointing out the suggestions they appear to convey, it is not proposed to go further.

The structure of the alloys containing between 61·43 and 64 per cent. copper were very similar. They were all composed of irregular-shaped polygonal grains, and these were enveloped by thin bands of what appeared to be a different constituent. Oxidation by heating, and etching by concentrated hydrochloric acid, did not develop structures of the nature of eutectics, but the surfaces became more or less purple coloured.

When the copper approached 67 per cent., the bands surrounding the crystalline grains became very pronounced, and they coloured on heating relatively in advance of the ground mass. Crystallites of a sheaf form, similar to those sometimes met with in certain obsidians, appeared in the centre of the grains. On etching with strong hydrochloric acid, the veins and crystallites were acted upon more rapidly than the ground mass. Judging from these facts, we must conclude that they contained more copper than the latter. Polishing alone did not develop the structure and the colour of the polished sections were brilliantly white and free from any indication of free copper.

When 68 per cent. copper was present, the veins and microliths increased in quantity, but still there was no indication of free copper.

With 68·5 per cent. copper, the veins, on polishing, appeared pale yellow on a white ground, and some of the microliths were faintly yellow.

When 69 per cent. copper was reached, the veins appeared, on polishing, of a distinct yellow colour. Exterior to the veins and crystallites there appeared to be projecting borders, which oxidised, on heating, to an intermediate colour. In Photo No. 9 these projections are very clearly illustrated. The specimen contained 69 per cent. copper and 31 per cent. antimony, and its structure was developed by heating. The veins coloured most rapidly, then followed the crystallites in the centre of the grains. The irregular borders followed, and finally the ground mass began to colour.

The heating was continued till the veins and some of the crystallites passed through all the colours to a steel whiteness. The borders, when this was just accomplished, were sky-blue and the ground mass orange-yellow.

The photographs were taken on isochromatic plates, and, of course, the steel-white veins, illuminated by vertical rays of light, reflected the most actinic rays, and gave the deepest impression on the sensitive plate; next in order followed the crystallites, and then the blue borders, and, lastly, the orange-yellow ground mass.

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* Here, as elsewhere, the colour is taken from the appearance of the fractured surface, or the surface after polishing, before being heated or etched.

Etching with concentrated hydrochloric acid left the constituents differently acted upon. The veins and crystallites were most deeply corroded, the borders less so, and the ground mass least of all. The partial interpretation of these observations is not difficult.

The different constituents evidently contained different proportions of copper—the veins and the crystallites most, the borders less, and the ground mass the least.

It appears probable that neither the veins, crystallites, nor borders had any constant composition in the alloys containing between 65 per cent. and 69 per cent. of copper, because they varied in colour in the different sections after simple polishing.

The borders surrounding the veins, &c. appeared to be of the nature of solidified solutions of the substances composing the veins which, when the alloys were passing from the liquid to the solid state, had not time to completely separate themselves from the solidifying ground mass.

This interpretation is only advanced tentatively, and must not be regarded as a dogmatic statement. More investigation is required, and until much more work has been done it would be premature to formulate any definite theory.

The rate at which the solidification is effected materially modifies the structure, and it is certain that the relative proportions of the constituents vary with the rate the alloys are solidified.

In the rapidly cooled alloy, containing 69 per cent. copper, there is a less proportion of the constituent highest in copper. The thick white vein in Photo No. 9 is scarcely visible in the quickly cooled alloy Photo No. 10, but there is a large increase of the needle-like crystallites.

### *Class 5.—Copper,* 69·00 *per cent. to* 100 *per cent.*

When 70 per cent. copper is present, that is, only 1 per cent. more than that contained in the eutectic, the structure is completely changed. The Photo No. 11 represents the appearance of this alloy etched with strong hydrochloric acid. The intermediate crystallites, the veins and borders, are now absent, and are replaced by dendritic skeletons, suggesting crystallites of octahedrons. These are pale yellow in colour, and suggest that they are copper, or copper containing some antimony.

The ground mass, on colouring by careful heating, appears as small irregular shaped grains, sometimes longer in one axis than in the other, and are slightly differently coloured, suggesting a slightly different composition. The difference in the colour, however, is so slight we are hardly justified in assuming there are two constituents. Whether it is a eutectic in the ordinary sense or not, it nevertheless can be regarded as part of the solidified mother liquor left after the dendritic crystallites had ceased growing, the second constituent of the eutectic having been attracted to and united with the cuprous crystallites.

With 80 per cent. copper the dendritic crystallites increase in quantity, as can be seen in Photo No. 12. The structure in this case was developed by heating. The white parts are the copper, or copper containing some antimony; the dark parts, the free portion of the eutectic or definite compound. No reagent was found which would dissolve out one or other of the constituents of this alloy, and we could not separate the yellow dendrites for analysis. It was therefore necessary, by metallographic methods alone, to endeavour to determine whether they were pure copper, or copper and some antimony as a solidified solution. To effect this the alloy was melted, and just before it solidified a fine piece of pure copper wire was thrust into the pasty mass. The alloy was then allowed to cool naturally. When cold it was ground down, so that the polished surface cut the copper wire.

After polishing and examining under the microscope, the copper inserted appeared much redder than the accompanying dendrites, and on heating it coloured in advance of them.

We may be satisfied, then, that the dendritic forms which fall out of solution in alloys containing 30 per cent. and less of antimony are not pure copper, but mixtures, probably in no definite proportion, of a preponderating amount of copper and a diminishing proportion of antimony, the latter decreasing as the proportion of the copper in the alloy is increased.

As the antimony is reduced, the dendritic forms pass from orange yellow and approach the colour of copper, and at the same time the eutectic is reduced in quantity.

With 1 per cent. of antimony and 99 per cent. of copper the free part of the eutectic can be easily seen on the polished section in isolated patches or seams between the grains of copper. Its colour is a neutral tint, slightly blue.

The definite separate constituent could be seen on simply polishing alloys containing 0·3 per cent. antimony, and it is possible that if solidification was effected very slowly it might be seen in a separate state when less antimony was present.

In the trials of the writer no special precautions were taken in cooling, as the crucibles, when taken from the fire, were allowed, with their contents, to cool on a metal plate. Made in this way, it is doubtful whether or not any of the separate antimonide could be detected in the alloys containing 0·2 per cent. antimony. The difference in the colour between the copper and the antimonide after polishing was so marked, we are satisfied that, if there had been anything like 0·1 per cent. of the whole mass in the separate state of the pale blue constituent, it would have been detected. We are led to believe that the greater portion, if not all, of the antimonide of the eutectic remained behind with the copper in solidified solution when 0·2 per cent. antimony is present.

It appears probable that when the copper was crystallising during its efforts to expel the more fusible compound to the exterior of the crystallising masses, that if it did not succeed in doing so, the borders would contain more antimony than the central parts of the crystalline grains, and that if the polished sections were heated there would be a gradation in the colour produced by oxidation, that the central portions of the grains would colour in advance of their external parts. On making the trial, what was anticipated actually occurred; the borders coloured last, and in a graduated order the parts at the actual junctions of the grains being the last to change.

The presence of even 0·05 per cent. antimony was capable of producing a marked difference in colour on the different parts of the oxidised sections.

The effect of etching with nitric acid was equally marked and indicated external crystalline borders which were less rapidly acted upon than the centres.

Although much pains was taken to detect in these borders alternate laminea of two constituents nothing of the kind could be observed even on the use of the best high power oil immersion objectives.

It would appear certain therefore that in the specimens prepared in the manner described, the antimonide of the eutectic which was incapable of being actually thrown out of solution by the crystallising copper, remained behind imprisoned and dissolved in the borders of the grains or crystalites of copper, in solidified solution.

Photo No. 13 gives a good idea of the character of copper containing the separated eutectic of antimony and copper, shown white in the photograph, and the half tone borders of the grains adjoining the separated eutectic where it had been retained as a solidified solution.

Professor Arnold in his most valuable communication on the "Influence of small quantities of impurities on gold and copper," published in Engineering, February 7, 1896, in his remarks concerning copper containing 0.2 per cent. antimony: says, "the latter metal produces very thick brown cell walls or sectional meshes which seem in volume or area altogether out of proportion to the percentage of the antimony present. However, on examination at high power, the antimonide meshes are found to possess a distinct compound structure consisting of alternate light and dark laminæ suggesting that in the first instance an attenuated antimonide involves the copper cells at a high temperature, but at a lower temperature the walls split up into plates of of pure copper and alternating with those of a less basic antimonide."

Professor Arnold's observations agree with those of the writer in regard to the thickness of the cell walls in the alloy with 0.2 per cent. antimony, but we have either seen the same thing differently in these cell walls or the conditions under which the two observers cooled their alloys may have been sufficient to account for alternate layers having been seen in one case and not in the other.

After etching with nitric acid the writer has noticed that the cell walls are unequally attacked and become roughened and that on wiping the surface with a cloth the parts highest in relief become bright, and the lower parts remain dark, but the difference was not noticed in the etched sections which were not wiped with a cloth.

Further trials are required to definitely explain the discrepancy. One thing is certain, that in all the alloys containing more than 50 per cent. copper the structures and probably the composition of the separated constituents are profoundly modified by the way the liquid alloys are cooled.

Owing to the difficulty of protecting the Le Chatelier pyrometer couples from the vapour of antimony the writer has refrained from presenting his results showing the curves of fusibility as the antimony vapour more or less vitiates the electric properties of the thermo junctions; he is therefore preparing new couples, coated with carbon, such as are used by Professor Roberts Austen with such success. When these are made the melting points of the alloys will be carefully determined and the results recorded.

| | | |
|---|---|---|
| 1st Class | Antimony, 100, 24·2, | Antimony alone, then antimony crystallised in the eutectic containing 24·2 per cent. copper. |
| 2nd Class | Antimony, 24·2, 48·5 per cent. | The eutectic alone then SbCu2 crystallised in the eutectic, the SbCu2 increasing with the copper till 51·5 is present when the whole mass is SbCu2, the purple compound. |
| 3rd Class | Antimony, 48·5, 38·5 per cent. | The purple compound alone, then imperfectly crystallised SbCu2 in what appears to be a second definite compound SbCu3 which is white, the purple compound decreasing and finally disappears when the antimony reaches 38·5 per cent., the whole mass being then composed of a white compound in fracture and on polishing, but which takes a purple colour on etching with hydrochloric acid. |
| 4th Class | Antimony, 38·5 and 31 per cent | The structure of the alloys containing between 38·5 and 36 per cent., are similar in appearance, and thin veins envelope the grains and from these veins needle-like processes penetrate into the grains for a very short distance. Some of the grains colour to a more pronounced purple than others on etching. As the antimony is decreased below 36 to 31 per cent., microliths of some compound appear in the centre of the grains and the veins round the grains increase in thickness, and in the copper they contain. When 31 per cent. antimony is present, the microliths are present in increased quantity, and at this point it would appear we have the second eutectic with a structure most complicated, probably consisting of more than one definite compound and no absolutely free copper. |
| 5th Class | Antimony, 31 to 0 per cent. | As the copper is increased above 69 per cent., it at first falls out of the eutectic in detached isolated globulites. They are not pure copper, but contain antimony, and arrange themselves in definite lines and angles, and as the copper is further increased join together to form dendritic crystallites. The microliths are absent, and it is probable had fallen into and become part of the yellow coloured dendrites at the moment of solidification.<br>The colour of the dendrites pass from yellow to red as the antimony is further reduced, and finally the last 0·1 or 0·2 per cent. are left behind at the borders, but not separated from the grains of copper, where it probably exists as some antimonide of copper in solid solution in the copper which in solidifying was unable to throw it entirely outside of itself. |

Mons. Charpy states that M. H. Le Chatelier has determined the melting points of these alloys and that the "curve of fusibility is composed of three branches cutting in two angular points, for the alloys containing 25 per cent. and 71 per cent. copper; the intermediate branch shows a maximum in the neighbourhood of 60 per cent copper."

"This," says M. Charpy "leads us to admit the existence of a definite compound corresponding to $SbCu_2$, a composition the electric conductivity determined by M. Mathiessen also presents a maximum."

Here again we fail to grasp M. Charpy's meaning, for if the maximum referred to in the curve of Le Chatelier in the neighbourhood of 60 per cent. copper means anything it points to a definite compound of $SbCu_3$ and not $SbCu_2$.

In his conclusions Mr. Charpy says: "In the alloy containing from 0 to 25 per cent. copper at the point when solidification commences, crystals of pure antimony are separated which gradually grow as the temperature falls; the liquid portion is thus concentrated in copper, and when it has reached the composition of the eutectic alloy solidifies at a constant temperature separating into antimony and $SbCu_2$. In the alloys containing from 25 to 60 per cent. copper the same thing happens; but at the commencement it is the compound $SbCu_2$ which separates out. In the alloys with from 60 to 70 per cent. copper the same compound separates out, but it is surrounded by a second eutectic alloy formed this time of $SbCu_2$ and copper. Finally from 70 to 100 per cent. copper, the copper would separate out in the eutectic alloy."

In the first part of his summary, M. Charpy and the writer are in unison, but, as will be seen from the following conclusions, we differ materially when we describe the alloys containing above 50 per cent. of copper.

### *Notes on the Eutectics.*

Before closing this section, reference must be made to the peculiarity of the first eutectic when in the presence of a preponderating proportion of antimony on the one side and the purple constituent on the other.

It is not the eutectic which is seen in the sections when 95 per cent. and 50 per cent. antimony are present, only one part of it, the other part having been absorbed by the greater mass of antimony on the one side and the $SbCu_2$ on the other in the act of solidification. For instance, with 95 per cent. antimony the cooling curve shows the melting point of the eutectic, which contains 24·2 per cent. copper, yet the structure shows only envelopes of the purple constituent surrounding the grains of antimony and not any eutectic.

The same thing occurs when there is present 50 per cent. antimony, *i.e.*, about 97 per cent. of the purple constituent and 3 per cent. of antimony over and above what is required to make with the copper $SbCu_2$, but it is strips or particles of antimony which remain between the grains of the crystallised purple compound, and not the eutectic.

The same thing occurs, but in a much more marked degree, when the cuprous dendrites fall out of the second eutectic, the result being that in the sections of the slowly cooled alloys, containing above 70 per cent. copper, there is not to be seen any eutectic between the dendritic crystallites but only one part of it, the other part evidently having fallen into the crystallites during solidification. This must be so for the cooling curve indicates a eutectic of the same solidifying point in all the alloys containing between 69 per cent and 80 per cent. of copper.

## COPPER AND TIN.

In one of his previous papers the writer drew attention to the fact that the crystallites which fell out of solution of the alloys rich in tin, containing 1 to 2 per cent. of copper were composed of SnCu and not $SnCu_3$, as was contended by other investigators, and that the crystallites (separated by dissolving away the excess of tin in dilute acid) from the alloys containing between 3 per cent. and 34 per cent. copper apparently had no definite chemical composition, the copper rising steadily in the separated crystallites with each increment of that element in the alloy. On further examination of these crystallites the writer has proved that they consist of at least two definite constituents, one covering the other.

In crystallising, the constituent containing the most copper falls out first, and it is on the surface of this that the second constituent, probably SnCu, crystallises.

This observation is interesting as the micro-structure now explains and confirms the significance of the lower retardations in the cooling curve obtained by Professor Roberts-Austen and Mr. Stansfield.

Besides making this note it is not proposed to give further details, reserving the full consideration of all the alloys of copper and tin for a future communication.

## New York Section.

*Meeting held on Friday, November 25th, 1898.*

MR. T. J. PARKER IN THE CHAIR.

### CHEMICAL SPECIFICATION FOR CHILLED IRON CAR WHEELS.

BY W. WALLEY DAVIS, JUNR.

The present study was taken up with a view to discovering, if possible, whether there is a definable and distinct relation between the chemical constituents and the physical properties of chilled iron car wheels; and further, whether by assigning a fixed chemical constitution and maintaining it by constant analysis, physical results, invariable or reasonably invariable, could be obtained. The chief requisites in a chilled car wheel are: first, ability to withstand concussion and shock; second, ability to expand or contract without developing cracks; and third, hardness of the surface of the chill. The measure of the last naturally has the greatest bearing upon the life of a wheel; and of the first two, upon the functions of the safety and capacity. What adds to the third requisite naturally detracts from the sum of the first two, and *vice versâ*, making an even balance necessary for safe and economical performance, and requiring thorough knowledge of the materials used, and good judgment in using and manipulating them. A thorough knowledge of the materials used in the manufacture of wheels means usually a comprehensive idea of the physical properties of the various component irons without special reference to the chemical analysis of the same, it being argued that a chemical analysis cannot be depended upon in this instance, partly on account of the variability of such analyses, and partly on account of the fact that it has not been absolutely determined which one or more of the elements has the most important bearing upon the physical characteristics of the finished wheel, and within what limits these may be varied without detracting materially from any of the three requisites.

Table No. 1.

*Analyses of Irons.*

| Reference No. | Grade. | C. Total. | C.C. | G.C. | Si. | Mn. | S. | P. |
|---|---|---|---|---|---|---|---|---|
| 1 | No. 4 ................ | 3·93 | 1·70 | 2·23 | 0·42 | 0·37 | Trace | 0·20 |
| | | 3·83 | 2·53 | 1·30 | 0·33 | 0·25 | 0·005 | 0·22 |
| 2 | No. 2 ................ | 4·17 | 1·01 | 3·16 | 1·27 | 0·85 | 0·005 | 0·22 |
| | | 3·94 | 2·51 | 1·43 | 0·94 | 0·67 | 0·009 | 0·21 |
| 3 | No. 4 ................ | 3·67 | 0·66 | 3·01 | 0·51 | 0·55 | 0·030 | 0·37 |
| | | 3·86 | 0·84 | 3·02 | 0·41 | 0·38 | 0·031 | 0·34 |
| 4 | No. 2 ................ | 4·09 | 0·50 | 3·59 | 0·71 | 0·70 | 0·011 | 0·43 |
| | | 3·80 | 0·75 | 3·05 | 0·68 | 0·51 | 0·015 | 0·47 |
| 5 | No. 2 ................ | 3·82 | 0·70 | 3·12 | 0·76 | 0·33 | Trace | 0·22 |
| | | 3·85 | 1·07 | 2·78 | 0·70 | 0·21 | 0·008 | 0·18 |
| 6 | No. 3 ................ | 3·13 | 0·64 | 2·49 | 1·20 | 0·30 | 0·003 | 0·23 |
| | | 3·40 | 1·23 | 2·17 | 1·04 | 0·24 | 0·009 | 0·18 |
| 7 | No. 4 ................ | 3·02 | 0·73 | 2·29 | 1·50 | 1·17 | 0·030 | 0·41 |
| | | 3·37 | 0·76 | 2·61 | 1·18 | 1·00 | 0·038 | 0·45 |
| 8 | No. 4 ................ | 2·88 | 0·67 | 2·21 | 0·89 | 0·86 | 0·031 | 0·38 |
| | | 3·34 | 0·59 | 2·75 | 0·83 | 0·73 | 0·041 | 0·40 |
| 9 | No. 4 ................ | 3·38 | 0·92 | 2·46 | 0·83 | 0·84 | 0·015 | 0·36 |
| | | 3·29 | 0·48 | 2·81 | 0·76 | 0·70 | 0·021 | 0·42 |
| 10 | No. 2 ................ | 3·61 | 0·65 | 2·96 | 1·62 | 1·22 | 0·004 | 0·41 |
| | | 3·74 | 2·29 | 1·45 | 1·59 | 1·17 | 0·008 | 0·38 |
| 11 | No. 4 ................ | 3·78 | 0·91 | 2·87 | 0·70 | 0·24 | 0·005 | 0·19 |
| | | 3·78 | 2·53 | 1·25 | 0·88 | 0·18 | 0·028 | 0·20 |
| 12 | No. 4 ................ | 3·47 | 0·59 | 2·88 | 0·39 | 0·15 | 0·005 | 0·23 |
| | | 2·96 | 2·84 | 0·12 | 0·42 | 0·02 | 0·011 | 0·19 |
| 13 | No. 2 ................ | 3·60 | 0·21 | 3·39 | 1·27 | 0·33 | 0·004 | 0·27 |
| | | 3·98 | 0·75 | 3·23 | 1·27 | 0·27 | 0·008 | 0·27 |
| 14 | No. 3 ................ | 4·04 | 0·48 | 3·56 | 0·46 | 0·31 | 0·008 | 0·44 |
| | | 4·11 | 1·32 | 2·79 | 0·42 | 0·28 | 0·021 | 0·47 |
| 15 | No. 2 ................ | 3·86 | 0·73 | 3·13 | 0·79 | 0·14 | 0·031 | 0·41 |
| | | 3·73 | 1·61 | 2·12 | 0·91 | 0·27 | 0·039 | 0·35 |
| 16 | No. 3 ................ | 3·73 | 1·32 | 2·41 | 0·93 | 0·50 | 0·012 | 0·33 |
| | | 4·00 | 1·03 | 2·97 | 1·02 | 0·37 | 0·018 | 0·34 |
| 17 | No. 2 ................ | 3·42 | 0·47 | 2·95 | 1·41 | 0·62 | 0·016 | 0·33 |
| | | 3·99 | 0·86 | 3·13 | 1·39 | 0·54 | 0·021 | 0·31 |
| 18 | No. 3 ................ | 2·56 | 0·32 | 2·24 | 2·30 | 0·15 | 0·025 | 0·27 |
| | | 3·39 | 1·10 | 2·29 | 1·93 | 0·24 | 0·010 | 0·27 |
| 19 | No. 4 ................ | 2·84 | 0·73 | 2·11 | 1·73 | 0·87 | 0·037 | 0·33 |
| | | 3·06 | 0·74 | 2·32 | 1·81 | 0·29 | 0·059 | 0·34 |
| 20 | No. 2 ................ | 2·80 | 0·44 | 2·36 | 1·75 | 0·61 | 0·032 | 0·31 |
| | | 3·06 | 0·74 | [illegible] | 1·70 | 0·39 | 0·051 | 0·28 |
| 21 | No. 4 ................ | [illegible] | [illegible] | 2·57 | 0·57 | 0·27 | 0·011 | 0·43 |
| | | 3·55 | 1·37 | 2·18 | 0·41 | 0·02 | 0·020 | 0·10 |
| 22 | No. [illegible] ................ | 2·93 | 1·00 | 1·93 | 0·39 | 0·32 | 0·006 | 0·36 |
| | | 2·98 | 2·83 | 0·15 | 0·39 | 0·11 | 0·023 | 0·38 |
| 23 | No. 4 ................ | 3·49 | 0·48 | 3·01 | 1·06 | 1·10 | 0·008 | 0·33 |
| | | 3·92 | 0·83 | 3·09 | 1·07 | 0·40 | 0·015 | 0·33 |
| 24 | No. 2 ................ | 4·06 | 0·91 | 3·15 | 1·32 | 1·25 | 0·005 | 0·37 |
| | | 3·78 | 0·76 | 3·02 | 1·08 | 0·34 | 0·013 | 0·37 |
| 25 | No. 4 ................ | 3·61 | 0·84 | 2·77 | 0·76 | 0·60 | 0·011 | 0·66 |
| | | 3·61 | 0·87 | 2·74 | 0·89 | 0·23 | 0·021 | 0·66 |
| 26 | No. 4 ................ | 3·37 | [illegible] | 1·84 | 0·90 | 0·10 | 0·013 | 0·35 |
| | | 3·05 | 1·41 | 1·64 | 0·96 | 0·05 | 0·038 | 0·34 |
| 27 | No. 3 ................ | [illegible] | [illegible] | 2·50 | 1·01 | 0·19 | 0·024 | 0·46 |
| | | 3·44 | 1·17 | [illegible] | 0·99 | 0·13 | 0·032 | 0·43 |

Note.—Drillings taken from chilled bars, except No. 26.

The methods of manipulating, pouring, annealing, &c., as well as the character of the "chill" used, while subject to strong controversy, have been narrowed down to several distinct methods, the details of which cannot be treated of here.

TABLE No 2.

SOLID LINES = ANALYSIS OF PIGS.
DOTTED " = " " TEST BARS.

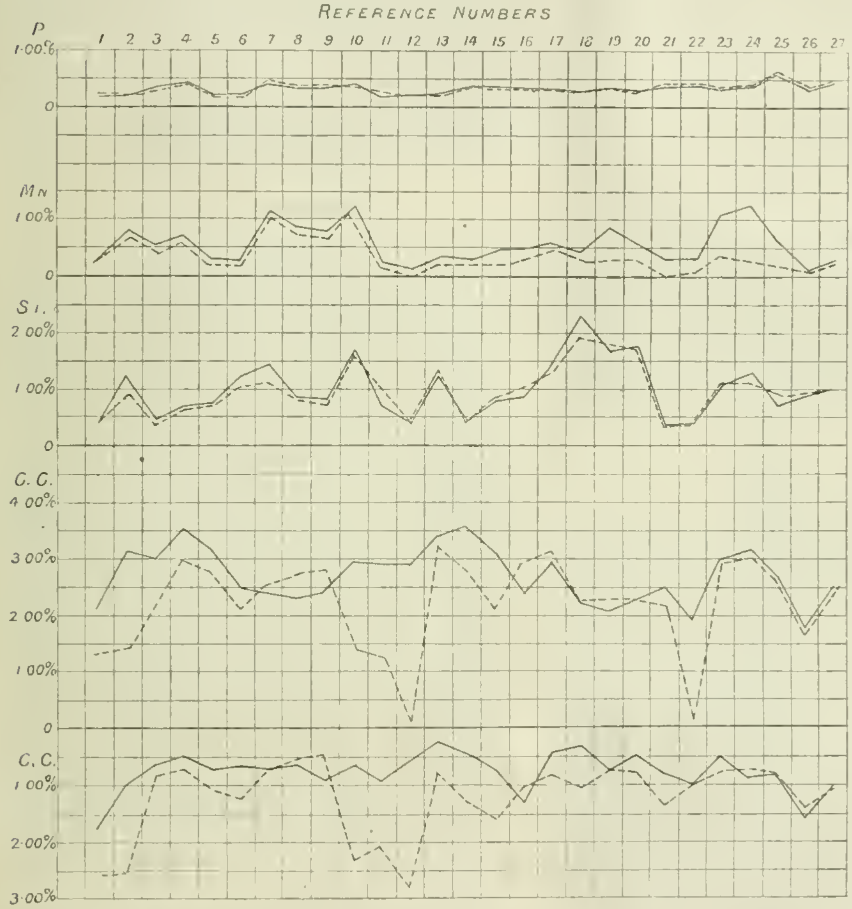
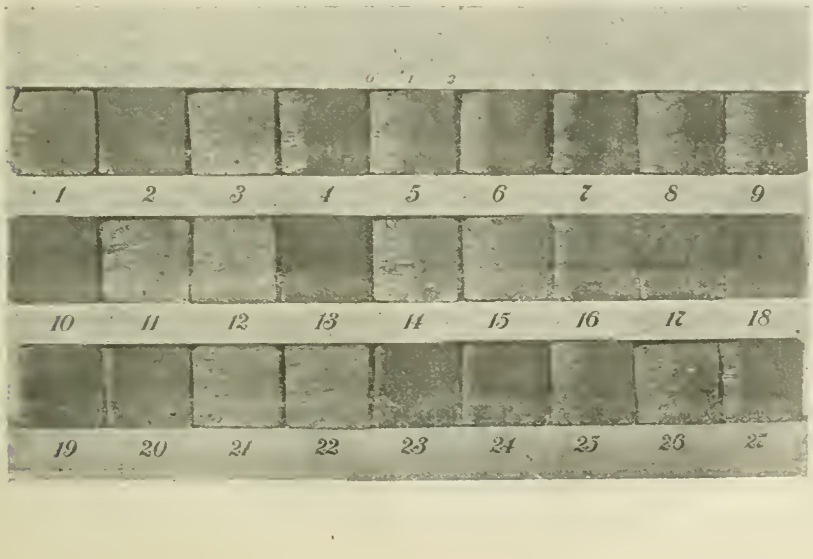


PLATE No. 3.



Aside from the regular inspection tests, wheels upon most of our railways are now subjected to the two specific ones, *i.e.*, the drop and thermal tests, the former being the measure of its ability to withstand a specified number of blows from a 140-lb. weight falling 12 feet, and the latter being a measure of its capacity for expanding under the application of heat upon the tread. For these tests one in fifty wheels is usually taken for each, and the character of the remainder determined by the one. While this is the only practicable way to carry this out, yet there are conditions which at times make it an unsafe one. It is very true that the chemical analysis of any cast-iron material of large bulk is often criticised on account of the claim that it does not represent the entire truth, and is not to be depended upon, as has been noted; but while this may be true in certain instances, yet it seems more rational to suppose that better and more invariable results may be secured if the founder works with a definite knowledge of the chemical structure of his irons, and combines them with the idea of producing a resultant yielding a fixed analysis. Charcoal iron, being the material which enters into the composition of wheels to the largest extent, was studied analytically in order to determine which brand seemed best adapted to the object in view.

For this purpose, 27 different samples were procured, representing mostly grades Nos. 2 and 4, from several available furnaces, and these lots were analysed both with reference to the pigs themselves and to the test-bar after remelting. In Table No. 1 these analyses are shown, the irons being numbered 1 to 27 inclusive, the analysis of the test-bar immediately following each pig analysis, but bearing no number.

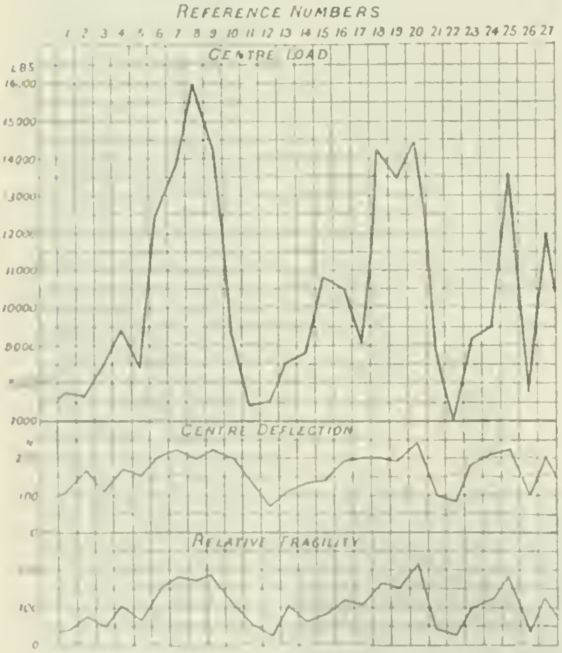
Table No. 2 exhibits these results graphically, the solid line showing the analysis of the pigs, and the dotted line that of the test bars.

The curves show that in melting the phosphorus remains constant for the most part, and that when under 1 per cent. the silicon likewise remains constant. Part of the manganese is always oxidised—the greater the amount, the greater the proportion. While the total carbon remains the same, the proportions of graphite to combined carbon vary in a marked degree, without reference to the silicon content. Part of this is due to the effect of the chill, as the analyses were made from the chilled bars where possible, and bars 11, 12, and 22 were chilled throughout, as the photograph (Plate No. 3) will show.

The bars as shown are 2 ins. square, and the amount of chill may be measured by the scale at the top. The photograph shows clearly to what extent the white iron blends into the grey—the more perfect the blending, the better the iron.

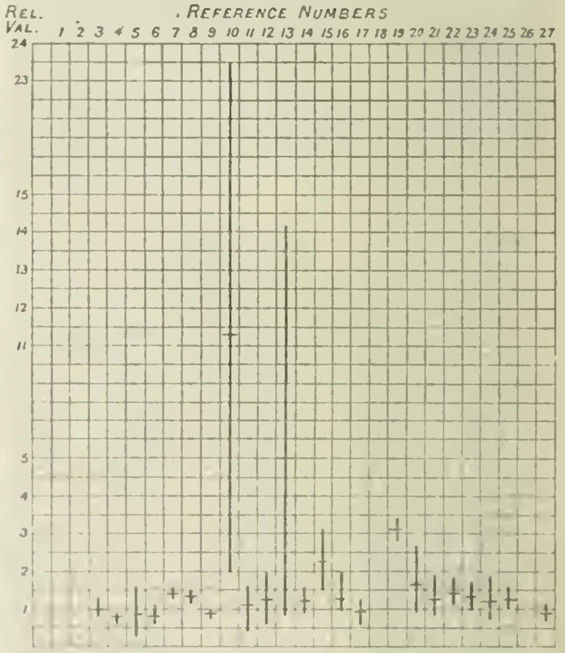
Table No 4 gives the plotted curves of all the physical tests performed, showing in detail the breaking load, centre deflection, and relative fragility of the average of all the unchilled test bars. The lowest curve is the most important here, as it shows the relative power of the metals to absorb work or shock. An examination of this curve indicates that numbers 6, 7, 8, 9, 18, 19, 20, 24, and 25, are particularly suitable in this respect, while numbers 1, 2, 3, 5, 11, 12, 14, 21, and 26, have little capacity for withstanding blows or shocks.

## TABLE Nº 4.



In order to make the comparative test of further value, a series of abrasive tests was made on bars 1 in. square by 6 ins. long, chilled on one end. These were placed in a plate provided with a number of 1-in. square openings, chilled end downward upon a carborundum wheel, 18 ins. in diameter, and revolved in a horizontal plane. Similar bars were made from a regular wheel mixture for comparative standards. The bars were first placed in the machine and ground down to a full bearing on the wheel, then accurately weighed, ground for from 3 to 5 hours, and finally reweighed, the loss of weight representing the amount of the metal abraded. Duplicates were not obtainable in this test; and while the irregularities developed made the test disappointing, in that no absolute dependence can be placed upon the results, yet the series is of value, in that it indicates that it is not so much the amount of the carbon in chilled iron which determines its hardness, but the form of its occurrence. Table No. 5 shows in graphical form the maximum, minimum, and relative loss, the standard bars of wheel iron being taken as units in each series of tests.

## TABLE Nº 5.



The longer the vertical line, the greater the irregularities in the test, showing that 10 and 13 are the worst, and 3, 4, 6, 9, 11, 17, and 27 the best.

In order that a theoretical composition for the iron may be arrived at by means of these physical tests, a combination of the figures leads to a regular grouping, all characteristics being considered as follows:—

Good:—Nos. 6, 7, 8, 9, 16, 19, 20, 25, and 27.

Fair:—Nos. 4, 15, 17, 18, 21, 23, and 24.

Poor:—Nos. 1, 2, 3, 5, 10, 11, 12, 13, 14, 22, and 26.

While it may be noted that the irons vary in a marked degree in the various constituents, the good or bad qualities not being distinctly traceable to the proportion of any single constituent, yet the following limits may be rationally deduced, and practice confirms the value of the deductions.

| Grade. | Chill. | Graphite. | Combined Carbon. | Si. | Mn. | S. Max. | P. |
|---|---|---|---|---|---|---|---|
| | In. | | | | | | |
| [illegible] | ½ | 2·50—3·00 | 0·40—0·90 | 1·25—1·75 | 0·50—1·00 | 0·03 | 0·25—0·40 |
| | ¾ | 2·25—2·75 | 0·50—1·00 | 1·00—1·50 | 0·50—1·00 | 0·03 | 0·25—0·40 |
| | | 2·00—2·50 | 0·75—1·25 | 0·75—1·25 | 0·50—1·00 | 0·035 | 0·25—0·40 |
| | 1 | 1·75—2·25 | 0·90—1·40 | 0·50—1·00 | 0·25—0·75 | 0·035 | ·25—0·40 |

While there is no doubt that the form in which the carbon occurs has the greatest bearing upon the character of the product, yet, not being able to satisfactorily determine this, more particular attention here is given to the complete analysis. Coke irons are used to a small extent in wheel mixtures, but their influence is not sufficiently strong to require special study here.

The tendency of most charcoal irons in grades 4 and 5 is to chill too heavily, whitening the plate of the wheel and making it brittle. A coke iron is efficacious here, in that it brings in a small increase in silicon, and has a depressing effect on the influence of the temper-carbon. It likewise brings up the manganese in the mixture and reduces the content of sulphur.

The wheels examined were selected either on account of failure to stand thermal or drop tests, or to give a respectable service under cars, or, on the other hand, because they had well met the requirements in all these particulars. The details of the analyses are given in the following table ;—

TABLE No. 6.

*Wheel Examinations.*

| Throat Chill. | Tread Chill. | Drop Blows. | Thermal Test. | Service. | C.C. | G.C. | Si. | Mn. | S. | P. |
|---|---|---|---|---|---|---|---|---|---|---|
| In. | In. | | | | | | | | | |
| .. | .. | .. | 0 | .. | 0·79 | 2·65 | 0·72 | 0·41 | 0·06 | 0·45 |
| 1/2 | 5/8 | 24 | .. | .. | 0·63 | 2·87 | 0·57 | 0·33 | 0·07 | 0·41 |
| 1/2 | 9/16 | 9 | .. | 8⅓ years | 0·66 | 2·75 | 0·67 | 0·31 | 0·05 | 0·35 |
| 1/4 | 7/16 | .. | 60 min. | 8⅓ ,, | 0·61 | 3·19 | 0·57 | 0·34 | 0·06 | 0·31 |
| 3/8 | 1/2 | .. | 0 | 7½ ,, | 0·56 | 3·14 | 0·72 | 0·26 | 0·07 | 0·35 |
| 3/4 | 7/8 | .. | 38 sec. | .. | 1·09 | 2·34 | 0·71 | 0·03 | 0·08 | 0·40 |
| 3/16 | 1/4 | .. | 0 | .. | 0·50 | 3·21 | 0·71 | 0·40 | 0·06 | 0·37 |
| 1/2 | 5/8 | .. | 0 | .. | 0·71 | 2·82 | 0·74 | 0·42 | 0·06 | 0·36 |
| 3/16 | 7/16 | .. | .. | 1 month | 0·99 | 2·66 | 0·81 | 0·29 | 0·05 | 0·44 |
| 3/8 | 1/2 | 53 | 10 min. | .. | 0·55 | 3·31 | 0·61 | 0·43 | 0·06 | 0·36 |
| 5/8 | 7/8 | .. | 0 | .. | 0·70 | 3·09 | 0·52 | 0·40 | 0·06 | 0·39 |
| .. | 9/16 | .. | 0 | .. | 0·60 | 3·28 | 0·58 | 0·30 | 0·06 | 0·37 |
| .. | 3/8 | 28 | .. | .. | 0·76 | 2·97 | 0·69 | 0·23 | 0·07 | 0·41 |
| .. | 5/8 | .. | 90 min. | 5 years | 0·81 | 2·80 | 0·55 | 0·27 | 0·07 | 0·49 |
| 3/16 | 3/8 | .. | .. | 2 months | 0·80 | 2·55 | 0·84 | 0·20 | 0·07 | 0·42 |
| 3/16 | 3/8 | .. | .. | 1⅚ years | 0·73 | 2·75 | 0·56 | 0·32 | 0·08 | 0·48 |
| 3/16 | 1/4 | .. | 0 | 1⅚ ,, | 0·88 | 2·65 | 0·59 | 0·29 | 0·06 | 0·45 |
| 5/8 | 3/4 | .. | .. | .. | 0·79 | 2·48 | 0·49 | 0·51 | 0·06 | 0·44 |
| 5/8 | 5/8 | .. | .. | .. | 0·32 | 3·51 | 0·62 | 6·32 | 0·06 | 0·31 |
| 7/8 | 1 | .. | .. | 1⅚ years | 1·14 | 1·90 | 0·74 | 0·22 | 0·12 | 0·43 |
| 1/2 | 1/2 | .. | 1 min. 20 sec. | .. | 0·73 | 2·46 | 0·68 | 0·22 | 0·09 | 0·43 |
| 9/16 | 11/16 | 34 | .. | .. | 0·68 | 2·75 | 0·56 | 0·53 | 0·05 | 0·46 |
| 1/4 | 1/2 | .. | .. | 1 month | 0·99 | 2·45 | 0·65 | 0·15 | 0·05 | 0·40 |
| 1/4 | 1/2 | .. | .. | 1 ,, | 1·05 | 2·35 | 0·33 | 0·16 | 0·05 | 0·39 |
| 5/8 | 5/8 | .. | 0 | .. | 0·77 | 2·72 | 0·54 | 0·37 | 0·07 | 0·44 |
| 3/8 | 1/2 | 45 | .. | .. | 0·76 | 2·65 | 0·58 | 0·44 | 0·09 | 0·44 |
| 5/16 | 1/2 | .. | 0 | .. | 0·95 | 2·70 | 0·67 | 0·53 | 0·09 | 0·41 |
| 5/16 | 1/2 | 46 | .. | .. | 0·65 | 2·74 | 0·64 | 0·46 | 0·08 | 0·43 |
| 5/16 | 7/16 | .. | .. | 5 months | 1·12 | 2·29 | 0·87 | 0·22 | 0·10 | 0·39 |
| 7/8 | 1 | .. | .. | .. | 1·42 | 1·96 | 0·80 | 0·29 | 0·10 | 0·41 |
| 3/4 | 13/16 | .. | .. | 5 months | 0·66 | 2·32 | 0·62 | 0·32 | 0·10 | 0·41 |
| 3/8 | 1/2 | .. | 0 | .. | 0·63 | 2·72 | 0·75 | 0·45 | 0·07 | 0·45 |
| 5/8 | 3/4 | 51 | 0 | .. | 0·72 | 2·73 | 0·51 | 0·37 | 0·07 | 0·48 |
| 5/8 | 3/4 | 55 | .. | .. | 0·73 | 2·83 | 0·56 | 0·39 | 0·06 | 0·44 |
| 1/2 | 5/8 | 48 | .. | .. | 0·85 | 2·73 | 0·71 | 0·35 | 0·07 | 0·39 |
| 5/8 | 3/4 | .. | 6 min. | .. | 0·73 | 2·87 | 0·66 | 0·39 | 0·06 | 0·42 |
| 5/8 | 3/4 | 41 | .. | .. | 0·66 | 2·83 | 0·59 | 0·43 | 0·07 | 0·52 |
| 5/8 | 3/4 | 40 | .. | .. | 0·78 | 2·77 | 0·61 | 0·47 | 0·07 | 0·43 |
| 1/2 | 5/8 | .. | 2 min. 5 sec. | .. | 0·90 | 2·74 | 0·49 | 0·47 | 0·08 | 0·43 |
| 1/2 | 11/16 | .. | .. | 8 months | 1·40 | 2·12 | 0·80 | 0·15 | 0·07 | 0·44 |
| 7/16 | 9/16 | .. | .. | 4 ,, | 1·40 | 2·21 | 0·81 | 0·12 | 0·07 | 0·37 |
| .. | .. | .. | .. | 5 ,, | 1·40 | 2·07 | 0·69 | 0·16 | 0·07 | 0·44 |
| .. | .. | | Burst in mounting. | | 0·47 | 2·94 | 0·63 | 0·39 | 0·07 | 0·44 |
| 3/8 | 1/2 | 49 | .. | .. | 0·91 | 2·85 | 0·58 | 0·41 | 0·04 | 0·43 |
| 1/2 | 5/8 | .. | 3 min. 5 sec. | .. | 0·81 | 2·62 | 0·61 | 0·28 | 0·06 | 0·40 |
| .. | .. | 47 | .. | .. | 0·74 | 2·73 | 0·67 | 0·24 | 0·05 | 0·45 |
| .. | .. | .. | .. | .. | 0·79 | 2·85 | 0·55 | 0·25 | 0·07 | 0·45 |
| 1/2 | 3/4 | | Burst in mounting. | | 1·09 | 2·47 | 0·55 | 0·24 | 0·06 | 0·39 |
| 3/4 | 7/8 | 28 | .. | .. | 1·19 | 2·23 | 0·55 | 0·11 | 0·08 | 0·42 |
| 1/8 | 5/16 | .. | .. | 13 years | 1·25 | 2·33 | 0·94 | 0·13 | 0·09 | 0·32 |
| 7/16 | 5/8 | .. | .. | 1 year | 0·93 | 2·46 | 0·56 | 0·15 | 0·06 | 0·42 |
| 1/2 | 5/8 | 34 | .. | .. | 0·65 | 2·82 | 0·58 | 0·25 | 0·07 | 0·43 |
| 3/8 | 1/2 | | Burst in mounting. | | 0·55 | 3·14 | 0·55 | 0·36 | 0·07 | 0·43 |
| 3/8 | 1/2 | .. | 90 min. | .. | 0·33 | 2·99 | 0·69 | 0·29 | 0·07 | 0·48 |
| 1/2 | 5/8 | .. | 90 min. | .. | 0·46 | 3·07 | 0·59 | 0·31 | 0·07 | 0·49 |
| 1/2 | 5/8 | .. | .. | 6 months | 0·86 | 2·57 | 0·62 | 0·18 | 0·06 | 0·50 |
| 3/8 | 9/16 | .. | .. | 4 ,, | 1·22 | 2·28 | 0·68 | 0·12 | 0·06 | 0·54 |
| 3/8 | 1/2 | .. | 45 min. | .. | 0·65 | 2·86 | 0·71 | 0·26 | 0·07 | 0·44 |
| .. | .. | .. | 0 | .. | 0·58 | 2·94 | 0·55 | 0·22 | 0·07 | 0·39 |
| 3/8 | 1/2 | .. | 50 min. | .. | 0·73 | 2·90 | 0·72 | 0·28 | 0·08 | 0·44 |
| 5/8 | 3/4 | .. | 60 min. | .. | 0·70 | 2·80 | 0·50 | 0·21 | 0·06 | 0·39 |
| 5/16 | 7/16 | .. | .. | 10 months | 1·06 | 2·72 | 0·73 | 0·19 | 0·07 | 0·43 |
| 3/8 | 1/2 | .. | .. | 4⅓ years | 0·65 | 2·90 | 0·60 | 0·16 | 0·05 | 0·39 |
| 3/8 | 1/2 | 58 | .. | .. | 0·70 | 2·85 | 0·68 | 0·30 | 0·07 | 0·41 |
| 1/4 | 1/2 | .. | .. | 10 months | 0·96 | 2·48 | 0·87 | 0·20 | 0·07 | 0·49 |
| 3/8 | 5/8 | .. | .. | 1 month | 1·18 | 2·31 | 0·58 | 0·13 | 0·06 | 0·41 |
| 1/2 | 3/4 | .. | .. | 4⅓ years | 0·65 | 3·05 | 0·60 | 0·27 | 0·07 | 0·44 |
| 3/4 | 7/8 | 32 | .. | .. | 0·84 | 2·48 | 0·49 | 0·24 | 0·08 | 0·44 |
| 5/16 | 1/2 | .. | .. | 9 years | 0·88 | 3·10 | 0·58 | 0·22 | 0·06 | 0·39 |
| 1/4 | 3/8 | .. | .. | 9 ,, | 0·78 | 2·89 | 0·60 | 0·24 | 0·06 | 0·34 |
| 1/8 | 1/4 | .. | .. | 9 ,, | 0·71 | 2·86 | 0·68 | 0·22 | 0·08 | 0·25 |
| 3/8 | 5/8 | .. | .. | 11 ,, | 0·63 | 2·89 | 0·66 | 0·26 | 0·07 | 0·34 |
| 1/4 | 1/2 | .. | .. | 9 ,, | 1·01 | 2·56 | 0·64 | 0·18 | 0·07 | 0·39 |
| 9/32 | 3/8 | .. | .. | 10 ,, | 0·77 | 2·91 | 0·68 | 0·27 | 0·06 | 0·45 |
| 1/4 | 5/16 | .. | .. | 8 ,, | 0·66 | 2·84 | 0·62 | 0·15 | 0·05 | 0·43 |

"0" under thermal test means "Did not crack."

Columns 1 and 2 give the depth of chill; column 3, blows of drop test (if tested in this way); column 4, time of cracking through minutes (if tested thermally); and column 5, length of service given by wheel. The analyses are given in the succeeding columns.

Without going into a detailed discussion of the problems presented by these figures, it can be stated briefly that the good wheels for the most part show a high percentage of graphitic carbon, while the poor wheels are low in it. The combined carbon as a rule follows an inverse ratio. The effect of the silicon is not strongly marked, and there is no distinct ratio between it and the graphitic carbon. Considering the thermal and drop-tests, the amount of manganese is important, since the higher the percentage of this element, the better the wheel stands the test. There is not sufficient silicon present to mask the effect of manganese, and it seems quite certain that in these metals the manganese has a greater influence on the final condition of the carbons than on any of the other non-metals.

The effects of manganese on steel are pretty well known, but its influence on low silicon, high carbon irons is not yet positively determined. By influencing the amount of temper-carbon, and by possibly forming a carbide itself, it has a strong influence on the properties of a chilling iron. The sulphur and phosphorus, in the limits within which they occur in the analysis shown, do not affect the wheels sensibly, but the limits for both should be made narrow, and the maximum low. The effect of these elements is the same upon wheel iron as it is upon other products of the cupola. As noted before, the silicon varies within such small limits, that it in itself seems to have but little influence, and the silicon must of necessity be low, best under 1 per cent.

Selecting then from these wheels those showing desirable results, the following classification is obtained:—

| | Thermal Test. | | Drop Test. | | Service. | |
|---|---|---|---|---|---|---|
| | Max. | Min. | Max. | Min. | Max. | Min. |
| Graphite | 3·28 | 2·65 | 3·31 | 2·65 | 3·48 | 2·23 |
| Combined carbon | 0·95 | 0·32 | 0·90 | 0·55 | 1·24 | 0·56 |
| Silicon | 0·75 | 0·50 | 0·70 | 0·50 | 0·94 | 0·58 |
| Manganese | 0·53 | 0·21 | 0·48 | 0·24 | 0·34 | 0·13 |
| Sulphur | 0·088 | 0·055 | 0·086 | 0·040 | 0·085 | 0·047 |
| Phosphorus | 0·48 | 0·35 | 0·52 | 0·36 | 0·49 | 0·25 |

Continuing these results, the following figures are obtained, and these represent the objective specification:—

| | |
|---|---|
| Graphite | 2·75–3·00 |
| Combined carbon | 0·50–0·75 |
| Silicon | 0·50–0·70 |
| Manganese | 0·30–0·50 |
| Sulphur | 0·05–0·07 |
| Phosphorus | 0·35–0·45 |

The total carbon should run about 3·50 per cent., and the silicon and manganese together 0·90 to 1·00 per cent.

The adoption of this specification has proved of advantage, in that it secures a high average in all the three requirements. It is manifestly possible to get a longer life from a wheel, under favourable conditions, which runs higher in combined and lower in graphitic carbon than this specification shows, but that which adds to its hardness and capability of resisting abrasion, detracts from its strength and ability to withstand shocks as already noted. The factor of safety must be considered as the most important in this question, making the wearing qualities subservient to this property, and the specified analyses of materials and product shown, are constructed to obtain the best results in this direction.

In order to show that these limits apply to wheels which have given good service, the last seven wheels in Table No. 6 were taken from scrap shipments at random and analysed, the records showing that the specified limits cover nearly all the points in these wheels.

Further to determine whether these limits would exclude poor wheels, there were selected for this purpose wheels that (*a*) broke through the rim in 10 minutes or less under the thermal test; (*b*) broke with 20 blows or less under the drop test; and (*c*) gave but two years' service or less. Of 22 wheels—five being in group "A," 17 in group "C," and none in group "B"—all but one failed to come within the limits suggested, and this one was in group "A."

These observations, therefore, lead to the conclusion that the analytical limits fixed as shown, will represent strong, safe wheels, wheels that will yield good service.

Mr. G. R. Henderson, M.E., co-operated in the physical part of this work.

## SOME PHOTOCHEMICAL FACTS AND THEORIES.

BY DR. LEO. BAEKELAND.

Some 20 or 25 years ago, the invention of the dry-plate very largely increased the possibilities of photography, and, as a result, a new industry was born. This industry started on a very modest scale: the first dry-plate manufacturers coated their plates by hand. After a while the consumption increased, and special machinery had to be invented for this purpose. The simplicity and new possibilities of the dry-plate produced a demand for an easier process of printing. Up to that time the old albumin process had been sufficient, but now those accustomed to the facilities of ready-made plates wanted also ready prepared paper. The great objection against albumin paper was that it had to be sensitised before it could be used, and could not be kept any length of time, and, for amateur photographers, this was specially objectionable. Shortly afterwards were put on the market some ready-sensitized papers, which could be kept for a long time, and which were prepared by machinery. Those papers were sold under the name of Aristotype papers. Some of these papers were coated with an emulsion of gelatin, which kept in suspension chloride of silver, combined or mixed with citrate of silver, or some other organic silver salt. Since then the number of these papers has increased enormously. Each manufacturer has his own ways of preparing his papers, and one uses one kind of silver salt and another a different one, but the base of such papers is always chloride of silver in addition to one or more organic silver salts. With this is mixed a certain amount of free acid in order to give the necessary keeping qualities. This free acid is usually some organic acid, mostly citric acid, sometimes tartaric acid, sometimes other acids which it is needless to enumerate here. These acids, of course, are taken with due consideration to their general properties, not only as enabling the paper to be kept for a certain time, but also in relation to the colour of the image, and the general printing and toning qualities of the paper. Other manufacturers, instead of using gelatin as a vehicle for carrying the silver salts, prefer collodion. Both collodion or gelatin have advantages and disadvantages. All the papers to which I have referred until now are known under the name of printing-out papers. They are called so because the image shows up directly so soon as the paper is submitted to the action of sunlight under a negative. But there is another class of papers called developing papers, and these developing papers do not show any image after exposure to light; the image appears only after developing, just the same as when making a negative on a dry plate. These developing papers are prepared with an emulsion more or less similar to those used for dry plates, that is to say, with a bromide of silver emulsion, with or without the addition of silver chloride. Both classes of papers have each their advantages and their drawbacks. The printing out papers can only be used by daylight. It takes some time, especially if the weather is cloudy, before a print is obtained, and after printing it is necessary to go through a series of delicate manipulations, washing, toning, fixing, &c. When Aristotype papers came into use the manufacturers tried to render the process as simple as possible, and therefore recommended a combined bath, which did the toning and the fixing at the same time. Since then, however, time has proved that none of those combined baths can be relied upon if a lasting photograph has to be produced. Most prints made on such papers fade and disappear; and certain disappointed photographers went back to the old albumin process, under the impression that those new machine-made papers could not give a permanent image. The trouble was really not due to the paper, but to the process of toning, because shortly afterwards, when the same old methods of toning in separate baths were applied to the new printing-out papers, it was possible to obtain permanent prints as well as with the albumin process. Beside this question of permanency, all these printing-out papers are at their best a very slow and a very tedious way of making photographs. It was thus natural that efforts should be made to obtain a quicker printing process by utilising the extreme sensitiveness of silver bromide by means of papers coated with bromide of silver emulsion. One of the objections to ordinary bromide paper is that its range of tones is rather limited, and that it has to be worked in dark rooms with a ruby light. People prefer rather a slower process, which can be worked in any room. But even this objection has been eliminated by the introduction of a variety of very slow bromide paper, better known under the name of "Velox," which is slow enough to enable us to handle it by ordinary gaslight in the evening, and quick enough to give an image in a few seconds. In the manufacture of photographic papers a good raw material is entirely indispensable. The raw paper from which photographic papers are made is the very best that can possibly be made; linen paper made with pure linen rags properly assorted. The manufacture of this raw paper is the monopoly of two firms in Europe—one in Germany and one in France. They are practically the only producers of this paper in the whole world, and as the consumption of these papers is increasing every year these two firms have, of course, a very desirable monopoly. In the process of manufacture of these papers it is necessary to avoid the use of such chemicals which would prove injurious to the silver salts or the photographic image; chloride of lime and hyposulphite of soda are equally dangerous here. For all purposes of washing, none but the very best quality of water can be used. As far as I know, those manufacturers who make photographic papers have the advantage of possessing some springs which produce in abundant quantity water that is nearly chemically pure. During the whole process of manufacture, the introduction of metallic impurities in the stock is carefully avoided. Hot boxes, grinding gears, and possible introduction of greasy matter, are only some of the possible troubles. The smallest particles of iron or any other metal produce much mischief to the manufacturer and the consumer. You can readily see, therefore, that it would never pay to use anything but the best material; even the very best is not good enough. This paper is imported either in its natural condition or with a preliminary coating with blanc fixe or sulphate of baryta. Most sensitisers receive the paper in the latter condition, and apply directly the sensitive emulsion to it. Troubles, however, arising from this preliminary baryta coating occur sometimes, and have become so serious that some sensitisers have started to do their own baryta coating. Such paper is sold in big rolls of various widths. Some manufacturers prefer narrow rolls; others prefer wide rolls, 41 ins. or 54 ins. This paper has a good strength, but at the same time is remarkably pure, and is very uniform in its texture. The good qualities of the paper are specially noticeable when it is soaked in water. It has already been super-calendered to give it a smoother surface. Ordinarily this paper is coated afterwards, by means of special machinery, with a mixture of baryta and gelatin. This has for object to produce a superficial layer, which fills up the pores of the paper, and equalises its surface and prevents the silver emulsion from soaking into it. This baryta coating produces a matt surface, which can be used as it is for matt-surface papers, which lately have become rather popular. If, on the contrary, the paper is intended for the glossy variety, it will have to be run again through special cylinders. Those cylinders are very heavy machinery weighing as much as 14 tons, which may require as much as 30 to 40 horse-power. They consist of a set of rolls of about 14 to 16 ins. diameter and about 46 ins. in length. There are usually about seven of these rolls, which lie one on top of another. By means of mechanical arrangements, high pressure can be exerted on those cylinders so as to press them together. Those rolls alternate, so that next to an iron roll comes a cotton roll. The lowest roll is of chilled iron, the next roll is of cotton obtained by pressing raw cotton on to a shaft by hydraulic power. These cotton rolls are afterwards turned down and polished on the lathe, so that they finally become as smooth as polished ivory. The paper is run several times in succession between those cylinders until its surface has become glossy enough.

All the operations which I have described hitherto are done in full daylight. The sensitising emulsion is now to be applied to the paper, and from this time the whole manufacturing process is performed in dark rooms, where non-actinic light is used. For printing-out papers which are not very sensitive, ordinary lamplight or incandescent light will do no harm; for bromide papers, however, none but ruby lamps can be used. Electric lamps with ruby bulbs are most convenient for this purpose.

Bromide of silver emulsion is a solution of gelatin in which is suspended very finely divided bromide of silver. The preparation of this finely divided bromide of silver is one of the delicate parts of this industry. Many of you are aware that a number of years ago, when Stas made his memorable researches on atomic weights, he described several different varieties of bromide of silver. At that time this great chemist was certainly not aware that these varieties of bromide of silver to which he called our attention were one day going to become the origin of a very important industry. Since then it has been shown that, instead of the few varieties which he described, there are a good many more—some of them very important from a photographic standpoint, other ones without any use at all. Some varieties are not sensitive enough to light; some other ones, again, are too sensitive, and on account of that have to be excluded, because it is not possible to handle them practically. The production of a suitable variety of bromide of silver is the object of the emulsion-maker. I have here some bromide of silver emulsion just as is used for bromide paper or dry plates. It is in solidified condition; heat will liquefy it, and when in this condition it can be applied to paper. In the daylight the colour is a very light green; in the evening this colour is somewhat indistinct. Bromide of silver is so sensitive, that light, even during a very small fraction of a second, will entirely change its general properties, and it is constantly liable to be spoiled during its process of manufacture. Not only is it necessary to use none but the very best and purest chemicals and gelatin, but certain conditions of temperature have to be maintained with utmost care. Furthermore, these emulsions must be used within a certain time, as otherwise their quality would change considerably. The kind of gelatin used for bromide emulsions has a very clean appearance, but certain cheap kitchen gelatins look a good deal better. Its main qualities are not judged by its looks, but by its chemical and physical qualities. One of the main points in this photographic gelatin is that it should have a neutral reaction. It should not contain any products which would have a reducing action on silver salts. It should contain no chlorides, and as few inorganic products as possible; it should be as colourless and as clear as possible; it should especially not contain any greasy matters. A very important factor in the quality of a gelatin is its viscosity. Under certain conditions gelatin will flow and distribute itself very well on glass or paper; in other conditions it will behave very differently and be repelled by the surface of the paper. Some batches of gelatin having apparently the same chemical composition, showing very good chemical tests, may prove entirely worthless from an emulsion-making standpoint. Inexperienced people are then liable to think that there is grease in such a gelatin; but careful chemical examination shows that this is not the cause of the trouble. It seems to be a loss of viscosity which produces this result. I have tried more than once to find a method for determining beforehand the viscosity of photographic gelatin, and have tried one after another the different viscosimeters which have been proposed by various authors. Up to this time, however, I have never been able to find any viscometric method which was sensitive enough for this particular purpose. The only test I know of in order to decide whether a certain kind of gelatin is good or not, is to make some emulsion with it after it has answered the requirements of a preliminary chemical test. In order to make an emulsion the gelatin is dissolved in water, and to this solution a soluble bromide is added. Some prefer bromide of potassium; some others, bromide of ammonium; some others again think they have discovered a very great thing by using other bromides which have only imaginary advantages. This gelatin solution, to which the bromide has been added, is brought now to a certain temperature. Then it is mixed with a solution of nitrate of silver. This mixing is usually done by using a stirring apparatus; and all this sounds very easy to do on a small scale, but if some hundreds of ounces of silver have to be emulsified at one time it becomes a very serious operation. It is so very easy to lose a whole batch of emulsion. If a solution of nitrate of silver is mixed with a solution of bromide of potassium, to which gelatin has been added, the precipitate of bromide of silver will remain in suspension instead of going to the bottom, as would be the case if you take plain water solutions. If these solutions are of the right concentration, not too strong, nor too diluted, it will be possible to produce very finely divided bromide of silver which will not settle even after very long standing. Neither would it be possible to retain some on a paper filter by filtering the emulsion, even if this were done several times in succession. It becomes a very hard matter to separate this bromide of silver. In order to separate it at all, it would be necessary to use a centrifugal machine or to submit the emulsion to boiling during several hours. The emulsion, as it is obtained now, is not by any means sensitive enough for ordinary photographic purposes. Its sensitiveness has to be increased. This is done in several ways; for instance, by keeping it near the boiling point of water for a certain time, thus changing considerably its properties. Not only has it become more sensitive to light, but, at the same time, the size of the particles has increased. This change is ordinarily designated under the name of ripening. In some other methods, this ripening is obtained at a lower temperature by adding a certain amount of ammonia to the emulsion. Why this ammonia should hasten the ripening in such an extraordinary way is still a question open to discussion. In so far as this condition of sensitiveness of bromide of silver seems to be accompanied by a change in the size of the particles of this salt, it is very possible that the noticeable solubility of bromide of silver in ammonia has very much to do with this action, and it would then be easy to explain why the fact of boiling the emulsion should produce the same result. You know that bromide of silver becomes somewhat soluble at high temperature in certain salt solutions, such as alkaline bromides. Very probably when a bromide of silver emulsion is being heated those particles undergo partial solution, followed by reprecipitation, and in this way they acquire larger dimensions about in the same way as a precipitate of barium sulphate becomes granular if boiled long enough with some saline solutions. After the bromide of silver has been ripened to the proper degree by one of the above-described methods, it has to be freed from any soluble salts which are still in the mixture. Indeed, when nitrate of silver is mixed with bromide of potassium, we obtain, beside silver bromide, an equivalent amount of nitrate of potassium; furthermore, there is always used some excess of bromide of potassium, and this, too, will have to be eliminated. These soluble salts would have a very disturbing action on the general properties of the emulsion. One of the ordinary methods for eliminating these soluble salts is washing. In order to do this the emulsion is solidified by cooling, cut up in shreds, and then can be washed in running water for several hours. The washed emulsion jelly is melted and brought up to a proper temperature; it then goes to the coating rooms, where it is applied to the paper.

The coating machinery which is used for photographic paper varies somewhat in construction. Usually those machines agree in one point; that is, the sensitive emulsion flows into a silver pan, of which the length is a few inches more than the width of the paper. The paper is in rolls of 1,000 to 2,000 yards long. The machinery draws the paper through the silver emulsion; then the coated paper is drawn further over a rubber blanket, which carries it in a horizontal position while the emulsion is setting on the paper, this result being obtained by means of refrigerating machinery. The coating rooms are kept the whole year round at a constant temperature, which for some purposes may be as low as 54° F. By means of an automatic machine the coated paper is folded and placed on a hanging rack, where it hangs down in loops so as to enable it to dry. These coating rooms are from 200 to 300 ft. in length, so that by the time the loops have advanced slowly to the back end of the room, the paper is ready to be wound up in rolls. In these coating rooms all employees are compelled to wear white gloves, as bare fingers might injure the sensitive coating. In the plant of the Nepera Chemical Co., we have done away as much as possible, on all machinery, with greasy bearings and belting. We use ball bearings and electric transmission as much as possible, and this renders work much cleaner, and guards against dust and oil. After the paper is dry, it is wound up in rolls of about 14 ins. diameter, which are run now through rotary cutters, which cut up the paper in smaller rolls of 20 or 16 ins. wide, or any other desirable width. These rolls in turn are cut in large sheets by means of a sheet-cutter; all these machines are run by steam or electric power, and are watched by employees who have very little to do but to start and stop the cutters whenever a roll goes in or out. These sheets are piled now one upon another, and each pile of about 500 sheets is trimmed down to smaller sizes by a power trimmer. Each trimmed sheet, however small it may be, has to be examined carefully before it is put in a package, so as to eliminate any imperfect paper. Most imperfections are due to the raw paper. Some cannot be detected even by careful examination, and will only show afterwards after the image is developed. This assorting of the paper is done by girls, who examine each sheet by an incandescent electric light, and throw out any defective sheet. Here, too, white gloves are indispensable.

I ought to say something about the many sources of trouble in this industry. The raw materials, in themselves, are rather variable in quality, and, furthermore, the manufacturer is bound to deliver to the consumer a product of good and uniform quality. He is expected always to deliver the same paper; and if one takes into consideration the variations of the raw material and, of climatic conditions, this becomes a difficult problem. Some five years ago, consumers of photographic paper were easy to satisfy; but since then competition has not only improved the standard of quality, but selling prices have been cut down considerably, while the price of most all raw material has increased. In consequence, profits have become so small that, in order to make this industry profitable, you are obliged to manufacture a tremendous amount of paper during the year in order to cover general expenses; and if any non-successes in manufacturing occur, losses become very serious. Formerly, many an amateur who once had made a few dozen plates or a few sheets of paper to his own satisfaction and to the satisfaction of his own friends, thought he had in himself the nucleus of a very profitable enterprise, and started to manufacture on a larger scale, and more than once he found out, after many disappointments, that things did not go so easily as he had anticipated. The conditions are about the same in this industry as in every chemical industry. It requires a steady, watchful eye, and, at the same time, very thorough business methods, to make a success. In order to give you some idea of the perplexing problems that come up once in a while, I may say that, a few years ago, it happened that one of our customers wanted, for a special purpose, a large amount of bromide paper which had to be coated on a special kind of raw paper. This raw paper was of the "enamelled" variety, *i.e.*, paper previously coated with a layer of baryta. We ran the paper through our bromide emulsion coating machines, and at the same time coated a quantity of our regular stock with the same emulsion. During these operations nothing unusual was noticed. When, however, the paper was tested, it behaved in a most extraordinary way in the developer. All over the prints were scattered millions of little white spots. The smallest of the spots were about the size of a point of a needle; the largest ones were not over 1 mm. in diameter. Some of them were slightly round, while others were slightly elongated. Anyone who has manufactured bromide paper in very large quantities knows that similar spots may be produced by numerous causes, among which are included defective emulsions, defective mechanical appliances, dust, &c. In this instance, however, the cause of the trouble had to be looked for elsewhere, in so far as the spots showed only on this particular raw paper, and they appeared regularly whenever this paper was used, while the same coated emulsion, at the same time and under identical conditions, on the regular stock paper invariably produced an excellent article, entirely free from the imperfection mentioned above.

Taking into consideration that the spots only manifested themselves during development, I surmised that they were probably due to the aggregates of unreduced bromide of silver, which, for some reason or another, had lost its sensitiveness to light, and which, therefore, did not respond to the reducing action of the developer. Indeed, the paper before developing showed a very uniform appearance, and it was impossible to detect anything abnormal in the coating by merely looking at it.

However, to settle this question beyond a doubt, I determined to make cross-cuts of the spots, and to examine the section of the film under the microscope, so as to ascertain whether they were produced by any depression in the film, air bubbles, or similar mechanical imperfections. In order to do this, I proceeded in the following way:—

I cut out of the prints, and in such places where the white spots were most apparent, small strips of about 5 mm. long. I embedded these strips in paraffin while holding them in an upright position, and after the paraffin was hardened, I made cross-cuts of the strips of paper by means of a microtome, following the methods employed in microscopical laboratories.

These cross-cuts were only a fraction of $\frac{1}{1000}$ in. thick, and could easily be examined under the microscope. This enabled me to observe successively many cuts of the same strip, until the razor of the microtome reached the centre of the spot. Thus I was able to satisfy myself that the film presented everywhere an even thickness on any section across the spot, and that there was no depression of the film at the place where the spot was located. At this spot silver particles were entirely absent in the film, for the reason that the hypo had dissolved the "irreducible" or unsensitive bromide of silver which had existed there. But the whole structure was such as to show that the layer of the emulsion was originally perfectly homogeneous.

Examination by reflected light under the microscope, using moderately magnifying power, had not shown any dust or foreign matters where the spots existed. In some of the white spots, however, I was able to detect a central black nucleus, faintly visible. By gently scratching the film away, with a small dissecting scalpel, so as to lay bare the underlying coating of baryta, I was able to render this black nucleus more apparent; when I had also removed the layer of baryta and had reached the actual paper base, the black nucleus could be plainly observed, although it was still very small. In many cases it was impossible, even by using magnifying power, to detect this black nucleus at all. In order to determine what this black nucleus was, I looked patiently over large quantities of defective prints, preferably in which the white spots were largest. By doing so, I was able to collect a few spots where the black nucleus was readily observable, and these I proceeded to dissect, as previously described. By the skilful use of the dissecting needle I was able to isolate a few of these black particles under the microscope. At first I was inclined to consider them as of metallic origin. Some of them were elongated. When treated with a drop of dilute hydrochloric acid, they showed at once under the microscope a yellow bronzed appearance, as copper would do; by treating this drop of hydrochloric acid after neutralisation with a ferrocyanide of potassium, I was able to prove, to my entire satisfaction, the characteristic reaction for copper. It was, of course, impracticable to submit the smallest particles to the same treatment, but, reasoning by analogy, and taking into consideration the identical shape and conformation of the small white spots, I had very good reason to believe that they were produced by the same cause. A noticeable amount of copper was also found in the ashes of all the defective prints. Those who have given thought to the metallic particles found in photographic paper, would probably wonder that such particles should produce white and not black spots. This, at least, was the idea of almost all to whom I mentioned the matter, and I must confess that, up to the time I made the above researches, I held the same opinion. Everyone who has used printing-out papers has at one time or another observed some very well defined black spots caused by a metallic splinter under the sensitive layer; and these spots are commonly known as "metallic spots." They are indeed metallic spots, but in printing-out papers they show themselves very shortly after the paper is made, and they are so prominent that they can be seen before printing. They appear so much the easier in such cases because the latter carry a certain amount of silver in a soluble condition, and, furthermore, the reaction of printing-out papers is already acid. These conditions favour a rapid and complete action between any metallic particle and the sensitive layer. In bromide paper, however, the conditions are totally different. The reaction of these papers is supposed to be neutral, and the silver contained therein is in the insoluble condition of bromide of silver, so that the action of a developer is required in order to bring out the fact that metallic particles are present. I have not found any other way to explain all this than by the following theory:—

Whenever a metallic particle is embedded in or near a layer of silver emulsion, it will create a special electrical condition by chemical action. The metallic particle will become "electro-negative," while the surrounding zone becomes "electro-positive." As a result, the metallic particle will cover itself with silver, and will be gradually dissolved and entirely replaced by an equivalent amount of the latter metal, and this will make a black nucleus. The zone around the metallic particle being electro-positive will liberate a very small amount of oxygen or bromine, or chlorine, or both together, and the immediate result is, that this zone becomes insensitive to light, consequently will remain undisturbed by the developer, producing there a white spot. It is a known fact, that any electric current by electrolysis will decompose bromides or chlorides, setting free the chlorine on the positive-electrode, while the metal will be liberated at the negative electrode. In order to verify my views in this matter, I made the following experiments. In a dark room, a sheet of bromide paper (unexposed to light) was placed in a small tray covered with water, to which was added a very small amount of bromide of potassium in order to increase its conductibility. In contact with this paper I fastened two platinum electrodes. These were so disposed that their points touched the paper slightly, and were situated at about $\frac{1}{4}$ in. distant from each other. These electrodes were connected with one single Leclanché cell and left in circuit for about 20 minutes. After this time I removed the electrodes, washed the paper, and exposed it for one or two seconds to the action of an ordinary lamp so as to fog the paper. I then developed the sheet, with ordinary metol-quinol developer. The paper developed all over its surface to a greyish-black, except in those two places where the electrodes had touched. The position of the positive electrode was indicated by an insensitive spot, so that the developer left this place entirely white. On the contrary, where the negative electrode had touched the paper there was a sharp black point indicating the reducing action of the negative pole. This experiment I repeated several times with the same result. I was able to produce artificially the same defect as the one referred to above, with the difference, however, that the black nucleus corresponding to the electro-negative zone was situated outside of the insensitive electro-positive zone. When a metallic particle is embedded in the paper or in the film, this creates a little microscopic electric cell, and the metallic particle being electrified negatively, produces the same phenomenon as the negative electrode referred to in the above experiment. The surrounding emulsion film becomes then the positive electrode of this tiny electric cell. There seems to be one objection to this explanation. It is that when printing-out papers contain a black metallic spot in the film they do not generally show the white insensitive zone to which I refer. This is indeed an objection which has been made to me. A close investigation of the facts has, however, entirely corroborated my views in a most convincing way. When a metallic spot appears in a printing-out paper only the black spot is visible, and no white zone appears around it, but only as long as the paper is very fresh. If, however, the paper is kept long enough so as to show pronounced symptoms of discoloration, then the white halo appears also. From that moment on, the general conformation of the spot becomes entirely similar to the smaller spots observed in the defective bromide paper. The only difference consists merely in the size. Furthermore, there is a very simple way of showing the real existence of this insensitive zone around the metallic spot even in fresh printing-out paper. All that is necessary to do is to put a sheet of the paper in a solution of diluted developer. The paper will darken at once, except in the zone immediately surrounding the metallic spot, thus producing a white halo, in every respect similar to those observed in bromide paper.

# Nottingham Section.

*Meeting held at Derby on Wednesday, November 30th,* 1898.

MR. J. O'SULLIVAN IN THE CHAIR.

## NOTES ON THE ARACHIDIC AND LIGNOCERIC ACIDS OF EARTHNUT OIL, AND THEIR ESTIMATION.

BY L. ARCHBUTT, F.I.C., F.C.S.

The fatty acids isolated by Renard from earthnut oil (Comptes Rend. **73**, 1330), and described by him, after Gössmann, as arachidic acid, have been shown by Kreiling, (Ber. **21**, (1888), 880), to contain also lignoceric acid. Reimer and Will, who were the first to isolate a sparingly soluble fatty acid from rape oil (Ber. **20**, (1887), 2385), believed that they had obtained behenic acid, basing this conclusion solely on the results of three badly agreeing analyses by combustion, though a mixture of arachidic and lignoceric acids might have given just the same results. Ponzio (J. für. pr. Chem. **48** (1893), 487) subsequently shewed that what Reimer and Will had obtained was arachidic acid. The following are the formulæ, molecular weights, and melting-points of these three acids:—

| Acid. | Formula. | Molecular Weights. | Melting Point. |
|---|---|---|---|
| | | | C. |
| Arachidic ....... | $C_{20}H_{40}O_2$ | 312·3 | 75·5 |
| Behenic....... ... | $C_{22}H_{44}O_2$ | 340·3 | 76·0 |
| Lignoceric ... ... | $C_{24}H_{48}O_2$ | 368·4 | 81·0 |

I have determined the mean molecular weight of the fatty acids obtained from earthnut oil by Renard's process. 1·800 grm. of the once recrystallised acids (melting-point 71°–72°) required for neutralisation 50·87 c.c. of decinormal potash proving the mean molecular weight to be 354·6, which would be that of a mixture containing arachidic acid 26·4 per cent. and lignoceric acid 73·6 per cent. In another experiment, 0·7595 grm. of the mixed acids neutralised 21·42 c.c. of decinormal potash, shewing the mean molecular weight to be 354·6.

After recrystallising the mixed acids many times from 90 per cent. alcohol, the melting-point was raised to 79·6° (corrected). 0·6318 grm. of the acid thus obtained neutralised 17·14 c.c. of decinormal potash, proving the molecular weight to be 368·5, which is that of pure lignoceric acid. It appears from these experiments that lignoceric acid forms the larger proportion of the fatty acids obtained by Renard's process from earthnut oil. I have not determined the mean equivalent of the acids obtained from rape oil by this process, but, judging from their solubility and melting-point, they are most probably a similar mixture of arachidic and lignoceric acids.

### *Solubility of the Mixed Acids in Alcohol, 90 per cent. by Volume.*

As arachidic and lignoceric acids are not entirely insoluble in the alcohol which is used to isolate and purify them, it is necessary to make a correction for the amount dissolved. According to Renard this correction, per 100 c.c. of 90 per cent. alcohol used for crystallisation and washing is:—

| | Grm. |
|---|---|
| At 15° C. ............................. | 0·025 |
| At 20° C. ............................. | 0·045 |

These numbers have hitherto been accepted without question. But in a recent paper by Tortelli and Ruggeri (this Journal, 1898, 876) it is shewn that Renard's correction is too low, and that the allowance to be made varies somewhat according to the weight of mixed acids obtained. As I suspected from my own experiments that Renard's correction was too small, I thought it worth while to check Tortelli's figures. For this purpose, the crystallised acids obtained from 20 grms. of pure earthnut oil were filtered under gentle suction on a Gooch filter, washed three times, with 10 c.c. each time of 90 per cent. alcohol, and then thoroughly with 70 per cent. alcohol, then redissolved in 100 c.c. of hot 90 per cent. alcohol, recrystallised, and again filtered and washed as before. I satisfied myself by preliminary experiments that after this purification nothing but arachidic and lignoceric acids remain. The weight of the purified acids was 0·7649 grm., and the melting-point by capillary tube (uncorrected) was 72·5° C. These acids were dissolved in 100 c.c. of 90 per cent. alcohol by heating in a corked flask containing a thermometer, and the clear solution was then cooled to 20° C., and after remaining at that constant temperature for one hour with frequent agitation, was filtered through a dry filter paper, rejecting the first portions. 25 c.c. of the filtrate were then evaporated, and the residue weighed. The remainder of the liquid was further cooled to 15° C., kept at that for one hour, and again filtered. 25 c.c. of this second filtrate were also evaporated, and the residue weighed. The results obtained were:—

| | Dissolved by 100 c.c. of 90 per Cent. Alcohol. |
|---|---|
| At 15° C. ........................ | 0·071 grm. |
| At 20° C. ........................ | 0·095 grm. |

These numbers pratically confirm Tortelli's higher figures. By recrystallizing these acids several times it was found that the solubility gradually diminished, and was finally reduced to the following:—

| | Dissolved by 100 c.c. of 90 per Cent. Alcohol. |
|---|---|
| At 15° C. ........................ | 0·033 grm. |
| At 20° C. ........................ | 0·046 grm. |

confirming Tortelli's lower figures. The acid having this lowest solubility, practically agreed in melting-point and molecular weight with pure lignoceric acid (*v. supra*).

The recrystallised acids obtained from Guzerat rape seed (this Journal, 1898, 1009), and which weighed 0·1908 grm., had the following solubility:—

| | Dissolved by 100 c.c. of 90 per Cent. Alcohol. |
|---|---|
| At 15° C. ........................ | 0·049 grm. |
| At 20° C. ........................ | 0·070 grm. |

The general correctness of Tortelli's and Ruggeri's numbers being thus confirmed, I have constructed from them the following table of corrections, which is more strictly in accordance with their experimental results than the one they publish.

| Weight of Fatty Acids obtained by Renard's Process. (grm.) | Correction, per 100 c.c. of 90 per Cent. Alcohol used for Crystallisation and Washing. (grm.) | | |
|---|---|---|---|
| | 15° C. | 17·5° C. | 20° C. |
| 0·1 or less............ | + 0·033 | + 0·039 | + 0·046 |
| 0·2 " ............ | 0·048 | 0·056 | 0·064 |
| 0·3 " ............ | 0·055 | 0·064 | 0·074 |
| 0·4 " ............ | 0·061 | 0·070 | 0·080 |
| 0·5 " ............ | 0·064 | 0·074 | 0·085 |
| 0·6 " ............ | 0·067 | 0·077 | 0·088 |
| 0·7 " ............ | 0·069 | 0·079 | 0·090 |
| 0·8 " ............ | 0·070 | 0·080 | 0·091 |
| 0·9 and upwards...... | 0·071 | 0·081 | 0·091 |

*Estimation of Arachidic and Lignoceric Acids.*

In Renard's description of his process he directs that the fatty acids from 10 grms. of oil are to be dissolved in 50 c.c. of 90 per cent. alcohol and precipitated with lead acetate; this precipitate is then filtered off and treated with ether, to dissolve out the oleate of lead, and the arachidic and lignoceric acids are subsequently obtained from the insoluble lead soaps. Renard does not say how much lead acetate is to be used for precipitation, and some chemists, including Tortelli and Ruggeri, neutralise the fatty acids with potash, or, what comes to the same thing, they neutralise the original soap solution with acetic acid, and precipitate the whole of the soaps with lead acetate in excess. I am convinced that this is a mistake. Whether Renard intended it or not I cannot say, but undoubtedly the most expeditious and accurate method is by fractional precipitation, adding the lead acetate to the solution of the *free* fatty acids, and using only 1 grm. of lead acetate for every 10 grms. of the oil operated upon. This proportion of lead brings down the whole of the arachidic and lignoceric acids as lead soaps, and leaves the greater part of the oleic acid dissolved in the alcohol. Not only is there then a smaller precipitate to manipulate and less oleate of lead to be dissolved out by ether—a tedious process,—but, owing probably to the diminished solvent action of the lead oleate solution upon the other lead soaps, actually more arachidic and lignoceric acids are obtained than when the whole of the lead soaps are precipitated. The process which I recommend is as follows:—

Saponify 10 grms. of the oil in a deep porcelain basin, using 8 c.c. of aqueous caustic soda solution (50 grms. NaOH in 100 c.c.) and 70 c.c. of rectified alcohol. Cover the basin, simmer gently down to about 20 c.c., rinse with hot water into a separating funnel, decompose with HCl in excess, and shake with ether to dissolve the fatty acids. Two extractions with ether are sufficient. After washing the ethereal solution with water distil it down in an 8-oz. flask, dry the fatty acids by heating the flask on a steam bath and sucking out the vapour, and then dissolve the acids by pouring 50 c.c. of 90 per cent. alcohol into the hot flask.

To the solution, which must not be allowed to cool below about 100° F., lest crystals of lignoceric or arachidic acid should separate, add 5 c.c. of a 20 per cent. aqueous solution of lead acetate, then cool to about 60° F., agitate, allow to stand for half an hour, filter, and wash once with ether. Do not attempt to wash more than once on the filter, but rinse the soaps back into the flask with ether from a washing bottle and digest in the flask with ether for a short time, then again filter and again rinse back. After doing this about four times altogether the lead oleate will have been entirely dissolved out.

Open the filter paper containing the insoluble lead soaps in a large plain funnel placed in the neck of a separating funnel, and before the soaps have time to dry rinse them into the separator with a jet of ether. The soaps which adhere to the paper and flask are decomposed and transferred by rinsing with hot dilute hydrochloric acid, followed by ether. Pour into the separator about 20 c.c. of hydrochloric acid (1·10 sp. gr.), shake well to decompose the lead soaps, draw off the aqueous liquid, and repeatedly wash the ethereal solution with small quantities of cold water until the lead chloride is removed. Then distil off the ether in an 8-oz. flask and thoroughly dry the residual fatty acids by heating on a steam bath. Finally, pour into the flask 50 c.c. of ethyl alcohol of exactly 90 per cent. strength (sp. gr. 0·8340), close the flask with a cork carrying a thermometer, heat cautiously until the fatty acids have completely dissolved, and then cool the solution to 15° C., when lignoceric and arachidic acids, if present, will crystallise out, either at once or after standing a very short time.

In order to estimate the amount keep the flask for one hour, with occasional agitation, in a water bath at either 15° or 20° C., or at some intermediate fixed temperature which is nearest to that of the laboratory, then collect the crystals on a small filter, using only the filtrate to rinse the flask, and wash the crystals three times with 10 c.c. each time of 90 per cent. alcohol, at the same fixed temperature. A paper filter may be used, but a Gooch filter used with gentle suction is better, as the mother liquor is more perfectly removed and the washing more thorough. The filtrate and washings with 90 per cent. alcohol must be then poured into a measuring cylinder and the acids thoroughly washed with 70 per cent. alcohol, in which arachidic and lignoceric acids are quite insoluble, until some of the washings give no precipitate when diluted with water. These washings are thrown away. It is not necessary, but I quite agree with Tortelli and Ruggeri that it is advisable, to redissolve the fatty acids thus obtained in 50 c.c. of 90 per cent. alcohol and recrystallise them, filtering and washing as before, adding the filtrate and washings with 90 per cent. alcohol to the first quantity in the measuring cylinder. Pure arachidic and lignoceric acids are thus obtained and are dissolved off the filter with boiling ether, distilled down, and weighed in a tared flask after drying at 100° C. for an hour. To the weight obtained is added the amount dissolved by the 90 per cent. alcohol, which is calculated from Tortelli's table (above).

In Table I. the particulars are given of 10 determinations made with the same sample of earthnut oil, five by the above method, using 1 grm. of lead acetate per 10 grms. of oil, two determinations in which 2 grms. of lead salt were used, one in which 4 grms. were used, and two by Tortelli's method in which excess of lead was used and three hours allowed for crystallisation of the arachis acids. The results are summarised in the following table:—

*Percentage of Arachidic and Lignoceric Acids obtained from the same sample of Earthnut Oil.*

| — | By Renard's Process, as described above, using per 10 grms. of Oil | | | By Tortelli's Process, using Excess of Lead Acetate and allowing Three Hours for Crystallisation of the Acids. |
|---|---|---|---|---|
| | 1 Grm. $Pb\bar{A}_2$ aq. | 2 Grms. $Pb\bar{A}_2$ aq. | 4 Grms. $Pb\bar{A}_2$ aq. | |
| | Allowing one hour for Crystallisation of the Arachidic and Lignoceric Acids. | | | |
| Not recrystallised | 4·98 | 4·86 | 4·83 | .. |
| | .. | 5·12 | .. | .. |
| Recrystallised ... | 4·72 | .. | .. | 4·51 |
| | 4·95 | .. | .. | 4·34 |
| | 4·70 | .. | .. | .. |
| | 4·70 | .. | .. | .. |
| Average ...... | 4·81 | 4·99 | 4·83 | 4·42 |
| Melting-point ° C. | 71° to 72°·5 | 70° to 71° | 71° | 72° |

These results clearly prove that 1 grm. of lead acetate is sufficient to precipitate all the arachidic and lignoceric acids from 10 grms. of pure earthnut oil, that the lead acetate in aqueous solution may be added to the free fatty acids in alcoholic solution, producing a crystalline precipitate easy to manipulate, and that for the final crystallisation of the arachidic and lignoceric acids one hour is ample time; all of which facts add materially to the simplification of what is at best a tedious analytical process.

As regards the melting-point of the mixed acids thus obtained, I have not been able to confirm Tortelli's results. He found in all his experiments the melting-point of the mixture to lie between 74° and 75° C. Working exactly according to Tortelli's and Ruggeri's directions I have not been able to obtain a higher result than 73·3° C., which was the "point of complete fusion" by Bensemann's method according to a good thermometer by Hicks, verified at Kew. The melting-points of the recrystallised acids by the ordinary capillary-tube method varied from 71° to 73·5°.

TABLE I.

*Estimations of Arachidic and Lignoceric Acids by different Methods in the same Sample of Earthnut Oil.*

| Weight of Oil taken. | Method. | Volume of Alcohol.<br>Temperature, ° C.<br>Solubility Coefficient. | Arachidic and Lignoceric Acids. Dissolved in the Alcohol. | Weighed. | Total. | Per Cent. | Melting Point by Capillary Tube. |
|---|---|---|---|---|---|---|---|
| Grms. | | | | | | | ° C. |
| 10 | Renard's: 2 grms. $Pb\bar{A}_2$ aq.; not recrystallised | 70 c.c.<br>20°<br>0·082 | 0·0574 | 0·4283 | 0·4857 | 4·86 | 71 |
| 10 | Renard's: 4 grms. $Pb\bar{A}_2$ aq.; not recrystallised | 70 c.c.<br>20°<br>0·082 | 0·0574 | 0·4258 | 0·4832 | 4·83 | 71 |
| 10 | Renard's; 1 grm. $Pb\bar{A}_2$ aq.; not recrystallised | 80 c.c.<br>15°<br>0·0625 | 0·0500 | 0·4480 | 0·4980 | 4·98 | 71 |
| 10 | Renard's: 2 grms. $Pb\bar{A}_2$ aq.: not recrystallised | 90 c.c.<br>15°<br>0·063 | 0·0567 | 0·4554 | 0·5121 | 5·12 | 70 |
| 10 | Renard's; 1 grm. $Pb\bar{A}_2$ aq.: recrystallised | 130 c.c.<br>15<br>0·061 | 0·0793 | 0·3931 | 0·4724 | 4·72 | 71 |
| 10 | Renard's; 1 grm. $Pb\bar{A}_2$ aq.; recrystallised | 135 c.c.<br>15°<br>0·061 | 0·0823 | 0·4125 | 0·4948 | 4·95 | 72 |
| 20 | Tortelli's; exactly as described in Chem. Zeit. 22, 1898, 600 | 265 c.c.<br>17·5°<br>0·079 | 0·2094 | 0·6935 | 0·9029 | 4·51 | 72 |
| 20 | Renard's: 2 grms. $Pb\bar{A}_2$ aq.; recrystallised; Gooch filter used | 250 c.c.<br>15°<br>0·070 | 0·1750 | 0·7649 | 0·9399 | 4·70 | 72·5 |
| 20 | Renard's; 2 grms. $Pb\bar{A}_2$ aq.: recrystallised; Gooch filter used | 240 c.c.<br>15°<br>0·070 | 0·1680 | 0·7718 | 0·9398 | 4·70<br>(Bensemann) | 72<br>72°·8—73°·3 |
| 20 | Tortelli's; exactly as described; Gooch filter used | 248 c.c.<br>15°<br>0·069 | 0·1711 | 0·0967 | 0·8678 | 4·34<br>(Bensemann) | 72<br>72°·3—73°·3 |

Finally, to show the degree of accuracy ottainable by the method I have described, the results of analysis of two mixtures of pure olive oil with 20 per cent. and 10 per cent., respectively, of pure arachis oil are given in Table II. 10 grms. of the oil was the quantity taken for analysis, 1 grm. of lead acetate was used for precipitation, and the mixed fatty acids obtained were not recrystallised.

I have to thank my assistant, Mr. T. H. Adams, for his help in carrying out the experiments recorded in this paper.

TABLE II.

*Results of Analysis of Mixtures of Olive Oil and Arachis Oil.*

| Composition of Oil taken. Olive Oil. | Arachis Oil. | Volume of 90 per Cent. Alcohol.<br>Temperature.<br>Solubility Coefficient. | Arachidic and Lignoceric Acids. Dissolved in the Alcohol. | Weighed. | Total. | Per Cent. | Melting Point. | Arachis Oil found. |
|---|---|---|---|---|---|---|---|---|
| Per Cent. | Per Cent. | | | | | | ° C. | Per Cent. |
| .. | 100 | 80 c.c.<br>15°<br>0·0625 | 0·0500 | 0·4480 | 0·4980 | 4·98 | 71° | .. |
| 90 | 10 | 73 c.c.<br>15<br>0·033 | 0·0241 | 0·0265 | 0·0506 | 0·506 | 71° | 10·2 |
| 80 | 20 | 73 c.c.<br>15<br>0·033 | 0·0241 | 0·0715 | 0·0956 | 0·956 | 71° | 19·2 |

### DISCUSSION.

Dr. STANLEY KIPPING, F.R.S., asked whether the author could explain why the solubility coefficient varied with the weight of acids.

Mr. ARCHBUTT said he could only suppose that, the substance being a mixture of two acids of different solubilities existing in unequal proportions, the solubility might depend upon the mass of alcohol relative to the total mass of acids. In reply to Dr. Sudborough, he stated that in one of his experiments for the removal of the arachidic acid the mixed acids had been recrystallised 18 times. Although nitric acid seemed at first sight applicable and more suitable than hydrochloric acid for the decomposition of the lead salts required to be effected in this process, it might be an objection that nitric acid could not be so easily washed out of the ether employed. He had been unable to obtain the melting-point of the mixed acids so high as that given by Tortelli.

Dr. Sudborough suggested that some difference in the diameter of the capillary tubes employed might account for this.

Mr. Archbutt said Bensemann's method had been used, and besides, he agreed with the Italians in the crystallisation temperature, which proved to him that the discrepancy mentioned could not be due to any variation in the thermometer. The solubility figures had been obtained by analysing mixtures of olive oil and earthnut oil. He quite agreed that it would not do to go on recrystallising indefinitely, because the composition of the mixed acids would be altered. It had to be admitted that the problem was not a simple one.

Mr. R. M. Caven asked whether there was enough in the small quantities obtained in some of the experiments to give a saturated solution.

Mr. Archbutt said the quantities given were those that crystallised out, and therefore the mother liquor must be saturated. All the figures had been obtained by experimenting on the same sample of oil. In reply to Mr. Pentecost, the author stated that earthnut oil, obtained from the pea nut, ground nut, or "monkey" nut, was used for cooking, and for adulterating olive oil. The best qualities were very much like olive oil, and not easily distinguishable from it except by the test described, and by their having a rather higher iodine value than olive oil. The other constants and specific gravity were very nearly the same in both oils. There were, he believed, several kinds of earthnut oil. It was, however, remarkable that the percentage of the sparingly soluble fatty acids present was very constant. Among the more commonly occurring oils, so far as was known, these acids (arachidic and lignoceric) only occurred in appreciable quantity in earthnut oil, in rape oil, and in oil of mustard; they were easily saponifiable.

# Yorkshire Section.

*Meeting held on Monday, November 28th, 1898.*

MR. T. FAIRLEY IN THE CHAIR.

## COLORIMETRIC DETERMINATION OF DISSOLVED OXYGEN IN WATER.

BY W. MCD. MACKEY AND R. E. MIDDLETON.

We describe a colorimetric process for the determination of dissolved oxygen in water, using a form of apparatus devised by one of us, which consists of two separate tubes, one graduated and the other ungraduated, each of the capacity of 265 c.c.

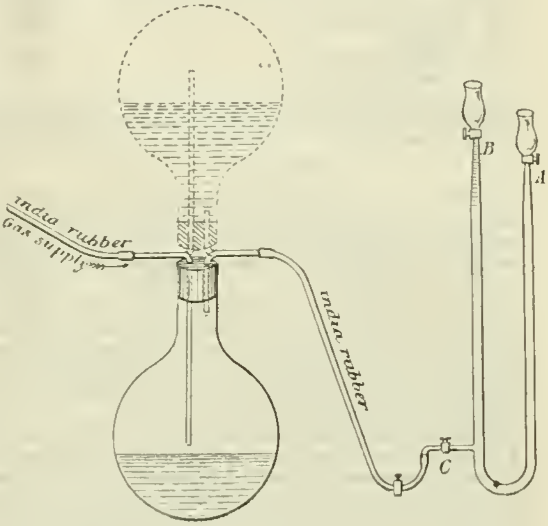
The sample to be examined is introduced into the ungraduated tube, preferably previously filled with coal gas. (If a river water were being examined it would be better to immerse the tube in the river, and open and again shut the taps below the surface.)

By means of the cup tap A, 5 c.c. of a 15 per cent. solution of potash is introduced, and by tap B, 5 c.c. of a 5 per cent. solution of pyrogallic acid, the tap C allowing displaced water to flow out.

Before doing this, approximately 5 c.c. of coal gas has been introduced by tap C; for that purpose inverting the apparatus and opening either tap A or B to allow the displaced water to flow out. This bubble of gas enables the contents of the two limbs to be thoroughly mixed, and a brown colour is obtained varying in intensity according to the amount of dissolved oxygen contained in the sample. The liquid attains its maximum intensity of colour in from 10 to 20 minutes.

Meantime, another portion of the same water (say 500 c.c.) is diluted with distilled water, and boiled down to the original bulk in order to expel oxygen, and just before removing the burner a cork with wash-bottle fittings is inserted in the mouth, the longer tube attached to the gas supply, and the other closed (immediately the burner is withdrawn) by a piece of rubber tube and a pinch-cock. The water thus cools in an atmosphere of coal gas.*

The flask is attached to the graduated tube at tap C, the pinch-cock opened, and gas allowed to pass through the whole apparatus to displace the air; the flask is then inverted (see figure). Dotted lines show flask inverted to fill tube with oxygen-free water.



This gives us a tube filled with water of the same tint as that in the ungraduated tube, but free from oxygen. Pyrogallic acid and potash are added as before, and air gradually introduced at tap B (tap C allowing displaced water to flow out), until after gentle agitation the same depth of colour is obtained as in the ungraduated tube. It is better, when introducing successive quantities of air, to transfer the residual nitrogen to the tube attached to tap A.

The colours are compared by holding the tubes between the observer and the light, much after the method for carbon determinations by colour.

When the depth of colour is the same in both tubes, the residual nitrogen being in limb B of graduated tube, tap A is opened, and, by means of tap C, water drawn off till the water in both limbs has the same level. The residual nitrogen is then read off.

The tubes have each a capacity of 265 c.c., and allowing 15 c.c. in each case for solutions and gas introduced, this gives 250 c.c.; and as oxygen constitutes about one-fifth of the atmosphere by volume, the measure of the residual nitrogen gives c.c. oxygen per litre direct.

Our principal difficulty at first was the difference of tint we obtained, which we began by attributing to the coal gas. We tried nitrogen and hydrogen, with really worse results, owing probably to the difficulty of getting an abundant supply; but we found eventually that we had been using our solutions too weak, and by increasing them to the strength stated we found the tint difficulty disappear.

We append the following table of results. Distilled water shaken with air according to Roscoe and Lunt's directions (see Sutton, page 260 (6th edition)) served us as a standard, and we give all the determinations we made in this direction, using a coal-gas atmosphere. Experiment 5 is the only one that is discordant. We also append a few determinations of Leeds' supply, &c.

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* Note.—The oxygen might be driven out by a current of coal gas, but we prefer boiling.

| Experiment. | Water. | Temperature and Pressure. | | Roscoe and Lunt. | Colorimetric Method. |
|---|---|---|---|---|---|
| | | ° C. | mm. | C.c. of O per litre. | C.c. of O per litre. |
| 1 | Distilled.......... | 15·5 | 751 | 6·81 | 7·40 |
| 2 | " .......... | 24·0 | 772 | 5·92 | 6·11 |
| 3 | " .......... | 17·0 | 758 | 6·68 | 7·80 |
| 4 | " .......... | 17·0 | 762 | 6·68 | 6·00 |
| 5 | " .......... | 19·0 | 765 | 6·41 | 4·63 |
| 6 | " .......... | 21·0 | 763 | 6·20 | 6·39 |
| 7 | " .......... | 17·5 | 775 | 6·74 | 6·92 |
| 8 | Leeds............ | .. | .. | .. | 6·48 |
| 9 | " ............ | .. | .. | .. | 6·22 |
| 10 | " ............ | .. | .. | .. | 4·30 |
| 11 | River Aire below Leeds. | .. | .. | .. | 4·14 |
| *12 | Effluent from a sewage works. | .. | .. | .. | 1·30 |

* Note.—This sample came in a bottle; no precautions had been taken to exclude air.

### Discussion.

Mr. Fairley thought that a scale of colours might be a more rapid and possibly equally exact way of making the estimation. He deprecated the use of coal gas for sweeping out the apparatus, since it often contained air added to assist in its purification.

Mr. Richardson also considered that colours, such as that exhibited, could easily be imitated by means of dyes. The coal gas should, at any rate, be passed through alkaline pyrogallate as a precaution; and he was afraid that some of the nitrogen introduced as air in the estimation would dissolve, making the result low.

Mr. Halliwell was afraid that since, as a rule, samples of sewage effluents and partly polluted waters collected on one day could generally only be examined the next, some of the dissolved oxygen might easily have entered into combination in the interval; and as the authors had stated that the colour produced weakened on standing for two or three hours, it would not be feasible to add the reagents at the time of taking sample and finishing the estimation on receipt of sample in the laboratory.

The Authors stated that the reason for the method adopted was the varying natural tint of different waters, which was probably sufficient to cause error. They had tried coal gas alone in their apparatus, and found no oxygen in it. The tints obtained in their experiments were fairly permanent if protected from light, and consequently Mr. Halliwell's difficulty might be overcome by adding the pyrogallic acid and alkali immediately the sample had been taken, and then keeping the whole apparatus in the dark until the tint obtained could be compared with the standard in the laboratory. Any such error in the nitrogen as apprehended by Mr. Richardson would be diminished to one quarter when calculated to oxygen. The change in tint, due to 1 c.c. of air, was very perceptible, and consequently the method gave results to within ½ c.c. of oxygen.

# Journal and Patent* Literature.

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## I.—PLANT, APPARATUS, AND MACHINERY.

### PATENTS.

*Filtering Presses, Impts. in and relating to.* J. Metzger, Worms-on-the-Rhine, Germany. Eng. Pat. 17,296, Aug. 10, 1898.

In filtering presses which are constructed with a series of built-up elements operated upon by the end-thrust of a screw, an intermediate chamber is arranged between the inlet and outlet chambers, so that the liquid to be filtered passes first through one set of filtering elements into the intermediate chamber, and then from this chamber through a second set of elements.—R. A.

*Filtering Apparatus [Sugar-refining, Brewing, &c.], Impts. in.* H. Bertrand and W. Knecht, both of Worms. Eng. Pat. 25,081, Oct. 29, 1897.

The apparatus comprises a copper holder capable of turning in bearings on a portable framework; a series of filter-frames, also of copper, fixed in the holder, and supporting perforated plates on which the dry filtering material is placed; a liquid-supply pipe furnished with an inspection-glass; a second inspection-glass, with pressure-gauge and testing-cock, for the liquid after it has passed through the filter, and a discharge-pipe furnished with a third inspection-glass and an outlet-cock.—R. A.

*Mixing Gases, Improved Apparatus for; especially applicable to the Distribution and Regulation of Mixtures of Ozone and Air.* M. Otto, Neuilly-sur-Seine. Eng. Pat. 17,259, Aug. 10, 1898.

Relates to a titrating mixing apparatus, particularly intended for mixing air and ozone in any required proportions. The supply-pipes of the two gaseous currents are each provided with a supply regulator, a meter, and a mixer. —R. S.

*Liquefied or Highly Compressed Gases, Impts. in Capsules or Receivers for Containing.* H. V. R. Read, Broad Street Avenue, London. Eng. Pat. 19,067, March 7, 1898. (Under Internat. Convent.)

The capsule is closed by a cap or cover, which acts as a valve, the flange of the cap having its burr left on, or being otherwise serrated, to permit the entrance of the gas, &c. past its edges. A packing ring surrounds the cap, and a seat is formed or provided within the capsule, to support the cap during the filling operation. Between the seat and the turned-over edge of the capsule is a space large enough to permit of a slight motion of the cap and packing ring, which, when the filling operation is completed, are forced by the internal pressure against the said edge of the capsule, so effecting a tight closure. The top of the cap is thinned, so that it, and not the cap, will burst in case of excessive pressure.—R. A.

*Kilns for Burning Earthenware, Fireclay Goods, and the like, Impts. in.* R. C. Staley, Burton-on-Trent. Eng. Pat. 16,335, July 27, 1898.

The kiln is provided with one or more burners at each end, from which run diagonal flues, beneath the floor, to the opposite ends and sides. These under-flues communicate with a space formed by double walls around and over the kiln, the outlet being at the top. An entrance to the kiln is left for introducing the goods, which is bricked up before the kiln is fired.—H. H. B. S.

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* Any of these specifications may be obtained by post by remitting 8d.—the price now fixed for all specifications, postage included—to C. N. Dalton, Esq., Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.

*Drying Materials and Impregnating the same with Liquids, Impts. in Apparatus for.* C. Hüber, Vienna. Eng. Pat. 28,363, Dec. 1, 1897.

The apparatus consists of a closed casing, heated by steam or other heating-fluid coils, and provided with means for exhausting the air therefrom and for introducing the impregnating liquid. It contains one or more trays, mounted on vertical pivots fixed to the casing, for containing the material to be dried or impregnated. The trays can be turned outwards on their pivots, for charging and discharging purposes. Within each tray is mounted a pan into which the material is put, the pan being mounted upon a central pivot so that it can be turned round thereon, for gaining ready access to its contents. The casing is closed by a hinged door. The heating coils are arranged in a horizontal plane both above and below the trays.—R. S.

*A High Temperature Furnace or Destructor for the Purpose of Burning House and Town Refuse, and other Combustible Substances.* W. Saint, Higher Broughton. Eng. Pat. 26,466, Nov. 13, 1897.

In these destructors, the furnaces are constructed in groups, set back to back, side by side, or otherwise, all communicating with a common distributing flue at the rear end, and with a further flue at the forward end, under the control of dampers or valves. The valves are so manipulated during working, that the unconsumed gases or the like of any one furnace may be passed, by means of the common distributing flue, through any one of the other furnaces, and from the latter into the flue at the front end of the furnace, and thence into the chimney. Air and steam may be forced into the furnaces as usual. In the drawings, two forms of destructor are shown, one consisting of six furnaces or cells set back to back, the other of a number set side by side. —R. S.

*Hamper or Receptacle for Bottles containing Acids, Essences, and the like; New or Improved.* Mrs. C. Delecroix, Forges d'Uzemain, Vosges, France. Eng. Pat. 25,793, Nov. 6, 1897.

The body of the basket is made of reticulated sheet metal, the meshes of which are progressively enlarged in rows from the bottom upwards to obtain the desired shape, and is hooked to metal rings at the top and bottom. The bottom is formed of metal rods connected by clips at their centres, and having their outer ends hooked at equal intervals on the bottom ring. Two tubes are threaded on the top ring to serve as handles. The basket rests on a circle or ring of flat iron, which is connected by clips or ties to the bottom ring.—R. A.

*Supplying Fluids at Predetermined Times and for Predetermined Periods to Vats or Receptacles for Chemical Processes, or for Analogous Purposes; Impts. in Apparatus for.* H. N. Prentice, Stowmarket, Suffolk. Eng. Pat. 24,603, Oct. 23, 1897.

The controlling valves, through which the fluids are supplied, are operated electrically by a solenoid arrangement, a timepiece being employed for making the contacts necessary to complete the electric circuits through the solenoid coils at the requisite times. The primary circuit completed by the timepiece actuates a relay, which switches in a stronger current for operating the valves. The relay circuit also includes a dash-pot or dash-pots adapted to break the primary circuit, and to regulate the time during which the valves shall remain open.—R. A.

## II.—FUEL, GAS, AND LIGHT.

*Acetylene: Dangers of Explosion of.* P. Wolff. Zeits. f. angew. Chem. 1898, 919—925.

Acetylene explosions can be divided into two classes—those which are the result of carelessness and unskilful handling, and those due to the peculiar chemical and physical properties of acetylene. According to Berthelot's investigations, acetylene is not explosive under atmospheric pressure. It becomes so under a pressure of 2 atmospheres; and under 38 atmospheres, at which it can be liquefied at 0° C., it exerts a pressure on explosion of 5,500 atmospheres, *i.e.*, nearly as great as gun-cotton. Danger lies in the local heating due to the decomposition of the calcium carbide. The temperature of decomposition of pure acetylene is 780° C., whilst mixed with air it is 480° C. Theoretically the temperature of the decomposition of calcium carbide is 1,100° C., but practically this is never reached, owing to conduction through the walls of the generating vessel. —T. A. L.

*Acetylene, Purification of.* P. Wolff. J. für Gasbeleucht. 1898, **41**, [42], 683—684.

The mean composition of the gas from a large number of samples of American and Swiss carbide was:—

| — | American. | Swiss. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Acetylene | 98·87 | 99·87 |
| Phosphuretted hydrogen | 0·04 | 0·02 |
| Sulphuretted hydrogen | 0·02 | .. |
| Ammonia | 0·06 | 0·04 |

Less sulphuretted hydrogen than formerly, and no siliciuretted hydrogen, are found in the gas from recent samples of carbide. The amount of sulphuretted hydrogen is less when water is in excess in the generator. Careful selection of the raw materials may reduce the ammonia and phosphuretted hydrogen in the gas to a few hundredths of 1 per cent., but even such small quantities of these impurities are highly objectionable. Frank's process of purification of acetylene gas gives good results. He uses acid solutions of metallic salts, of which cupric chloride in hydrochloric acid is the most suitable. A portion of the acetylene is converted into aldehyde. Wolff favours the use of hypochlorites, *e.g.*, bleaching powder. He adds to it a certain proportion of a chromate. The free chlorine is thus taken up, and the acetylene is not attacked.

A. Frank subsequently defended his process, and said that the acid solution could be contained in enamelled vessels of any capacity, and that the amount of aldehyde formed was very small. He stated that 1 litre of his solution purified 14 cb. m. of gas, and that it could then be regenerated by boiling and aeration.—J. A. B.

*The Radiation of Incandescence Mantles.* H. Le Chatelier and O. Boudouard. Comptes Rend. **126**, [26], 1861—1864.

The luminosity of incandescence mantles may be explained by means of the ordinary laws of radiation. The "luminescence" theory, which presupposes that the emissive power (*i.e.*, emissive energy, not light-emissive power) of such bodies is for certain rays greater than unity, is based on an observation that ZnO has a greater emissive power than Pt; but as the emissive power of the latter is only 0·25, the theory is not supported by this fact. The authors studied the radiation of the Auer mantle by applying a paste of the same composition to the surface of a thermo-electric couple and determining the relative intensities of radiation from the surface and from the background of the platinum which appeared through the fissures. They found that at all temperatures and for any ray, the emissive power is less than unity; the case is therefore simply one of a body, the emissive power of which differs from one simple ray to another and varies unequally with the temperature—which is the case with all coloured bodies, without exception. The emissive power is not abnormally high: it is lower than that of many bodies ($Fe_3O_4$, $U_2O_3$, &c.) the illuminating power of which is, on the contrary, very small. The luminosity should rather vary inversely as the emissive power: bodies of low emissive power must attain a higher temperature before they can diffuse by radiation the energy which they receive by contact with the hot gases; and, as is known, the higher the temperature is, the greater is the relative proportion of luminous rays, with colourless bodies at least.

The following results were obtained by placing in a certain part of the Bunsen flame the junction of a thermo-couple which had been flattened out to a disc of 1·5 mm. diameter and covered with the oxides named, the corresponding radiation from melting Pt being taken as the unity of intensity of radiation :—

| Body. | Temperature. | Red. | Green. | Blue. |
|---|---|---|---|---|
| Platinum ....... | 1290 | $10^{-2}\times 8·5$ | $10^{-2}\times 4·0$ | $10^{-2}\times 1·4$ |
| Oxide of iron.... | 1080 | 1·50 | 0·48 | 0·10 |
| Auer's mixture.. | 1380 | 7·00 | 12·50 | 12·50 |
| Thoria .......... | 1290 | 1·45 | 1·40 | 0·30 |
| Ceria............ | 1110 | 1·90 | 0·70 | 0·15 |
| Uranium oxide.. | 1070 | 0·30 | 0·25 | 0·05 |
| Lanthana....... | 1250 | 4·00 | 3·10 | 1·80 |

Thus Pt, the emissive power of which is one-quarter that of oxide of iron, gives ten times as much light.

Bunte's theory of the catalytic action of the ceria upon the combustible gases is erroneous. The authors find that the luminosity is the same whether the mantle be heated in the burning gases or in the hot, fully-burnt gases produced; and where combustion has been completed, contact action cannot come into play. Moreover, the temperature of the mantle is not over 2,000 C., as required by Bunte, but is sensibly the same as that of the carbon particles in ordinary gas flames (about 1,650° C.), the authors' results varying, according to the ray employed, from 1,590 to 1,710° C.

The authors conclude that the substance of the mantle possesses at its working temperature an unequal emissive power for the different kinds of rays; it is therefore to be regarded as at that temperature a *coloured body*. Its useful incandescence results from the fact that its very high emissivity, near to unity, for blue, green, and yellow rays is less for the red, and doubtless still feebler for the ultra-red. The proportion of energy radiated as visible rays is consequently very high; nevertheless, the absolute amount of energy radiated as light is smaller than that which would be diffused by a black body raised to the same temperature. But a black body heated under the same conditions and with the same area of radiating surface would attain only a much lower temperature and would also have only a very low illuminating power.—H. B.

*Choke Damp and Coal Dust, Ignition of, by Electricity.* Heise and Thiem. Elektrotech. Zeits. 1898, 3.

References are made to early experimenters in this subject, including Wullner and Lehmann, whose results in 1884–85 showed that with heated metal electrodes, the gas was not ignited with a spark produced by a current of less than 8 ampères, that with high-tension currents a spark 0·197 in. in length was necessary—with the use of condensers this length was diminished to 0·0985 in.—and also that the effect of glowing wires upon the explosive mixture depended upon the thickness of the wire. The authors have experimented with a mine gas, in the Bochum district of Westphalia, and a 500-volt current. With the glow lamp in a wooden chamber, the gas mixture always exploded when the glass was carefully removed and the filament exposed. When the glass was broken in the chamber, the gas sometimes ignited, but not always. With the arc lamps and with sparks from machines, the conclusion arrived at was that all visible naked sparks are dangerous. In mines lighted by glow lamps, precautions should be taken to ensure that the filament gets broken if the lamp glass breaks.—A. W.

*Oxygen in Illuminating Gas, Estimation of.* H. Lubberger. J. fur Gasbeleucht. 1898, 41, [43], 695.

*See under* XXIII., *page* 1189.

## PATENTS.

*Regenerative Furnaces for the Heating of Gas Retorts and the like, Impts. in and relating to.* G. Helps, Hinckley. Eng. Pat. 25,265, Nov. 1, 1897.

Hollow arch blocks are used to conduct the gases from any part of the generator to any position in a mixing chamber above, and also for distributing the gases from the latter. When two or more tiers of retorts are arranged in one setting, a central dividing wall is used so that the tiers may be used independently of one another.—R. S.

*Gaseous Products of Combustion, for Operating Engines or for other Purposes; New or Improved Means for Generating.* E. Hesketh, Eltham, Kent, and A. Marcet, London. Eng. Pat. 25,856, Nov. 6, 1897.

"An apparatus for generating gaseous products of combustion under pressure, the said apparatus consisting of, or comprising, a closed refractory lined vessel" (combustion chamber)—or closed vessel containing a perforated inner hood of refractory material—"and means for supplying separately thereto, and under pressure, the constituents of a combustible mixture, and a vessel containing water so connected with the first-named vessel that the products of combustion pass through the water before they issue from the vessel for use." The object of the invention is to reduce the temperature of the gases, so as to fit them for use in an ordinary engine; the water may also be introduced in the form of spray, into the combustion chamber, which at the same time is surrounded by a water-jacket.—C. S.

*Liquid Fuel Burners, Impts. in Apparatus for Regulating the Supply of Combustible Liquid or Vapour to.* H. A. House, jun., East Cowes, and R. R. Symon, London. Eng. Pat. 26,454, Nov. 12, 1897.

The inventors claim: a regulating apparatus consisting of "an expansible and collapsible chamber subject to the varying pressure of the steam in the boiler heated by the burner, a regulating valve operated directly by the movable part of such chamber, without the intervention of pin-jointed locks or connections, and a spring adapted to counterbalance more or less of the pressure in the chamber"; and "the use of a strainer and bye-pass valve in connection with the oil- or vapour-inlet passage to the regulating valve."—C. S.

*Gas Generators, Impts. in.* A. Orme, Wolverhampton, and F. J. Jones, King's Heath. Eng. Pat. 26,970, Nov. 18, 1897.

The gas generator consists of two chambers—an upper and a lower one. The fuel is charged into the upper chamber, and gas rich in hydrocarbons is driven off by the heat from the lower chamber. The fuel then falls through an opening or openings from the top chamber on to the top end of the inclined firebars of the lower chamber, or on to the fuel lying thereon. In this chamber it is burnt with a limited supply of air, whereby producer-gas is obtained. The gas outlets from the two chambers communicate with one another, so that the gases from the two chambers mix, and the cooler heavy hydrocarbons from the upper chamber are fixed by the greater heat of the producer-gas from the lower chamber, the formation of pitch in the flue being thus avoided or minimised.—R. S.

*Lime-light or the like, Impts. in Apparatus for Producing.* A. S. Newman, and Newman and Guardia, Ltd., London. Eng. Pat. 27,541, Nov. 23, 1897.

The oxygen at high pressure is admitted to a horizontal tube ending in a nozzle, and coal-gas or hydrogen at low pressure is admitted through a safety-valve to the annular space between the above tube and another tube surrounding it. The gases mix in a comparatively small mixing tube, having a trumpet-shaped mouth, into which the nozzle of the oxygen tube projects. The mixing tube is of nearly the same cross section throughout its length, and has a burner on its end.—R. S.

*Gas Producers, Impts. in or relating to.* C. Humfrey, Northwich. Eng. Pat. 28,367, Dec. 1, 1897.

Below the main feeding hopper is placed "a second inverted hopper connected by a conduit or conduits with the gas space near the top of the generator, whereby the products of partial combustion can rise up through the space below the hopper and enter the main gas space of the generator, and thus the space below the hopper becomes

an active gasifying space, instead of being a dead one or nearly a dead one, as at present." The invention is illustrated as applied to a Mond cylindrical gas generator.
—R. S.

*Artificial Fuel and Fire Lighters, Impts. in the Manufacture of.* H. Gardner, Liverpool. Eng. Pat. 28,496, Dec. 2, 1897.

The fuel is formed of a mixture of wood or peat, or wood and peat, with or without paper, paper pulp, and vegetable rags, and finally powdered limestone, saturated with inflammable matter such as one or more by-products of petroleum. The mixture may be moulded into various shapes and sizes, but is particularly suitable for making candles of.—R. S.

*Igniting-pills for Illuminating Gas, Impts. in the Manufacture of.* E. Jurgens, Hamburg; J. F. C. Jurgens, Altona, Germany; and The Automatic Universal Gaslighter, Limited, London. Eng. Pat. 29,828, Dec. 16, 1897.

Infusorial earth, alone, or with a small proportion of magnesium carbonate or kaolin, and a binding agent, is kneaded with water and made into small pills, which are air-dried, heated to redness, and, on cooling, are impregnated with platinic chloride solution until they have taken up, for each 50 parts of infusorial earth, "50 parts by weight of pure platinum." The pills are then again dried, and heated in a current of dry hydrogen or water-gas. Platinum black is thus formed throughout the structure of the pills.—E. S.

*Liquid Fuel, An Improved Method of and Apparatus for Burning.* H. H. Lake, London. From P. E. Thurow, Hamburg. Eng. Pat. 30,103, Dec. 20, 1897.

To obviate the disadvantages attendant on the use of steam or compressed air for atomising the liquid fuel, the inventor proposes to employ gasified oils, and has constructed a triple concentric tube burner, the central tube of which—through which the air requisite for combustion is admitted—projects beyond the other two (which supply the fuel and gas respectively), and, being shaped like a trumpet, overlaps the mouths of same. The relative rate of feed of the liquid fuel and atomising gas is regulated by screwing the three tubes or nozzles more or less into one another, thus altering the relative sectional area of their apertures. The gasified oil, employed for atomising, also itself acts as fuel.—C. S.

*Gaseous Combustion Products, and Utilising the same in Furnaces and the like; Improved Method of and Apparatus for Treating.* F. Thiele and A. Röttger, Glössingen, Westphalia. Eng. Pat. 11,267, May 17, 1898.

In a system of steam-boiler firing with artificial draught, there is first a preliminary distillation, and the gases obtained are drawn by a fan through a coil of pipes in a water reservoir, whereby the tar is deposited, and collects in a receiver, while the purified gases are forced on to a gasholder, from which they are passed back to heat the boiler. Fresh air may be admitted to the purified gases, as desired.
—R. S.

*Pipe Conduits, Gas Reservoirs, and the like; Process for Impregnating the Packing Material employed for the Stopping of Joints in.* K. E. Rosenthal and J. Billwiller, both of Berlin, Germany. Eng. Pat. 16,042, July 22, 1898.

The packing materials are impregnated with bodies of the kind which enter into combination with the component parts contained in the gases, or other specially introduced gases, or substances, so that, by chemical reaction, an increase in volume occurs, and thereby a gas-tight closing is produced. For packing material, which is impregnated with tar, a metallic salt, such as ferric chloride, is employed, the reactions with the sulphuretted hydrogen and ammonia contained in illuminating gas resulting in the formation of sulphide of iron and ammonium chloride.
—R. A.

*Hydrogen, Impts. in Apparatus for Producing and Carburetting.* J. Fielding, Upton St. Leonards, Gloucester. Eng. Pat. 17,516, Aug. 13, 1898.

The appliance consists of a generator—single or in duplicate—containing a perforated false bottom of invulnerable material, for supporting the charge of zinc, iron, &c., fed through a tapering tubular compartment. The gas produced therein is led through pipes into a carburetting vessel, whence it passes to the service pipe, the heat evolved by the reaction in the generator facilitating the operation of carburetting. When the gas pressure in the generator exceeds a limit determined by the aperture of the discharge pipe, the acid used is thereby forced into a side pipe, and thus removed from contact with the generating metal, so that the evolution of gas ceases until the pressure is reduced again.—C. S.

*Acetylene and other Rich Gases, Impts. in Methods of and Apparatus for Burning.* A. C. Fraser, Brooklyn, U.S.A. Eng. Pat. 27,016, Nov. 18, 1897. (Under Internat. Convent., April 19, 1897.)

The complete specification of six pages, accompanied by 19 diagrams and including 15 claims, describes in great detail a system of lighting by means of impinging jets of acetylene and air. The fifteenth claim is: "The described system of burning acetylene, comprising a series of burners having two ducts for gas and air, and converging jet orifices therefrom, a source of gas under pressure, gas- and air-supply pipes leading from said sources respectively and branching to the respective burners, and means for governing the pressures of gas and air transmitted through said pipes to the burners, to maintain them in uniform ratio, to the effect specified." The acetylene and air respectively are pumped into two gasholders over water, various safety devices being described; under the desired pressure they pass through governors, and are then led throughout the building by a parallel system of pipes. By means of a fixture described, they then enter a tube, which is divided into two collateral compartments, and pass through a special cock to the burner. In one form of burner one jet of gas and one of air, issuing from a fish-tail burner, impinge on each other at such an angle as to spread the flame out in a thin film. Various other forms are also described.—H. B.

*Acetylene Gas, Impts. in Automatic Apparatus for the Production of.* A. Wartenweiler, Engwang, and R. Spengler, Hasli, Switzerland. Eng. Pat. 23,547, Oct. 13, 1897.

A cord attached to the bell of a gasholder actuates a lever cock on a pipe which supplies water to generators containing calcium carbide. The generators are divided into chambers, into which the water flows in turn, and branch pipes and cocks regulate the supply to each generator. The gas passes from the generators through a water-seal to the gasholder, by the movements of which the supply of water is controlled through the cord and lever tap.—J. A. B.

*Acetylene or other Gases, Impts. in Apparatuses for the Production and Use of.* P. Guadagnini, London. Eng. Pat. 23,977, Oct. 18, 1897.

A carbide receiver, comprising a number of tubes, in each of which carbide or a carbide cartridge is placed, is fed with water through its base. The gas generated in the first tube passes through the other tubes in series, while the water, as soon as the first tube is exhausted, flows from it into one of the channels intervening between the tubes, and thence to the base of the second tube; and in similar manner throughout the series. When two carbide receivers are employed, the water flows over from the first exhausted receiver into a ball, the weight of which is thereby increased sufficiently to move a counterweighted lever to which it is attached, and thereby open a water-way to the second receiver. For portable apparatus a tubular water-reservoir is provided to obviate excessive splashing. A gas-collecting chamber, which may be of the bell gasholder pattern, receives the gas from the carbide vessels.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* L. de Ravel, Marseille, France. Eng. Pat. 25,002, Oct. 28, 1897.

The bell of a gasholder, by its rise, raises a lever, which, when the bell descends, drives forward, by means of a pawl, a [illegible] wheel which revolves cylinders, over which there [illegible] endless band. A hopper lets carbide fall into a box [illegible] the endless band, and the box is agitated by the [illegible] of the toothed wheel, and distributes the carbide on to the endless band, which conveys it to a chamber, in the bottom of which is a tank of water, into which the carbide falls. The gas evolved is conveyed to the gasholder. Provision is made for the renewal of the water in the tank, and the removal of the lime produced.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Manufacture of.* The Acetylene Light Syndicate, Ltd., and J. Gore, both of Trowbridge, Wilts. Eng. Pat. 25,952, Nov. 8, 1897.

Water flows from the tank of a gasholder or other water reservoir through a pipe, in which is a valve actuated by the movements of the gasholder bell, to one of two chambers containing carbide, and the gas evolved passes into the gasholder. A cock determines into which chamber the water shall flow, but in the event of one chamber becoming exhausted, a bye-pass permits the water to flow on to the second chamber.—J. A. B.

*Acetylene, Improved Apparatus for Use in the Production of.* G. E. Arkell, Cullingworth, Yorks, and J. W. Bailey and J. Clapham, both of Keighley, Yorks. Eng. Pat. 26,269, Nov. 11, 1897.

A generating chamber containing carbide is supplied with water, through a pipe provided with a cock, from a chamber in which water is maintained at a constant level by inflow through a pipe provided with a cock, from a superposed reservoir. The two cocks are moved by levers, which are coupled together so that they act in unison, and the levers are actuated by the movements of the bell of a gasholder. The gas evolved passes from the generating chamber through the water in the chamber, in which the water stands at a constant level, and thence to the gasholder. The water for use in the generation of the gas is thus first employed for washing gas previously generated.—J. A. B.

*Acetylene Gas Generators, Impts. in.* J. Kremer, Gilly, Belgium. Eng. Pat. 26,776, Nov. 16, 1897.

A hopper containing carbide is supported by a frame rising from the base of the tank within a gasholder. A vertical rod, carried by a cross-bar attached to the bell of the gasholder, slides through, and closes an orifice at the base of the hopper when the bell rises above a given height, and thus stops the fall of the carbide into the water in the tank. In the crown of the bell is an air-lock device, by which carbide may be fed into the hopper while the apparatus is at work, and springs attached to the crown of the bell facilitate the discharge of the carbide through the orifice of the hopper.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* J. Rebel, Angoulême, France. Eng. Pat. 27,288, Nov. 20, 1897. (Under Internat. Convent.)

The generator comprises two chambers, in the lower of which is water, and in the upper a shoot for carbide in the form of a truncated pyramid, the base of which is curved, and allows the carbide to escape on to an endless band, which passes over rollers, one of which is moved forward by a pawl and ratchet device when the bell of the distributing gasholder descends. The carbide falls from the band into a conduit provided with two angular bends, leading into the lower chamber. The conduit is filled with petroleum oil, which prevents the rise of moisture to the carbide in the upper chamber. The gas passes from the top of the lower chamber to the distributing gasholder.—J. A. B.

*Acetylene Gas-Generating Apparatus, Impts. in Devices for Feeding the Carburet in.* J. Rebel, Angoulême, France. Eng. Pat. 27,300, Nov. 20, 1897.

The feed of carbide from an endless band into a water tank is controlled according to the movements of the bell of the generating gasholder by means of a cord, one end of which is attached to the bell and the other to a weight. The cord passes round a loose pulley, which carries a pawl, which engages with a ratchet wheel on the shaft of the drum over which the endless band passes.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Generation of.* H. Grubb, Rathmines, Ireland. Eng. Pat. 28,264, Nov. 30, 1897.

An elevated tank supplies water to a closed chamber by a pipe reaching nearly to the bottom of the latter. Water flows from this chamber to a generator containing carbide, from which the evolved gas passes into the upper part of the chamber and forces back water into the elevated tank until its level is below that of the inlet to the generator. The supply of water to the latter is thus stopped until gas has been removed from the chamber for consumption. Valves are provided on the gas outlet, water inlet, and waste pipe, and an arrangement is described by which these valves are automatically operated when the lid of the generator is removed and replaced.—J. A. B.

*Illuminating Gas (Acetylene), Impts. in Apparatus for Making.* H. Bryant, Aransos Pass, Texas, U.S.A. Eng. Pat. 28,824, Dec. 6, 1897.

Water is supplied to carbide in a generating vessel through a pipe, in which is a valve which is closed by the rise of the bell of a gasholder. The gas generated passes direct to the burners, but any surplus quantity goes into the gasholder until the water supply is cut off by the rise of the bell, whereupon the gas pipe leading from the generator to the gasholder and to the burners is also closed. Gas subsequently generated passes into a vessel capable of storing it at high pressure until it is required for use. The gas is consequently supplied to the burners at a fairly uniform pressure.—J. A. B.

*Acetylene, Impts. in Apparatus for the Production of.* E. V. Prevost, O. Rans, and A. Cerkel, all of Paris. Eng. Pat. 29,201, Dec. 9, 1897.

Calcium carbide is placed in a tubular vessel, in the top of which are holes for the exit of gas to the burners, and through the bottom of which passes a capillary tube containing a strip of blotting paper, up which water passes by capillarity to the carbide. The invention relates to portable apparatus.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production and Storage of.* J. McConechy, Glasgow. Eng. Pat. 30,585, Dec. 28, 1897.

The supply of water to carbide, which is contained in a generator below the gasholder tank, is cut off, when the bell rises, by the action of a spring or catches on the lever of a tap on the supply pipe. This lever is connected with a rod which passes up a central tubular opening in the bell of the gasholder, so that the tap is opened through a cross-bar, which spans the opening, striking the head of the rod when the bell descends.—J. A. B.

*Acetylene Gas Generator.* J. S. Ferguson, Minneapolis, U.S.A. Eng. Pat. 2863, Feb. 4, 1898. (Under Internat. Convent.)

Carbide is contained in boxes arranged along two sides of a chamber in the lower part of which is water. The boxes are supported by catches, which are released in turn by cams mounted on a shaft, which is rotated by a rod attached to the bell of the gasholder into which the gas, formed when the carbide is precipitated into the water, passes. The rod comes in contact with a tooth of a ratchet wheel on the end of the shaft when the bell descends owing to the consumption of the gas stored in the holder, and thereby rotates the shaft to such an extent that one box is released each time the bell descends.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* H. H. Lake, London. From J. Grand, Lyons, France. Eng. Pat. 8382, April 7, 1898.

Carbide is discharged from a conical hopper into a reservoir of water through a valve, which is opened to admit the passage of carbide by mechanism operated by the descent of the collecting gasholder, and is closed by its own weight. The gasholder bell floats in an annular water seal, and the space enclosed by the inner tube of the seal is packed with purifying material, through which the gas passes on its way to the bell and thence to the burner.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating and Storing.* J. Stott, Oldham, Lancs. Eng. Pat. 8439, April 9, 1898.

Water flows from an overhead tank, through a filter, to the base of a generator in which carbide is disposed on superposed trays, and the evolved gas passes through a tubular atmospheric condenser to a holder, in which it is collected by the displacement of water. Two or more generators may be coupled to one supply for use in turn.—J. A. B.

*Acetylene Gas Generators, Impts. in or relating to.* A. J. Boult, London. From Società Italiana Pel Carburo di Calcio acetilene ed altri gas, Rome. Eng. Pat. 12,491, June 3, 1898.

Carbide is fed from the orifice of an inverted cone into a tank of water by means of a disc provided with helical blades, which is rotated by clockwork or other mechanism, subject to control according to the movements of the gasholder. A plunger device for removing lime from the tank without interrupting the action of the apparatus, and means of supplying fresh water to the tank, are described.
—J. A. B.

*Acetylene Gas, Impts. in or connected with Generators for.* K. E. Rosenthal, Berlin. Eng. Pat. 14,049, June 24, 1898.

A number of tubes extend from a horizontal partition in a closed vessel into water contained in the lower part of the vessel. Each tube is furnished with a slide, above which carbide is stored. The slides are removed in turn, and the carbide allowed to fall into the water, by means of a screw which is rotated when the gasholder sinks. On the shelf is stored sufficient carbide for second charges of the tubes, and the recharging from this store is effected by a plate operated from without.—J. A. B.

*Acetylene Gas, Impts. in or relating to Apparatus for Producing.* F. Gehlert, Basle, Switzerland. Eng. Pat. 17,079, Aug. 8, 1898.

Above the water in a tank (which is provided with a cover which, by screws, forms an air-tight joint with the tank) there is suspended a basket of carbide from spring-clips held by a screw passing through the cover. On turning the screw, the clips release the basket and allow the carbide to fall into the water, and the gas evolved passes to a gasholder. The residue is flushed out from the generator before the cover is removed for recharging.—J. A. B.

*Acetylene Gas Generators, Impts. in.* T. Manger, Bamberg, Bavaria. Eng. Pat. 17,449, Aug. 13, 1898.

A pipe, pivoted on a cock on a water-supply pipe, is weighted at one end, and at the other end opens above a funnel, which leads to generators containing carbide. When the free end of the pipe is raised, the cock is closed, and no water passes. A lever attached to the bell of the gasholder depresses the free end of the pipe when the bell sinks, and opens the cock, so that water flows to the generators. A chain attached to the weighted end of the lever and the handle of a cock on the pipe which conveys the water from the funnel to the generators, transfers the supply to a second generator when the first is exhausted.—J. A. B.

*Acetylene Gas Generators, Impts. in.* C. R. and E. W. Gillett, both of Chicago, U.S.A. Eng. Pat. 17,612, Aug. 16, 1898.

A closed water tank supplies water to a tray of carbide in a chamber, one side of which is hinged for recharging the tray, by means of a pipe with check valve, percolator, wad, and drip-curtain. The gas passes through a pressure-reducing valve to the burners, but an arm of the exit pipe leads by a second reducing valve and a non-return valve to the upper part of the water chamber, and thus causes a constant pressure of gas to be exerted on the water therein. Whenever this pressure exceeds that of the gas in the carbide chamber, water passes to the latter, and generates gas, until the pressure exceeds that on the water in the tank, whereupon the flow of water is arrested.—J. A. B.

*Acetylene Gas Apparatus, Impts. in.* K. G. Gustafsson, Stockholm. Eng. Pat. 18,063, Aug. 22, 1898.

Carbide is fed through a shoot into a vessel containing water covered by a layer of oil, and falls down an inclined plane to the bottom of a flue, which extends up above the level of oil, and ends in a chamber having an exit below the level of the water. The gas is generated at the foot of the flue, and passes up it, and through the exit into the space above the oil, and thence to the burners. The foot of the flue is below the level of the water.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for Generating.* E. Engasser, Colmar, Alsace. Eng. Pat. 18,220, Aug. 24, 1898.

A generator containing superimposed receptacles for carbide is fed with water through a pipe provided with a cock, which is automatically actuated by a chain attached to the bell of the gasholder which receives the generated gas. A purifier, safety siphon, and cooling worm are provided.
—J. A. B.

*Acetylene Generators or Lamps, Impts. in.* J. Bilbie and H. Drivet, London. Eng. Pat. 18,543, Aug. 30, 1898.

A water-reservoir is surmounted by a water-jacketed carbide receptacle, in which the carbide rests on a perforated disc, to the lower side of which, water ascends by means of a wick. The wick is withdrawn from contact with the disc, and the action of the water on the carbide stopped, by screwing the water-reservoir round by half a turn.
—J. A. B.

*Acetylene Gas, Impts. in and relating to the Production of.* R. Quatannens-Moens, Dixmude, Belgium, and E. Career-Dilger, Bruges, Belgium. Eng. Pat. 18,591, Aug. 30, 1898.

A drum, containing a number of compartments filled with carbide, is rotated by transmission gearing when the bell of the gasholder descends, and the shutters of the compartments are mechanically opened in turn, and the carbide discharged into a funnel opening on to a distributing cone above water in a reservoir. The cone is of greater diameter than the mouth of the funnel, so that gas rising vertically does not give access to the drum. The gas passes through a washer to the gasholder, and thence to a purifier containing, in successive layers, sulphate of iron, wadding, and calcium chloride.—J. A. B.

*Acetylene Gas, Impts. in Apparatus for the Production of.* R. Soderquist, Helsinborg, Sweden. Eng. Pat. 18,943, Sept. 5, 1898.

The bell of a gasholder floats in an annular water-channel, and supports a perforated carbide receptacle in a central cylindrical space within the holder. The carbide receptacle is surrounded by an outer cylindrical vessel perforated at some distance from its base. Water is supplied from a duct provided with valves to the exterior of this vessel, and flows through the perforations to the carbide receptacle. The gas generated raises the bell and lifts the carbide receptacle out of contact with the water which has flowed into the cylindrical vessel. Special means of attaching the carbide receptacle to the crown of the gasholder, so that it may be readily removed through the base of the apparatus for refilling, are described.—J. A. B.

*Acetylene; A New or Improved Apparatus for Generating and Utilising Gas, such as.* J. Boss, Pimlico, Eng. Pat. 19,369, Sept. 12, 1898.

The bottom of the tank of a gasholder forms the top of a generating chamber, and contains a ball-valve free to rise or fall when the pressure on one side exceeds that on the other, and thus to give passage to gas or water, as the case may be. The bottom of the generating chamber is inclined, and forms the top of a water-chamber connected at its upper part with the generating chamber so that water may flow from it to the latter. Carbide is fed into the generating chamber through an opening provided with a manhole cover. A pipe with valve connects the generating chamber with the lute of the gasholder.—J. A. B.

*Acetylene Lamps, Impts. in.* F. Schmid, Vienna. Eng. Pat. 26,676, Nov. 15, 1897.

The claims are for (1) a valve device for regulating the supply of water to the carbide; (2) an appliance for distributing the water uniformly over the carbide; (3) and (4), constructing the carbide receptacle with a number of separate compartments disposed in a ring, so that a centrally-arranged funnel which receives the water-supply may be rotated so as to deliver the water to a fresh supply of carbide, as that in each successive compartment becomes exhausted; (5) a regulating valve for preventing excessive gas pressure; (6) a carbide receptacle in which the charge is fed forward either by gravity or by springs, against a grating against which is suspended a felt disc for distributing the water, the pulverulent exhausted residue falling between the grating and the disc to the chamber below; and (7), (8), and (9) constructions and arrangements of reflectors.
—H. B.

*Acetylene Gas, Impts in [Bicycle] Lamps or Apparatus for Use in Burning.* R. Holliday and R. Holliday and Sons, Ltd., Huddersfield. Eng. Pat. 2078, Jan. 26, 1898.

The claims are for the construction, &c., of an acetylene lamp in which the water is brought into regulated contact with calcium carbide by the use of a capillary siphon or wick.—H. B.

*Acetylene Gas Lamps, Impts. in and relating to.* W. N. Moore and J. Karr, Washington, U.S.A. Eng. Pat. 16,317, July 26, 1898.

There is described and claimed a lamp particularly adapted for use on bicycles. The lamp body has a water-chamber in its upper portion, the bottom of the chamber having the shape of an inverted cone, which has at its apex a discharge opening controlled by a needle-valve, which extends through the top of the water-chamber to a milled head outside the lamp. The lower portion of the lamp-body contains the carbide holder, which is removably connected with it, and a "percolator" extending centrally from the bottom of the carbide holder up to and surrounding the apex of the bottom of the water-chamber. "The percolator consists of a foraminous tube, as one made of wire gauze or the like, which is filled with an absorbent material, as cotton or other absorbent material that will not readily pack, so as to deliver the water fed thereto to the carbide in a diffused state." An annular space separates the walls of the carbide holder from the body of the lamp, and a pipe leads the acetylene from this space to a suitable burner.—H. B.

*Acetylene Gas Lamps, Impts. in.* H. K. Spence and The British Acetylene Gas Generator Co., Ltd., Kirkcaldy. Eng. Pat. 17,327, Aug. 11, 1898.

There is claimed (1) in acetylene lamps, suitable for cycles, table use, &c., a carbide container having a perforated bottom to which water is supplied from the water-chamber below, by the capillary action of a wick the lower part of which communicates with the water; (2) mounting the carbide container—say by securing to its top a spindle which passes through a stuffing-box to a knob outside the lamp casing—so that its perforated bottom can be raised from or pressed down upon the water-wick; and (3) forming an extension of the water chamber by leaving a space between the outer casing and the gas-chamber, so that should the lamp be upset, the water can flow into the extension and thus be kept apart from the wick.—H. B.

*Increasing [by Acetylene] the Illuminating Power of Gas made from Coal or other Material, A Method of.* J. R. Wigham, Dublin. Eng. Pat. 28,526, Dec. 3, 1897.

By means of "a governor of approved construction or any other means" a continuous supply of coal gas of uniform quality and pressure is passed to the burners, and a regulated proportion of acetylene is passed into the supply pipe, so as to increase the illuminating power to the required standard. The rate of passage of the acetylene is regulated by "a micrometer arrangement, which consists of a screw valve with fine screw adjustment and conical seat," an index being fitted to the device to show the rate of flow.—H. B.

*Carbide, The Preservation of, and its Subsequent Treatment for Use in the Production of Gas; Impts. in.* C. J. Lundstrom, Stockholm. Eng. Pat. 23,793, Oct. 15, 1897.

The claim is for a method of preserving carbide, consisting in impregnating or covering it with a suitable material (*e.g.*, oil, turpentine, or similar matters) which is practically insoluble in water, and in connection therewith the subsequent removal of such matters by (*a*) heating the carbide, or (*b*) treating it with such solutions (*e.g.*, of soda lye, boric acid, &c.) as will sufficiently remove the preservative coatings to permit the generation of the acetylene, or (*c*) mixing the water used for generating the gas with the substances mentioned in (*b*).—H. B.

*Storing and Using Calcium Carbide or the like for Acetylene and the like Gas-generating Apparatus; Impts. in Means for.* E. S. Bond, Handsworth. Eng. Pat. 26,843, Nov. 17, 1897.

For cycle lamps and similar purposes the carbide is usually placed in a perforated tube which is carried in the apparatus. To avoid the loss of small particles through the perforations, and other troubles, the patentee prepares tubular cases having perforated sides and provided with movable caps at each end. One end of each tube is preferably contracted taperwise so that it may be telescopically passed into the opposite end of a similar tube, so as to form a tubular continuation. Within the tubes is placed a lining of thin soluble paper; then the carbide is filled in and the caps are put on. The claim is for the combination of the perforated case with the lining, for the purpose specified.—H. B.

*Calcium Carbide, Impts. in or relating to the Manufacture of.* J. Bastick, Manchester, and W. A. Thornton, Wetherby. Eng. Pat. 29,008, Dec. 8, 1897.

The quantities of coke and lime required for the production of calcium carbide are passed, either separately or roughly mixed, to the crusher, so that they are fed simultaneously to the grinding machine, where the materials are reduced to the required size and thoroughly mixed together in it and its hopper, prior to entering the elevator which conveys them to the conveying trough, whence they pass into the furnace.—G. H. R.

*Carbide Containers, and in Containers for Acetylene Gas Apparatus; Impts. in.* R. Abeles, Dresden-Löbtau, Germany. Eng. Pat. 20,646, Sept. 30, 1898.

The claim is for: "Carbide containers and containers for acetylene gas apparatus which are provided with an enamel, or vitreous or like coating, for the purpose of preventing injury to the same by corrosion, and thereby of enhancing their usefulness and durability." The containers may be of any suitable form, and the coating process may be of any known kind.—H. B.

*Hydrogen Gas Generator, A New or Improved.* G., H. H., and S. H. Hawkins, Upton Park, Essex. Eng. Pat. 25,084, Oct. 29, 1897.

The hydrogen gas generator consists of three superposed, detachable chambers, the top chamber receiving the charge of dilute sulphuric acid, and the two lower chambers, containing iron or zinc shavings, supported on grids, acting as generators. The acid chamber is connected by suitable pipes with both generators independently, and the middle and bottom chambers are also so connected that the liquid from one chamber can be forced by the pressure of gas into the other when necessary. Other pipes form safety-valve arrangements, while vent pipes, pipes for drawing off the gas, with accessory fittings, are shown. One or both generators may be used at a time, so that one may be charged whilst the other is operative, thus giving continuous action. When the gas is generated in larger quantities than is required, it forces the liquid in the generators through the supply pipes until it is below the respective grids, thus checking the gas production.—E. S.

*Burners for Incandescent Gas Lighting. Impts. in and in the Manufacture of.* J. Wilson, Birmingham. Eng. Pat. 24,328, Oct. 21, 1897.

The burner-tube consists of two truncated cones, joined at their narrow parts, the angle of divergence of the sides of each being about 60° or, if varied, not less than 11°. The "waist" of this tube is crossed by a flat bar (arranged in a vertical plane), an upper extension of which is bent into a holder for the mantle-support, and the ends of which pass through slots in the joined ends of the two cones, thus locking them together. The funnel-shaped mouth of the burner-tube is fitted as usual with wire-gauze, through which the holder for the mantle-support passes, and the holder is fitted at its upper end with a deflector disc.

The first claim is for the use, in such an atmospheric burner, of a "Bray's regulator burner" for the purpose of governing the amount of gas passed into a conical mixing chamber as described; the other three claims are for various parts of the burner.—H. B.

*Bunsen Burners, Impts. in or relating to.* A. J. Boult. From E. Verbeke, Brussels. Eng. Pat. 20,273, Sept. 24, 1898.

In the Bunsen burner claimed, the gas-inlet injector is surmounted by a short tube which is cylindrical for the first 2—3 cm., and then conical until the diameter is reduced to one-half. Round the lower part of the tube are several air-inlet holes. The burner-tube proper slips over the short tube, fitting its lower part exactly, and it is correspondingly perforated, so that by rotating the burner-tube, the air-holes may be more or less closed. Some distance up the burner-tube is a second series of air holes, surrounded by a rotatable, correspondingly perforated collar. A metallic grating, spiral netting or other baffle, is placed within the mouth of the burner-tube.—H. B.

*Incandescence Mantles [Woven Fabric], An Impt. in.* J. Moeller, Westminster. Eng. Pat. 28,706, Dec. 4, 1897.

There is claimed "an incandescence mantle made of birdcage net, and treated in the known manner for incandescence:" the "birdcage net" consisting of rows of long narrow meshes separated by lines of interwoven threads, which at the end of each mesh cross diagonally, some to the right and some to the left, to be again looped with others which cross diagonally in like manner.—H. B.

*Incandescence Bodies of Electrical Glow Lamps, Manufacture of Homogeneous Mixtures for.* C. D. Abel, London. From The Allgem. Electricitäts Ges., Berlin, Germany. Eng. Pat. 18,941, Sept. 5, 1898.

Homogeneous mixtures for electrical incandescence bodies, composed of conductors of the second class, are formed from intimate mixtures of metallic oxides, prepared from mixtures of aqueous solutions of their salts, either by precipitation by means of a suitable precipitant, or by electrolytical separation from such solutions, or by evaporation and subsequent driving off of the acid radicles, by heating to incandescence.—G. H. R.

*Atmospheric Gas Burners, Impts. in.* T. Fletcher and Fletcher, Russell, and Co., Ltd., Warrington. Eng. Pat. 26,834, Nov. 17, 1897.

It is claimed that a burner which is incapable of lighting back, which gives complete combustion of the gas, and is incapable of becoming choked in use, is obtained by the following construction. The outlets, at which the gas and air mixture is ignited, are of equal size, and between the outlets and the pipe leading the mixture to the burner (*i.e.*, the mixing tube), is fixed a diaphragm of wire gauze or perforated sheet metal. The openings in the diaphragm should not exceed about one-sixteenth of an inch across. The size of the holes in the burner is such that the sum of the areas of the outlets is equal to the sum of the areas of the openings in the diaphragm, and each sum should separately be equal to the cross-sectional area of the mixing tube, allowance being made for frictional resistance.

In some of the figures shown, the mixing tube expands at its upper end considerably to the part at which the diaphragm is fixed, in order that the total area of the diaphragm openings may be equal to that of the unexpanded part of the tube. In cases where the diaphragm has to be fixed in such a position that, if flat, the openings could not possibly have the required area, it is made more or less dome-shaped, or placed in a sloping position, so as to obtain this area.

When the burner outlets are perforated through a vertical surface, the disc constituting the top of the burner extends over the outlets, thus providing a flange, which protects them from falling matter.—H. B.

*Vapour-Burner Apparatus, Impts. in Self-generating.* P. Glud and C. O. Nielsen, Copenhagen. Eng. Pat. 18,037, Aug. 22, 1898.

The combustible hydrocarbon is fed to the apparatus by air-pressure or by gravitation, and the supply tube is fitted with a starting cup, all in the usual manner. Within the vertical feed-tube, at a part between the starting cup and the burner-mouth, is fixed the generator, consisting of a hollow cylindrical body, closed at its lower end, and screw-threaded externally, so as to fit the feed-tube; it is further provided with an external helical channel, which communicates at its upper end with the interior of the cylinder, and at its lower end receives the oil supply. The upper end of the generator carries the vapour nozzle, which directs the vapours into the burner-top. The latter consists of a tube, which expands into an inverted cone, perforated by air-ports slightly above the vapour nozzle, and carrying at its upper wider end a disc, which serves both as spreader and as the top of the air-mixing chamber. The heat communicated by the flame to the metallic tubes is sufficient to maintain the generation of the combustible vapours. All parts are made easily detachable, for ease in cleaning.—H. B.

## III.—DESTRUCTIVE DISTILLATION TAR PRODUCTS, Etc.

*Paraffins, Sulphonation of the.* R. A. Worstall. Amer. Chem. J. 1898, **20**, [8], 664—675.

From the results of the present investigation the following conclusions may be drawn:—

1. Aliphatic hydrocarbons are readily capable of direct sulphonation.

2. Cold fuming sulphuric acid is without perceptible action, whilst if the temperature employed be too high, the sulphonic acids are destroyed. The correct temperatures would appear to be the boiling points of the respective hydrocarbons.

3. Fuming sulphuric acid gives mainly monosulphonic acids, whilst sulphuric anhydride results in the formation of disulphonic acids.

4. Yields of about 40 per cent. of the theoretical are obtained, both of the mono- and of the disulphonic acid, the remainder of the hydrocarbon being oxidised to carbonic anhydride and water.

These results, in connection with those obtained by the nitration of paraffins, communicated in a former paper, disprove the statements in regard to the difference between aliphatic and aromatic hydrocarbons, in that the former are not capable of nitration by nitric acid or of sulphonation by sulphuric acid. The paraffins examined by the author were hexane, heptane, and octane. Owing to the circumstance that Orndorff and Young found propane to be slowly absorbed by fuming sulphuric acid, the author is extending this investigation to the behaviour of the gaseous members of the aliphatic series towards fuming sulphuric acid.
—D. B.

*Normal Saturated Hydrocarbon, The Decomposition of a, by Aluminium Chloride.* C. Friedel and A. Gorgeu. Comptes Rend. 1898, **127**, [17], 590—594.

It has been observed, in organic syntheses with aluminium chloride, that this reagent has a decomposing action on certain hydrocarbons. Hexamethylbenzene, naphthalene, and even benzene, are broken up, on heating with aluminium chloride, into a variety of hydrocarbons. It has also been noticed that aluminium chloride has a powerful action on American petroleum distillates, whilst Russian distillates of the same boiling points are practically unattacked. In studying this action it was considered advisable to start with the simple case of a saturated, normal, open-chain hydrocarbon, and normal hexane was selected. Expecting to obtain some of the simpler hydrocarbons, the authors prefaced their experiments by preparing pure methane, ethane and normal propane, and butane, and by characterising these in determining their solubilities at certain temperatures and pressures in amyl alcohol (b.p. 130°—132° C.). It was remarked, by the way, that amyl alcohol readily absorbs air and combines with some of the oxygen, and it must be carefully freed from air and preserved over mercury. Normal hexane was prepared, partly by the action of hydriodic acid on mannite and decomposition of the secondary iodide produced by zinc and hydrochloric acid, and partly by the action of sodium on propyl iodide; the absence of unsaturated compounds was verified by testing with a drop of bromine. 25 grms. of aluminium chloride were placed in a tube, 2·5 cm. in diameter and 15 cm. long, and 4 grms. of hexane were added. The mixture was heated, and the products of the action passed through a reflux condenser into two U tubes, one cooled in ice and the other in a freezing mixture. When the action had slowed down, another 4 grms. of hexane were added, and this was repeated three or four times. The U tubes were then allowed to attain the temperature of the room, and the gas given off was collected over mercury. The liquid remaining in the U tubes was fractionated, and was found to contain unaltered hexane and a hydrocarbon boiling at about 35° C., which was found on analysis to be normal pentane. The gaseous hydrocarbon collected over mercury consisted chiefly of butane. Thus the action of aluminium chloride on normal hexane results in the removal of one $CH_3$ group and the formation of pentane, which is itself also decomposed in the same way, with the production of butane. This, on account of its great volatility, escapes from the sphere of action, and is not perceptibly decomposed into propane. The residue in the tube should contain compounds richer in carbon atoms, but the authors could only recognise that it was a very complex mixture, and had not sufficient material to separate any of the components.—J. F. B.

*American Petroleum, Composition of.* S. Young. Proc. Chem. Soc. 1898, [198], 175.

An investigation has been made of the hydrocarbons in American petroleum boiling between, say, 25° and 115° C., and evidence has been obtained of the presence of the following:—

Isopentane, b.p. 27·95°; normal pentane, b.p. 36·3° C.; pentamethylene, b.p. about 50°; isohexane, b.p. about 61°; normal hexane, b.p. 68·95°; methylpentamethylene, b.p. about 72°; benzene, b.p. 80·2°; hexamethylene, b.p. 80·8°; isoheptane, b.p. 90·3°; normal heptane, b.p. 98·4°; methylhexamethylene, b.p. about 102°; toluene, b.p. 110·8° C. Of these, the two pentanes and normal hexane have been obtained pure, normal heptane nearly so, and hexamethylene and isoheptane fairly pure. Hexamethylene has been obtained nearly pure from American and quite pure from Galician petroleum (Proc. Chem. Soc. 1898, **14**, 103). It is not improbable that other isomeric hexanes, and, therefore, possibly heptanes, may be present, and there are indications of the existence of small quantities of dimethyl pentamethylene in American petroleum. A comparison of the results obtained with American, Galician, and Russian petroleum shows that the same classes of hydrocarbons—paraffins, polymethylenes or naphthenes, and aromatic hydrocarbons—are present in the petroleum from all three sources, but that the relative amount of naphthenes and, in all probability, of aromatic hydrocarbons is greatest in Russian and least in American petroleum.

*American Petroleum, The Separation of Normal and Iso-Heptane from.* F. E. Francis and S. Young. Proc. Chem. Soc. 1898, [198], 176.

The heptanes cannot be separated in a pure state from petroleum by fractional distillation, owing to the presence of naphthenes with boiling points not sufficiently far removed from those of the two paraffins.

*Benzene and Normal Hexane, The Boiling Points and Specific Gravities of Mixtures of.* D. H. Jackson and S. Young. Proc. Chem. Soc. 1898, [198], 176.

In the fractionation of American petroleum from which the aromatic hydrocarbons have not been removed previously by treatment with a mixture of sulphuric and nitric acids it is noticed that the benzene comes over chiefly at about 65° C., the fractions above and below this temperature containing smaller and smaller amounts of it.

The approximate estimate of the amount of benzene is easily obtained by treating the fractions repeatedly with mixed nitric and sulphuric acids and diluting the acid with water. The dinitrobenzene separates after some time in needle-shaped crystals. The most probable explanation of the lowering of the boiling point of benzene appeared to be that aromatic hydrocarbons and paraffins, though miscible in all proportions, approximate in their behaviour to partially miscible liquids. The results obtained show that this is the case, for there is always expansion on mixing, and the boiling point of normal hexane is hardly raised by the addition of even 10 per cent. of benzene, whilst that of benzene is very rapidly lowered by adding hexane.

It is therefore impossible to separate pure normal hexane by distillation of any mixture of this paraffin with benzene, though it is easy to separate pure benzene from a mixture containing only a moderate amount of hexane.

*Paraffins and other Hydrocarbons, The Action of Fuming Nitric Acid on the.* F. E. Francis and S. Young. Proc. Chem. Soc. 1898, [198], 177.

In the separation of pure normal hexane from American petroleum, the methylpentamethylene was removed by heating the nearly pure hydrocarbon with fuming nitric acid during several days. It was observed at the same time that the low-boiling fractions, containing isohexane, were also purified by this treatment, and that, on diluting the nitric acid, considerable quantities of a trinitro- derivative of isohexane separated as a crystalline solid.

Other isoparaffins were found to react rapidly with fuming nitric acid when heated on a water bath, and to yield liquid or solid nitro-compounds, but the normal paraffins were attacked only very slowly, and, in these cases, the acid, when diluted, did not become turbid. Details are given of the action of fuming nitric acid on isopentane, isohexane, isoheptane, iso-octane, and di-isobutyl.

The behaviour of the paraffins, when heated with fuming nitric acid, is compared with that of other classes of hydrocarbons.

S. Young further said that the olefines present in American petroleum must be inconsiderable in amount, as very little bromine is required to produce a permanent coloration; they would, of course, be eliminated during the treatment with mixed nitric and sulphuric acids.

With respect to the action of nitric acid, it must be remembered that the result is greatly influenced by the conditions; thus, Konowaloff had shown that normal hexane and normal octane yield secondary nitro-compounds on heating with dilute nitric acid under pressure. The authors had found that the isoparaffins undergo nitration readily when heated with fuming nitric acid in a reflux apparatus on a water bath. The normal paraffins were much more slowly attacked under similar conditions, and as the nitric acid remained clear on dilution, it was evident that nitro-compounds had not been formed from them in any appreciable quantity.

## PATENTS.

*Retorts for the Distillation of Shale and other Substances, Impts. in and relating to.* A. Gordon, Uphall, Linlithgow. Eng. Pat. 28,653, Dec. 4, 1897.

The retort, into which the shale is charged, consists of an external circular brickwork casing, having a central column within it, leaving an annular space between it and the outer casing. This structure is supported several feet above ground, preferably on iron pillars. The lower portion of the central column is formed of brickwork, and the upper part of a hollow iron cylinder, and in the brickwork portion, flues are formed around it. In the outer brickwork casing flues are also formed near its inner surface. The lower portion of the retort is divided into several spaces by walls, radiating from the central column to the outer casing. These walls have also flues formed in them. The heating gases undergo combustion in these flues, and passing away up through the iron portion of the central column and a funnel erected on the top of it, communicate the necessary heat to the material in the retort. The central column and division walls with their flues being entirely surrounded and in contact with the shale to be heated, it is claimed that the loss of heat is much less than with ordinary circular or rectangular retorts, which have only external flues, and in which much of the heat is lost by radiation into the outside brickwork. In order to effect the continuous discharge of the spent material, an iron ring or annular table is placed under the bottom of the retort, with scrapers working thereon and actuated by rods and worm gearing for the purpose of discharging the spent material into a hopper below, fitted with a screw conveyor.
—D. B.

*Oil, An Improved Process of Refining.* W. A. Smith, Cleveland, U.S.A. Eng. Pat. 30,662, Dec. 28, 1897.

The process, which is especially adapted for refining oils containing sulphurous compounds, such as are found in the Lima district in Ohio, Canada, and other places, consists in heating the oil or its distillate with spirits of turpentine in the proportion of 1 gall. of the latter to every 500 galls. of the product to be treated. When neutral or heavier distillates are used, rosin may be substituted with advantage for turpentine, 3 lb. of rosin being employed to 1,100 galls. of distillate. The mixture is heated until the temperature reaches a point immediately beyond which distillation begins. The liquid is then allowed to cool, pumped into agitators, and treated with sulphuric acid, the agitation being effected by blowing air through the liquid. The acid is then drawn off, and this operation repeated until the acid ceases to act on the distillate. The latter is then washed with water, which removes the excess of turpentine or rosin introduced into the mixture. After drawing off the water, a small quantity of caustic soda is introduced for the purpose of neutralising any slight amount of acid remaining behind after the treatment. The sulphurous compounds if not eliminated, cause, during combustion, disagreeable odours and deposit deleterious matter on the wick, thereby reducing the candle power of the lamp and the value of the oil.—D. B.

*Asphalt, Artificial; Impts. in the Manufacture of, and in Apparatus to be Employed therein.* G. H. Grey, Kingston-on-Thames. From I. W. Hayward, Cleveland, U.S.A. Eng. Pat. 18,302, Aug. 25, 1898.

For the manufacture of asphaltum from petroleum residue, ground limestone or the like, in the necessary proportion, say 20 to 60 per cent., according to the condition or quality of the residue or the product to be made, is added to the petroleum residue, preferably before the oxidising process. The retort containing the mixture is heated to the requisite temperature, and while the mixture is in a heated condition, air, preferably under pressure, is admitted and efficiently diffused through the material in order to rapidly oxidise the same, and bring it to the required degree of consistency. The retort is caused to rotate on a stationary hollow shaft, provided with hollow branches or arms, through all of which the air is forced, the arms also serving as agitators. To further facilitate the oxidation, ozone or ozonised air or oxygen may be forced into the material while in a heated condition.—D. B.

*Paraffin Scale, An Improved Method of and Apparatus for Treating.* J. Armour and F. Alison, Loanhead, Midlothian. Eng. Pat. 20,086, Sept. 22, 1898.

This invention is an improvement on Eng. Pat. 8756 of 1886 (this Journal, 1887, 442). In purifying paraffin scale by the process technically known as "sweating," it is absolutely necessary that the cakes should be turned over so as to properly expose both sides of each cake to the action of the heat. Hitherto this has been performed by hand labour, which is not only extremely arduous and trying, but also very expensive. According to the present invention, the paraffin scale is run into cells retained in a vertical position, in which it is allowed to cool and solidify. The temperature of the house is then raised so as to sweat the wax in the cells. During this operation the cells are turned over from side to side by a mechanical tilting arrangement, and when the sweating process has been completed they are again brought to the vertical position. The wax is then melted and run off.

By this invention the following advantages are said to be gained:—(1.) Economy in labour. (2.) The paraffin is thoroughly and equally acted upon by the heat. (3.) As the paraffin wax is turned by the tilting of the cells, there is no necessity for turning the paraffin cakes by hand. (4.) The product obtained is superior to that produced by the old methods. (5.) The cooling and the sweating are performed in the same house and in the same apparatus. By retaining water at the bottom of the cells during the cooling process the paraffin is prevented from completely filling up the cells at the bottom, so that, when the water is drained off, a free passage is left for the running off of the impurities separated during the sweating process.—D. B.

### Erratum.

This Journal, 1898, page 912, table: under "Simon-Carvès" and "Net cost per ton of coke," for "0·158" read "0·124."

## IV.—COLOURING MATTERS AND DYES.

*Nitro-Bodies to Azo- and Hydrazo-Bodies, Electrolytic Reduction of.* K. Elbs and O. Kopp. Zeits. f. Elektrochem. 1898, 5, [9], 108—113.

For the electrolytic reduction of aromatic nitro-bodies in alkaline solution, the authors recommend that the nitro-bodies should be electrolysed at the boiling-points of their solutions in aqueous alcohol, to which sodium acetate has been added, in order to make the solutions conduct. The solutions only become alkaline during the process of electrolysis, and the degree of alkalinity is conditioned by the number of sodium ions separated out. In reducing small quantities (up to 20 grms.) of the nitro-bodies, an ordinary porous pot, filled with a cold saturated solution of sodium carbonate, and containing a platinum anode, is placed in a slender beaker, the narrow annular space containing the electrolyte and a cathode consisting of a cylinder of nickel gauze. If the liquids are introduced hot into the cell and a sufficiently high current density maintained (8—16 amp. per sq. dcm. of cathode surface), the alcoholic solution is soon raised to the boiling-point. Under these conditions nitro-bodies are easily converted into azo-bodies in 20—40 minutes, and as a rule, without any indication of the formation of azoxy-bodies as intermediate products. In the preparation of hydrazo-bodies, on the other hand, the intermediate formation of azo-bodies is very well marked. As soon as the last of the nitro-body has been reduced hydrogen appears at the cathode, and if a constant current be employed during the electrolysis, it is possible to calculate to a fraction of a minute when the evolution of hydrogen will begin. The appearance of hydrogen shows that the reduction of the nitro-body to the azo-body is complete, and that the further reduction to the hydrazo-compound has begun. In order to make full use of the current, it is now necessary to reduce the current density to one-tenth or one-fourth of its original value, *i.e.*, to 1—4 amp. per sq. dcm., so that the second phase of the reaction takes just as much time as the first, although only one-fifth of the current is required.

A further reduction with splitting up of one molecule of the hydrazo-body into two molecules of the corresponding amino has hitherto only been observed in one particular case.

Recipes are given for the preparation of small quantities of azobenzene, hydrazobenzene, *o*-azotoluene, *o*-hydrazotoluene, *p*-azotoluene, *p*-hydrazotoluene, *p*-azo-*o*-xylene, *p*-hydrazo-*o*-xylene, *m*-diamido-azobenzene and *m*-diamidohydrazobenzene, of which the following may serve as an example:—

*Azobenzene.*—Composition of cathode electrolyte: 20 grms. of nitrobenzene and 5 grms. of sodium acetate, dissolved in 200 c.c. of 70 per cent. alcohol; highest permissible current density, 10 amp. per sq. dcm.; quantity of electricity required, 17·4 amp. hours; yield, 95 per cent. of the theoretical.

*Hydrazobenzene.*—When the above reduction is complete, the current density must be diminished to 2—3 amp. per sq. dcm. and the process of reduction continued until about 10 per cent. more than the theoretical quantity of electricity (1·35 amp. hours) has been supplied.

A series of experiments is next described which was made to ascertain the highest permissible current density. The cathode electrolyte was kept at a known temperature and the current density gradually increased until hydrogen was evolved; it was then diminished until the evolution of gas ceased and the mean of the two readings taken as the highest permissible current density. In estimating the surface of the nickel gauze only one side was considered and no attention was paid to the meshes of the gauze. The results are contained in tables which show the increase of the highest permissible current density with increase of temperature and concentration of the electrolyte.—J. S.

*m-Nitro-p-Toluidine, Electrolytic Reduction of.* K. Elbs and R. Schwarz. Zeits. f. Elektrochem. 1898, **5**, [9], 113—115. (Compare preceding abstract.)

In the preceding paper it was shown that *m*-nitraniline could be easily and almost completely reduced electrolytically to *m*-diamido-azobenzene and *m*-diamidohydrazobenzene, and in the present paper the authors show that the same method is applicable to the reduction of other nitramines, including the homologues of *m*-nitraniline, although in the case of the last substances, the yield of the hydrazo-bodies is not so good. No more than the theoretical quantity of electricity need be supplied.—J. S.

*Yellow Colouring Matters of Rhus Cotinus and Rhus Rhodanthema.* A. G. Perkin. Proc. Chem. Soc. 1898, [198], 183.

Venetian sumach, the leaves of *R. Cotinus*, contains myricetin and not quercetin as stated by Lowe (Zeits. Anal. Chem., 1871, **12**, 127). The leaves of *R. rhodanthema*, the yellow cedar of New South Wales, contain quercetin and gallotannic acid. The stems of both plants contain fisetin, and the leaves thus contain the more highly oxidised colouring matters, as quercetin and myricetin are considered to be hydroxy- and dihydroxy-fisetin respectively. Other members of the *Rhus* family hitherto examined contain no fisetin in the stem. The Venetian sumach examined, contained 16·7 per cent. and the *R. rhodanthema* leaves 9·5 per cent. of tannin.

The leaves of *Artocarpus integrifolia* (Jackwood tree), *A. incisa* (bread fruit), and *A. lakoocha* are devoid of colouring matter.

*Colouring Matters of the New Zealand Dyewood "Puriri."* A. G. Perkin. Proc. Chem. Soc. 1898, [198], 183.

"Puriri" (*Vitex littoralis*) is a large tree, growing in northern New Zealand, and chiefly used for building and similar purposes. Its tinctorial properties are little known. It contains two colouring matters in the form of glucosides.

*Vitexin*, $C_{15}H_{14}O_7$ or $C_{17}H_{16}O_8$, the chief product, is a canary-yellow crystalline powder, distinguished by its sparing solubility in most solvents. Owing to the difficulty of obtaining substitution products without decomposition, its molecular weight is at present uncertain. *Acetylvitexin*, $C_{15}H_9O_7(C_2H_3O)_5$ or $C_{17}H_{10}O_8(C_2H_3O)_6$, forms colourless needles melting at 251°—256°. On fusion with alkali, phloroglucinol, acetic and parahydroxybenzoic acids are formed, whilst boiling aqueous or alcoholic potash solutions give phloroglucinol and parahydroxyacetophenone. Further, the product of the ethylation of Vitexin with boiling alcoholic potash yields para-ethoxybenzaldehyde, para-ethoxybenzoic acid, and a phloroglucinol derivative. Warm nitric acid (sp. gr. 1·54) forms metadinitroparahydroxybenzoic and picric acids, but when dilute acid is employed, a sparingly soluble nitro-compound, $C_{15}H_6O_5(NO_2)_4$, of unknown constitution, is also obtained in small quantity. This consists of small lemon-yellow needles, which dye mordanted calico; it is converted by strong nitric acid into picric acid, and appears to be closely related to Vitexin. With nitrobenzene, it forms an addition product, $C_{15}H_6O_5(NO_2)_4,C_6H_5NO_2$, crystallising in orange-coloured needles which lose nitrobenzene at 150°, or on digestion with alcohol. Vitexin gives very pure yellow shades on calico mordanted with chromium and aluminium salts, but owing to its insolubility these are of a poor character. They somewhat resemble the corresponding shades given by Apigenin, and as the chief decomposition products of both colouring matters are identical, it is probable that Apigenin and Vitexin are closely related.

*Homovitexin*, $C_{16}H_{10}O_7$ or $C_{18}H_{18}O_8$, is present in the wood only in minute quantity. It crystallises in fine primrose yellow needles, melts at 245°—246°, and is distinguished from Vitexin, which it otherwise resembles, by its ready solubility in alcohol. Fusion with alkali gives phloroglucinol and parahydroxybenzoic acid, and treatment with dilute nitric acid yields metadinitroparahydroxybenzoic acid. It dyes mordanted fabrics feebly.

The shades given by "Puriri" with chromium and aluminium mordants are distinguished by their pure yellow tone, and may have some commercial utility.

*Paranitro-Ortho-anisidine.* R. Meldola. Proc. Chem. Soc. 1898, [199], 226.

As paranitro-ortho-anisidine (m.p. 139°—140°), which was described in a paper published in conjunction with Messrs. Woolcott and Wray (Trans. Chem. Soc. 1896, **69**, 1330), has recently been made the subject of a patent by the Fabriques de Produits Chimiques de Thann et de Mulhouse (Ger. Pat. 98,637 of 1897), the author records some further observations which have been made with the substance, as it is proposed to continue its investigation.

The acetyl derivative, purified by repeated crystallisation from water so as to get rid of the isomeric metanitro-derivative simultaneously formed, melts at 153°—154°, the melting point 143°—146° given in the former paper being that of the mixed isomerides. The acetyl-derivative of *p*-nitro-*o*-anisidine can be reduced by dissolving in hot water and agitating with iron dust and a little acetic acid. The *p*-amido-*o*-acetanisidide thus formed is somewhat unstable, and has not yet been isolated. It is readily diazotisable, and the $NH_2$-group has, by this means, been replaced by iodine with the formation of *p-iodo-o-acetanisidide* [$NHC_2H_3O : OCH_3 : I = 1 : 2 : 4$]. This compound crystallises in beautiful, silvery scales melting at 175°—176°.

## PATENTS.

*Dyestuffs of the Phthaleïne Series, Impts. in the Manufacture of.* O. Imray, London. From The Basle Chemical Works, Basle, Switzerland. Eng. Pat. 18,477, Aug. 29, 1898.

The dyestuffs formed by the alkyl ethers of the dialkylrhodols (by condensing dialkylamido-oxybenzoylbenzoic acids with resorcinol) suffer from the defects that they are sensitive to alkalis and their zinc lakes when steamed turn bluish-violet from a yellowish-red. If, however, the ethers of the dialkylrhodols be condensed with formaldehyde,

condensation products, which are yellowish-red dyestuffs are obtained, the zinc lakes of which are not altered on steaming. A solution of 42 kilos. of the ethyl ether of dimethylrhodol in 200 kilos. of sulphuric acid (66° B.) is mixed with 16 kilos. of formaldehyde (40 per cent.) in 100 kilos. of sulphuric acid. After standing some days, the melt is poured on to ice and the sulphate is salted out. In order to convert it into the chloride, the precipitate so obtained is dissolved in 60 kilos. of alcohol and 70 litres of water, and 70 kilos. of hydrochloric acid (20° B.) are added to the hot solution, the chloride being finally salted out. When dry it forms a brilliant greenish powder soluble in hot water and alcohol, the latter solution showing no fluorescence. The solution in concentrated sulphuric acid is yellowish-brown. The dyestuff gives yellowish-red shades on tannined cotton and when printed with zinc oxide gives a lake of the same shade, which is unaltered by steaming.
—T. A. L.

*Diamido-oxydiphenyl and like Bases, Producing.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 25,136, Oct. 29, 1897.

When oxyazobenzene is reduced in an acid solution, it yields aniline and *p*-amidophenol, whilst its alkyl ethers are converted by the same treatment into semidines (derivatives of the *o*- and *p*-amidodiphenylamine). If, however, the acid esters be reduced in the same way, the resulting products are oxydiamidodiphenyl bases. For instance, acetyloxyazobenzene yields oxydiamidodiphenyl, $HO(H_2N)C_6H_3(C_6H_4.NH_2)$. The following example illustrates the method employed :—10 grms. of finely ground acetyloxyazobenzene are gradually added, with constant agitation, to 60 c.c. of a solution containing 40 grms. of tin dissolved in 100 c.c. of hydrochloric acid (38 per cent.). The temperature is not allowed to exceed 40° C., and, after standing 24 hours, the double salt which crystallises out is filter-pressed and dissolved in 20 times its weight of water. The tin is precipitated with sulphuretted hydrogen, and, after concentration in a current of carbonic acid, dilute sulphuric acid is added to the solution to precipitate any benzidine formed. The filtrate is neutralised, first with caustic soda, and finally with sodium carbonate. The oxydiamidodiphenyl so formed is easily soluble in alcohol, glacial acetic acid, and hot water, separating in needles. It melts at 148° C., and is a primary diamine, since one molecular proportion condenses with two molecular proportions of an aldehyde, and it also yields a diacetyl derivative on boiling with glacial acetic acid. It has the constitution $[5.2.1]HO.H_2N.C_6H_3.C_6H_4.NH_2[1.4]$, and combines with salicylaldehyde to form the derivative $HO.C_6H_4.CH:N.C_6H_4.C_6H_3(OH)N:CHC_6H_4.OH$, which crystallises in yellowish-brown leaflets melting at 207° C. With *p*-nitrobenzaldehyde it forms—

$$O_2N.C_6H_4.CH:N.C_6H_4.C_6H_3(OH).N:CHC_6H_4.NO_2$$

a flakey orange-red powder melting at 218° C., whilst the diacetyl derivative melts at 269° C. It is proposed to employ the product as a photographic developer or for producing dyestuffs. Other acetylated *p*-oxyazo compounds behave similarly, provided the position para to the azo group is unoccupied.—T. A. L.

*Black Cotton Dyestuff, Production of Direct Dyeing.* S. Pitt, Sutton. From L. Cassella and Co., Frankfort-on-the-Maine, Germany. Eng. Pat. 25,234, Oct. 30, 1897.

By heating oxydinitrodiphenylamine with alkaline sulphides and sulphur in an aqueous solution, a product is obtained which dyes cotton directly deep blue-black shades, fast to rubbing and light, and absolutely fast to washing and milling. The oxydinitrodiphenylamine is obtained by reacting with 1.3.4-dinitrochlorobenzene on *p*-amidophenol in an alcoholic solution in presence of an acetate. For the production of the dyestuff, 15 parts by weight of oxydinitrodiphenylamine, 75 of crystallised sodium sulphide, 30 of sulphur, and a little water are heated together to about 140° C. until the melt is almost dry. The temperature is then raised to 160° C. to remove the last traces of water. The dyestuff can be used directly. It may be purified by dissolving it in water and precipitating with an acid. Bluer shades are obtained by employing homologues of the above substance, as, for instance, oxytolyldinitrodiphenylamine obtained from 1.3.4-dinitrochlorobenzene and amido-*o*-cresol.—T. A. L.

*Azo-Dyestuffs [Brown, Black], and in the Production of Fast Dyeing on Animal Fibre ; Impts. in the Manufacture of.* S. Pitt, Sutton. From L. Cassella and Co. Frankfort-on-Main, Germany. Eng. Pat. 28,107, Nov. 29 1897.

The diazo compound of 2.3.3′-amidonaphthol sulphonic acid combines with various components to yield azo dyestuffs, which, however, are technically of no value, since they are not fast to washing, and are very fugitive to light. The patentees have now discovered that if the dyed fabric be treated with bichromate, the shade is changed to a deep black, and at the same time becomes fast to milling and light. As a typical example, 26 kilos. of sodium 2.3.3′-amidonaphthol sulphonate and 7 kilos. of sodium nitrite are dissolved in water and the solution is run into 50 kilos. of hydrochloric acid (21° B.), cooled with ice. The diazo compound which separates is filtered off and added to 14·4 kilos. of α-naphthol dissolved in soda-lye. The dyestuff so obtained dyes wool a reddish-brown, which, on treatment with a solution of sodium bichromate, is changed to a violet-black.—T. A. L.

*Colouring Matters [Blue], Impts. in the Manufacture of.* H. H. Lake, London. From Farbwerke Mühlheim vormals A. Leonhardt and Co., Mühlheim-on-the-Maine, Germany. Eng. Pat. 28,604, Dec. 3, 1897.

It has been shown in Eng. Pats. 13,565 and 18,623 of 1890 (this Journal, 1891, 760 and 917) that blue basic dyestuffs are obtained by reacting with nitrosodimethylaniline on dimethyl-*m*-amidocresol or analogous compounds. It is now found that the same dyestuffs are obtained by employing instead of the *m*-amidocresols, condensation products of these with formaldehyde. One of these products is obtained as follows :—30 kilos. of dimethyl-*m*-amidocresol, 300 litres of water, 24 kilos. of caustic soda-lye (40° B.), and 7·6 kilos. of formaldehyde (40 per cent.), are heated together until the smell of formaldehyde has disappeared. The solution is filtered and neutralised at 30° C. with acetic acid, when the condensation product, tetramethyldiamidodihydroxyditolylmethane, is precipitated. A blue dyestuff is obtained from this when 10 kilos. of it, together with 19 kilos. of nitrosodimethylaniline hydrochloride and 50 litres of spirit are heated under a reflux condenser until the nitroso compound has disappeared. The melt is then diluted with 500 litres of hot water, sodium acetate is added to precipitate impurities, and the filtered solution is mixed with salt and zinc chloride. A coppery resinous mass separates, which solidifies to green crystals having the same properties as those already described in the specifications mentioned above. In place of nitrosodimethylaniline, nitrosodiethylaniline or benzene-azo-dimethylaniline may be employed.—T. A. L.

*m-Oxybenzaldehyde-o-Sulphonic Acid, Manufacture of.* O. Imray, London. From Farbwerke vormals Meister, Lucius und Brüning, Hoechst-a/Main, Germany. Eng. Pat. 28,707, Dec. 4, 1897.

*m*-oxybenzaldehyde, said to be of great value in the making of acid dyestuffs of the Malachite Green series, cannot be sulphonated in the usual way. The process is claimed for the manufacture of the sulphonic acid of *m*-oxybenzaldehyde $[CHO : OH : SO_3H = 1 : 3 : 6]$, which consists in diazotising the corresponding amidobenzaldehyde sulphonic acid in acid solution and boiling the solution until the evolution of nitrogen ceases. The product is also claimed.—A. C. W.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

*Mercerising Cotton, A Process for; not accompanied by the Shrinkage of the Thread.* L. Lefèvre. Bull. Soc. Chim. 1898, **19**, [20 and 21], 859—860.

Ducat (this Journal, 1898, 840) states that shrinkage during mercerisation is obviated by the addition of glucose to the caustic soda bath. It has long been known that glycerin would answer the same purpose (H. Koechlin). In the French Patent, 264,546, March 2, 1897, the addition of alcohols, esters, hydrocarbons, essences, &c. is recommended. With the use of these substances the shrinkage amounts to 2—5 per cent.; the statement that glucose prevents all contraction, needs more confirmation.—A. C. W.

*Cotton Fibre, The Destruction of, by the Lower Fungi.* Holle. Meeting of the Botanical Section of the Society of German Scientists and Physicians at Düsseldorf, through Chem. Zeit. 1898, **22**, 824.

It was observed that raw cotton, damaged by sea-water and allowed to remain in a moist condition, became so weakened that it became utterly unfit for spinning. At the same time a discoloration, partly green and partly brown, and a most disagreeable odour were developed. The author has studied and prepared specimens of the fungi, which bring about this deterioration of raw cotton. In some cases they are colourless, and can only be distinguished by staining, in others they are brown coloured.

Bleached unsized cotton strongly resists the attack of fungi; but, unless the size contains an antiseptic, moist storage of sized cotton is apt to cause a weakening of the fibre, due possibly to the organic acids produced by the decomposition of the size substance (starch, &c.)—J. F. B.

*Cotton Fabrics, The Mercerising of.* A. A. Smirnoff and B. A. Rosenthal. Färber Zeit. 1898, **9**, [20], 309.

Cotton cloth is mercerised by passing through a number of tanks zigzag fashion, round two series of rollers placed horizontally at the top and bottom of the tanks. The first tank contains the mercerising solution, caustic soda of 40° B., the cloth after leaving passes through a pair of squeezing rollers, the upper one of rubber, and the lower one of copper, into the second tank which contains only air, and here the liquor has time to act on the goods. The third and fourth tanks contain boiling dilute caustic soda solution. In the fifth and sixth compartments, the fabric is washed by jets of water placed between the bights, in the seventh any alkali remaining is neutralised by a boiling dilute solution of sulphuric acid, whilst in the eighth tank the mercerising is finished by washing, as in the fifth and sixth, with jets of water.

The goods are again washed with running water, wrung out, dyed, and finally dried in a stretched condition on an ordinary tentering machine. In this way, it is stated, gloss effects, equal to any produced by more complicated machinery, may be obtained.—H. I.

*Artificial Silk, Note on a New.* E. Knecht. J. Soc. Dyers and Col. **14**, [12], 252—253.

This product which is known as *Vanduara silk*, is a product of the action of formaldehyde on gelatin. A solution of gelatin of the requisite consistency, and maintained at a uniform temperature, is delivered in fine threads on to an endless band, so arranged that on reaching the end, the filaments can be reeled continuously on to bobbins. They are then transferred to reels, a slight twist being given to the fibre in the process, and are placed for some hours in an atmosphere of formaldehyde, when the fibre is ready for use. The new artificial silk has a lustre equal to the best nett silk, but in addition shows a scintillating effect, which is more apparent in a strong light. It is tolerably strong, but does not compare favourably in this respect with real silk. On immersion in water, it curls and becomes flabby and tender, but regains its strength on drying. It is insoluble in alcohol, ether, alkalis, and acids, and when a light is applied to it, it burns like ordinary silk, with the same characteristic odour of burnt wool or feathers. Vanduara silk is dyed in the mass before drawing to a thread or spinning, and it is said that only 1 lb. of colour is required on an average to produce a full shade on 3 cwt. of the silk. The fibre can be produced, it is said, at a profit at 3s. 6d. per lb., whilst the dyeing costs less than 0·25d. per lb.—T. A. L.

*Waterproofed Cotton Fabrics ("Batist"), A Scheme for the Examination of.* R. Thal. Chem. Zeit. 1898, **22**, 867 and 880.

*See under* XXIII., *page* 1190.

## PATENTS.

*Hanks of Yarn, Impts. in the Method of and Apparatus for Scouring.* G. Ormonroyd and T. Mitchell. Bradford. Eng. Pat. 24,468, Oct. 22, 1897.

Hanks of yarn are spread on to cross bands between endless chains and are passed by means of guide rollers, alternately through scouring bowls and squeeze rollers. Any number of bowls can be arranged in line according to the number of washings required, the endless chains and bands being, of course, adjusted to them. The final bowls for rinsing contain clear water only.—C. M.

*Vegetable Fibre, A New Process for Mercerising.* H. E. Newton, London. From The Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 27,020, Nov. 18, 1897.

A bath is prepared by mixing two parts (by weight) of soda lye of 38° B. with one part of glycerin. Cotton is placed in this bath until it has the appearance of parchment. It is then washed and dried. Under these conditions there is no shrinkage, and the cotton increases in strength 23 per cent.

Stronger lye may be used, and the cotton may, if desired, first be wetted with glycerin, and then treated with the soda lye separately.—C. M.

*Vegetable Fibres, Impts. in the Treatment of [Decorticating].* C. Wetherwax Best, New York State. Eng. Pat. 28,491, Dec. 2, 1897.

China grass and other vegetable fibres are placed in baskets made of copper gauze, each having a spiral frame fitted, horizontally, in it. Between the convolutions of the spirals the fibres are loosely packed. Several of these baskets, placed one above the other, are placed in a digester capable of withstanding 100 lb. pressure, and the following solution is made to circulate through them by suitable devices described. 20 lb. of caustic soda are dissolved in 8 galls. of water, and cooled to 80° F. 130 lb. of tallow or fat, free from salt, are then heated to 120° F., and thoroughly mixed with the soda. After 24 hours, that is after saponification, the mixture is remelted with 100 lb. of fat, and then 30 galls. of water are added. When nearly cold, 30 lb. of glycerin in 8 galls. of water are slowly stirred in.

Of this mixture, 4 oz. to 8 oz. per gallon of water are used for degumming. This takes about one hour under a pressure of about 80 lb. per square inch, at a temperature of 325° F. After thoroughly rinsing with water, the fibres may then be decorticated with the following mixture:—one part by weight of caustic soda and three parts of carbonate of soda dissolved in the proportion of ¾ lb. to 1 lb. per gall. At the same temperature and pressure as above, the decorticating takes about two hours (see also this Journal, 1898, 241).—C. M.

*Fabrics Coated with Rubber or the like Substances, Impts. in the Treatment of.* P. A. Birley and C. Macintosh and Co., Ltd., Manchester. Eng. Pat. 29,014, Dec. 8, 1897.

Cloth, immediately after being coated with rubber or like material in a spreading machine is passed over tentering chains attached to the machine. By mechanism, described in the patent, the cloth is subjected to an increasing tension in the direction of its width, and is held under such tension until quite dry. Treated in this way it is claimed that the cloth does not shrink after being coated, as is usually the case.—C. M.

*Mercerising or Finishing Yarn, Apparatus for.* A. Wyser, Aarau, Switzerland. Eng. Pat. 29,613, Dec. 14, 1897.

Cotton yarn is mounted on a vertical reel, the diameter of which, by suitable mechanism, described in the patent, can be increased or decreased at will.

The reel is then placed in a vertical cylinder, fitted inside with horizontal pipes, which have holes facing towards the centre. The reel is rotated, the mercerising fluid being sprinkled on to the yarn, and the waste led off by a pipe at the bottom of the apparatus.

When mercerised, the yarn is washed by continuing the process with cold water. To remove, the reel is taken out, its diameter reduced, and the yarn lifted off (see also this Journal, 1898, 149, 240, 345, 452, 573, 756, and 840).
—C. M.

*Textile Fabrics and other Materials, Impts. in Coating.* T. B. Powell, London. Eng. Pat. 29,842, Dec. 16, 1897.

Three pounds of gelatin are steeped in cold water for 12 hours; the water is then run off, and the product heated in a steam-jacketed pan with 8 oz. of glycerin. The whole is then thoroughly mixed with 1 oz. of formic aldehyde, and poured on to a smooth surface, such as glass, on which a weak solution of ox-gall has been put. The material to be coated is pressed on to it when it is nearly set, but still tacky. By this process fabrics are made which have all the properties of leather or leather cloth.—C. M.

*Rendering Materials Fire-proof, Impts. in; and in Extinguishing Fire.* L. Litynski, A. Rodakiewicz, and F. Kurowski, Lemberg, Austria. Eng. Pat. 14,532, July 1, 1898.

According to the first claim, "the application and use of the improved preparation for the impregnation of all kinds of combustible substances such as wood, paper, cardboard, textiles, straw, and the like, by which the same are rendered uninflammable and completely fire-proof, characterised by the employment, separately or together, and in combination with sodium chloride and a necessary quantity of a sufficiently strong solution of de-fatted quillaia, glue, gelatin, horn, or similar animal substances, and the said solution being saturated with carbonic acid and of manganese oxide, calcium oxide, potassium hydroxide, potassium carbonate, and sodium oxide, or of chemical waste or by-products which contain the said salts."

For extinguishing fire, the described compound is applied, mixed with water.—E. S.

*Fire-Proofing Compositions, Impts. in.* G. X. Dime, New York, U.S.A. Eng. Pat. 17,497, Aug. 13, 1898

The composition consists of 1 part of microcosmic salt and 15 parts of ammonium chloride. In some cases an addition is made to the composition, of 1 part of sodium or potassium borate. Wood is treated by drying, and immersion in the heated solution of the composition until it is sufficiently saturated to sink. Lace and other fabrics are subjected to immersion and drying only. When celluloid is to be rendered fire-proof, the composition is placed in the material of which the celluloid is made.—E. S.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

*Indigo Vat, Production of an; by Electro-deposited Zinc* A. Binz. Zeits. f. Elektrochem. 1898, **5**, [9], 103—108.

In a previous communication (this Journal, 1898, 759) the author put forward the view that the reduction of Indigo Blue to Indigo White in the ordinary alkaline zinc-dust vat is the result of the direct action of the zinc on the Indigo Blue, and not of the hydrogen evolved by the action of the zinc on the alkali. He now describes some further experiments which are intended to support this view. Goppelsroeder, von Wartha, and Mullerus have already shown that the formation of an electrolytic indigo vat in the cold is very incomplete, but the author, on carefully repeating his experiments, has arrived at the conclusion that the conditions in the electrolytic vat are not comparable with those which obtain in the ordinary zinc-dust vat. The difference is chiefly due to the fact that in the electrolytic vat, using caustic soda as electrolyte, the hydrogen which is evolved at the cathode carries the suspended Indigo along with it upwards through the solution, and scarcely acts chemically on it at all. In the zinc-dust vat, on the other hand, the Indigo remains lying at the bottom, and is almost quantitatively converted into Indigo White.

If the author's view be correct, then, on replacing the caustic soda solution in the electrolytic vat by a solution of sodium zincate, electrolytic zinc will be liberated at the cathode, and the formation of the vat should take place more readily. This was proved to be the case in the following experiment:—The cathode compartment consisted of a cylindrical vessel 20 cm. high and 4·5 cm. wide, into which was introduced a zinc disc (4·5 cm. diameter) and a tightly-fitting zinc cylinder (area of exposed surface 217 sq. cm.). The anode compartment, containing a platinum anode, was an earthenware pot 25 cm. high and 1·7 cm. internal diameter. The electrolyte was prepared by slowly adding a solution of 75 grms. of zinc acetate in 190 c.c. of water to a solution of 50 grms. of caustic soda in 50 cc. of water. A mixture of 20 grms. of Indigo and 25 grms. of finely-powdered zinc acetate was then added to 200 c.c. of the above electrolyte, and introduced into the cathode compartment, whilst the porous pot contained 25 c.c. of the electrolyte. The current strength employed was 0·25 ampère during the day, and 0·15 ampère during the night.

After 72 hours' continuous electrolysis, 20 c.c. was withdrawn from the cathode compartment. In appearance it was a brown vat mixed with some insoluble green substance. After standing for a short time in a test tube, three-fourths of the liquid was perfectly clear, and 10 c.c. of this was found to contain 0·418 grm. of indigotin and to possess a faint basic smell.

After electrolysing for another period of 48 hours, 10 c.c. of the vat contained 0·553 grm. of indigotin, whilst after electrolysing for 48 hours more, the amount of indigotin had diminished to 0·527 grm. in 10 c.c., and there was a distinct smell of ammonia. From this experiment it follows that during the electrolytic separation of zinc from a solution of sodium zincate, a complete transformation of Indigo Blue into Indigo White takes place. The reaction goes on in the cold, whereas hydrogen, without the application of heat, practically does not act at all.

In another experiment the author shows that if the same current is sent through two sets of divided cells containing respectively sodium zincate and caustic soda, then fifteen times as much Indigo Blue is transformed into Indigo White in the cell in which zinc is deposited as in the cell in which only hydrogen is evolved.—J. S.

*Sumac, Examination of.* Spica. Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [12], 526.

*See under* XXIII., *page* 1191.

*Light, Action of, on Dyed Cotton Goods.* C. Rheinländer. Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [11], 481—482.

The author gives the results of some comparison experiments carried out with cotton dyed to the same shade with natural colouring matters and with the so-called "Diamine Colours" (L. Cassella and Co.). The samples were exposed under glass for four hours daily, from June 15 to July 15, and the following observations were made:—

1. *Indigo Blue* compared with Diamine Blue E.F. Both were unchanged after 10 days. In 20 days the former had become greyish and the latter redder in shade, while at the end of the exposure, the indigo had a greenish, and the diamine colour a violet hue.

2. *Light Brown.*—Catechu with "Bichrome" compared with Diamine Brown M. Here the natural dyestuff had in 30 days faded less than the artificial.

3. *Dark Brown.*—Catechu and "Bichrome" with Logwood and Fustic compared with Diamine Catechio, Diamine Brown M, Diamine Fast Yellow, and Diamine Blue Black. The artificial colouring matters showed greater fastness than the natural dyestuffs.

4. *Grey.*—Sumach on Iron Mordant compared with Diamine Blue Black, Diamine Orange, and Diamine Fast Yellow. The sumach had faded at the end of 10 days, and in 30 days was bleached, while the diamine colours suffered comparatively little.

5. *Dark Grey on Indigo Ground.*—Camwood, Fustic, and Logwood on an Alum Mordant compared with Diamine Blue Black. Very little difference in fastness was here noted.

6. *Dark Blue.*—Indigo compared with Diamineral Blue R. The former was unchanged after 30 days and the latter had suffered but little. A sample dyed with one-fourth the amount of indigo and topped with logwood, faded much more than the diamine colour.

7. *Violet.*—Logwood on Sumach and Stannous Chloride compared with Diamine Blue 3 R. The latter was hardly changed, while the former was bleached by 30 days' exposure.—H. I.

### *Sodium Chromite.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [11], 461.

This substance was first successfully applied as a mordant in cotton dyeing by H. Koechlin, who prepared it by adding caustic soda solution (3 parts at 38° B.) and glycerin ($\frac{1}{18}$th part at 32° B.) to the mordanting bath containing chromium acetate solution (1 part at 20° B.).

Schmid prefers to dissolve 17·3 lb. of precipitated chromium hydrate in 18 litres of caustic soda (33° B.), and to dilute the product with $6\frac{3}{4}$—9 litres of water and $1\frac{1}{4}$ lb. of glycerin. In order to keep Schmid's solution, excess of caustic soda is added, and just before using it, it is saturated, by adding the requisite amount of chromium hydrate.

The following dyestuffs will dye on this mordant without further additions.—Magenta, Poirrier's Violet, Malachite Green, Phosphine, Methylene Blue, Alizarin, Alizarin Orange, Alizarin Blue, Anthracene Blue, Gallocyanine, Coerulein, Logwood, Quercitron bark, Persian berries, and Orchil. If chrome alum be likewise employed, Bismarck Brown, Victoria Blue, the Eosines and Alkali Blue may also be applied on this mordant.—H. I.

### *Wool, The Mordanting of, with Aluminium Salts.* G. Eberle and F. Ulffers. Färber Zeit. 1898, **9**, [20], 312—314. (See also this Journal, 1898, 1041.)

The authors are led to the following explanation of the mordanting of wool with aluminium salts. As mordants, acid salts of strong acids are employed because of the instability of the aluminium salts of the weaker acids. A normal alkali salt of a weak acid is added as an assistant, an interchange taking place, the strong acid uniting with the alkaline base and the aluminium with the weak acid. The wool substance, by taking up the aluminium and weak acid, helps in the hydrolysis of the new aluminium salt.

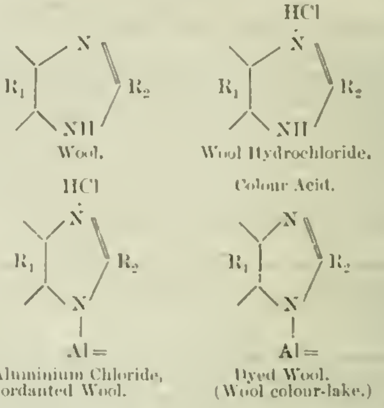
Some apparent exceptions to the conclusions arrived at in the above-cited paper receive the following explanation:—

*Sulphocyanic Acid* (according to Ostwald's measurements a strong acid) when used in the mordanting bath, exerts no unfavourable action on the subsequent dyeing. This is explained by the fact that in the hot bath this acid is decomposed into $CO_2$, $CS_2$, and $NH_3$. *Oxalic and Malonic Acid*, although comparatively strong, do not act disadvantageously proportionally to their strength. These acids undergo no decomposition in the bath, and the authors, in explanation of their anomalous behaviour, cite the observations of Lapraik (J. fur Prakt. Chem. **47**, 305—339) that these acids, in contact with chromic salts, form derivatives of two new and weak acids—chromo-oxalic and chromo-malonic. The potassium salt of the former $K_6Cr_2(C_2O_4)_{12}$, similar to potassium ferricyanide, and also potassium chromo-malonate, may be crystallised. Succinic acid forms no such compounds. Alumina is dissolved with ease by oxalic acid; less easily, however, by malonic acid (see table, this Journal, 1898, 1041), forming in both cases remarkably stable solutions; but it is not at all dissolved by succinic acid. In the first two cases the formation of similar compounds seems probable, and this is supported by the behaviour of these two acids in the mordanting bath, where malonic behaves as a weaker and oxalic as a much weaker acid than in aqueous solution.

It is found that the presence of free acid when mordanting with potassium bichromate favourably influences the subsequent dyeing. Here the free acid combines with the potassium and liberates chromic acid, which is reduced to chromic oxide by the cream of tartar, oxalic acid, or other oxidisable substance usually employed, and in this way the acidity of the bath is lost.

*Boro-sulphuric Acid* or "*Egalisol*" (G. Eberle and Co., Ger. Pat. 1895, 986) is mentioned as a useful weak acid and substitute for cream of tartar.

Finally, the authors regard wool as an amidine-like substance, and not as an Amido-carboxylic acid, and they represent the changes taking place during mordanting with, *e.g.*, aluminium chloride, and dyeing by the following formulæ:—


HCl
N
R₁
R₂
NH
Wool.
Wool Hydrochloride.
Colour Acid.
Al=
Wool-Aluminium Chloride, Mordanted Wool.
Dyed Wool. (Wool colour-lake.)

Amidines are strongly basic and weakly acid substances; the entrance of an acid group—*e.g.*, HCl—into the wool molecule during mordanting increases its acidity, and the wool can now unite with the alumina or other base. In the subsequent dyeing operation this acid group is expelled more or less completely and quickly according to whether it be a strong or weak acid, by the weak colour-acid of the dyestuff, the change taking place by mass action only. Hence, the presence of strong acid in the mordanting bath unfavourably affects the dyeing process. This view is supported by the observations recorded in the previous paper, and by the fact that the thorough washing of mordanted wool, in removing the acid, increases its affinity for dyestuffs.

—H. I.

### *Albumin, The Behaviour of the Sulphur in Ash-Free.* E. Harnack. Ber. 1898, **31**, [13], 1938—1913.

Ash-free albumin (prepared by treating copper albuminate successively with caustic soda and hydrochloric acid, and dialysing) contains the same proportion of sulphur as the original material, but in a different form, since it no longer blackens an alkaline solution of lead oxide when boiled with it. The albuminates of copper, zinc, and platinum do not blacken the lead solution, whilst lead albuminate does so; the sulphur in the original albumin, therefore, probably undergoes oxidation during the precipitation by the metallic solutions. Ash-free albumin, itself insoluble in water, forms soluble compounds with bases and acids: the latter are precipitated by excess of acid or by normal salts, but not by alcohol, ether, and phenol, whilst the former are also not precipitated by normal salts. The halogens form stable intermolecular compounds with albumin, some of which are of technical interest. The sulphur in these compounds is also in the oxidised form, and does not blacken lead solution. By further action of the halogens, sulphur is split off in three equal fractions, showing that the albumin molecule contains at least three atoms of sulphur, and has a molecular weight of not less than 5,000.—T. E.

### *Naphthol Purple or Violet.* Leipziger Färber- und Zeugdr.-Zeit. 1898, **47**, [11], 486.

This dyestuff, manufactured by the Fabrik Chemischer Producte in Thann und Mulhausen, is employed for dyeing deep violet-blue shades. Yellow resist styles are obtained by printing the goods, which have been previously padded with naphthol oleine, with a mixture of stannous chloride, chromium acetate, and Persian berries extract. By passages through a Mather and Platt apparatus, the colour is developed and the cotton is then dyed in a solution of Naphthol Purple, washed, hot soaped, treated with copper sulphate solution, and finally washed and dried.—H. I.

*Lanacyl Colours and Oxydiamine Violet, Fastness to Light of.* L. Lefèvre. Rev. Gén. des Mat. Col. 1898, **2**, [21], 344.

*Oxydiamine Violet,* although not absolutely fast to light, is faster than any other dyestuff which, alone or in combination, gives the same shade.

*Lanacyl Blues R and B B* are equal to the Sulphocyanines in fastness to light; *Lanacyl Marine Blue* is the least fast of the series. They do not withstand milling. [See this Journal, 1898, 1042.]—E. B.

*Azo-Dyestuffs, Insoluble, Employment of, in Cotton-Dyeing.* E. Blondel. Rev. Gén. des Mat. Col., 1898, **2**, [21], 342—344.

Comparatively few of the numerous dyestuffs of this group find employment in dyeing. Those most largely used are Paranitraniline Red, Naphthylamine Claret, and Ortho- and Metanitraniline, and Nitrotoluidine Oranges.

The first-named of these is the most important; the second is characterised by giving a colour which it is impossible to obtain from any other dyestuff; while the oranges are superior in shade to lead chromate oranges, and are, moreover, non-poisonous.

The colours are less fast to light than Alizarin Red, but they are sufficiently fast for general purposes. They are extremely fast to bleaching-powder, and withstand soaping and the action of aqueous acids at a boiling temperature. By reason of the last property they are adapted for use in dyeing cotton yarns, when these have to be woven with woollen yarns which are to be ultimately dyed in the piece, provided that copper salts are not used in mordanting the latter.

As the colours are fast to milling, they may also be advantageously employed in dyeing cotton yarns intended for the manufacture of mixed cotton and woollen tissues which have to be milled.

They are, further, suitably employed in dyeing tissues which are afterwards "raised," as they penetrate the texture of the tissues well, and they may be used in dyeing cotton in the state of "wool" and sliver, purposes to which Alizarin Red cannot be applied.—E. B.

*Indigo, The Proportion fixed on the Fibre, on Dyeing Fabrics with.* A. Binz and F. Rung. Zeits. angew. Chem. 1898, [42], 957—960.

According to the author, no systematic experiments have as yet been made to determine, in the case of Indigo, how much of the dye is taken up by the fabrics, and how much left in the bath. This is partly due to the fact that, whilst in the case of many other dyes the contents of the vat correspond with the amount of material introduced, this does not always hold good with the indigo-bath. Thus, the solution may become weaker through the decomposition of the Indigo White, by the formation of a deposit, as in the lime zinc-dust process, or by the oxidising action of the air. As to which is the best method of dyeing with Indigo, the author regards the question as an open one. The cleanest bath (*i.e.,* without deposit) is obtained by the use of hydrosulphite solution as a reducing agent. This is also advocated by von Georgievics (Der Indigo, Leipzig, 1892) and by the Badische Anilin und Soda Fabrik, and in spite of the fact of its being expensive, has been adopted in many dyeworks, both for wool and for cotton fabrics. It was therefore employed by the author in these experiments, thus eliminating the questions of the determination of Indigo in a deposit, and of the cost of recovering it. The charge for the main vat was prepared by mixing 300 litres of sodium bisulphite at 38° B., 500 litres of water, 35 kilos. of zinc dust (75 per cent. of zinc), and 22 kilos. of burnt lime. The clear hydrosulphite solution thus obtained was mixed with 120 kilos. of indigo paste (B.A.S.F. 20 per cent.) and 70 litres of soda lye at 40° B., and after warming for half an hour at 45° C., the whole was made up to 1,330 litres. The solution was completely clear and without deposit, and it was proved experimentally that its contents, as determined by evaporation of an aliquot portion, were in close agreement with the calculated numbers. The Indigo, mixed with the hydrosulphite, goes into solution quantitatively as Indigo White, and loss by "over-reduction" does not occur.

A series of quantitative determinations showed that after dyeing a number of fabrics during the first day, and then standing over-night, the bath was stronger in the morning, although no addition had been made. Thus, in the evening, 50 c.c. of the solution contained 0·1448 grm. as against 0·1528 the next morning. But this recuperative power gradually diminished, and at the end of the sixth day the vat contained a fine bluish-green substance in suspension, and 50 c.c. of the clear solution contained 0·1080 grm. A fresh charge was then necessary. The general conclusion arrived at on this point is that the mean percentage of Indigo White in the liquid in the dyeing vat remains constant, and there is no deposit, if the charge in the main vat is renewed at sufficient intervals.

To determine the percentage of Indigo fixed to the fibre, an experiment was carried out on a large scale. In the course of this, 7,759 articles of cotton, with a total weight of 46,527 kilos. were dyed, of which 4,249 (25,500 kilos.) received a dark colour, and 3,519 (21,527 kilos.) a light or medium colour. For this 3,204 kilos. of Indigo paste (pure B.A.S.F. 20 per cent.) corresponding to 640·8 kilos. of pure Indigotin were used. The amount of Indigotin fixed to the fibre after the souring, washing, and drying was determined in each batch by the glacial acetic acid method. (Brylinski. This Journal, 1898, 609.) The percentage of water in the cotton was allowed for by taking the mean results of several determinations. The waste liquor from the washing was collected in tanks, and after subsidence of the Indigotin, the clear supernatant liquid was run off and the amount of Indigotin in the residual deposit determined by the hydrosulphite method. The results thus obtained were:—

| | | Per Cent. |
|---|---|---|
| Indigo taken | 640·8 kilos. | |
| Indigo fixed on fibre | 563·22 kilos. | = 87·89 |
| Indigo in the washings | 72·06 „ | = 11·24 |
| Total | 635·28 „ | = 99·13 |

—C. A. M.

*Turkey Red [Calico Printing], Alkaline Discharge of.* W. Triapkine. Rev. Gén. des Mat. Col. 1898, [21,] **2**, 350—360. (See this Journal, 1898, 347 and 661.)

*Operations after Printing.*—The printed tissues should be immediately steamed. Delay in performing this operation is attended by a weakening of the discharge colours, owing to their becoming neutralised by the carbon dioxide and other acid gases present in the air, and to their excessive absorption of moisture. As glucose is hygroscopic, the colours printed on tissues prepared with it, become enfeebled more rapidly than those which are printed on unprepared tissues. The tissues dyed yellow shades of Turkey-red may be kept unsteamed longer than those dyed blue shades.

Sometimes the tissues on leaving the printing room, are cooled, especially when they have been dried at a high temperature, *e.g.,* 70°—75° C. The object of this is to prevent a reaction between the constituents of the colour-mixtures containing Indigo from taking place before the tissues are steamed, and also to enable the latter to absorb the necessary amount of moisture during the steaming operation.

*Steaming* is performed in a steam aniline ageing-machine or similar apparatus at a temperature of 100°—102° C. A large volume of steam is required. The steam supply-pipe should be about 4·5 cm. in diameter. It is advisable to attach a pressure gauge to it and to admit the steam into the steaming-chamber at a pressure of $\frac{1}{3}$—$\frac{2}{3}$ of an atmosphere. Sometimes the bottom of the chamber is enlarged so as to hold a layer of water, through which the steam is passed, the spray produced being retained by copper sieves placed over it.

The solvent action of the alkali on the Turkey-red lake is promoted by moisture in the steam, but there is a danger of the patterns "spreading," if the degree of moisture present exceed a certain limit. More moisture is required in steaming glucose-prepared than is needed for unprepared tissues.

The duration of steaming varies from ½ a minute to 4 minutes. It is determined by the nature of the Turkey-red lake, by the depth of engraving of the printing rollers, and by the composition of the discharge mixtures employed. White discharges require more steaming than colour discharges. In the case of white discharges of reds dyed with Alizarin, two passages through the machine, each of two minutes' duration, are given.

The action effected by steaming is twofold: first, the red lakes are resolved into soluble compounds, namely, sodium alizarates and silico-alizarates and analogous compounds, sodium aluminate, and fatty soaps; secondly, in the case of the yellow and green discharges, lead oxide is precipitated in the fibre, and in the case of the blue and green discharges, the Indigo present is reduced. Probably also, when the colour mixtures contain such compounds, tin and zinc oxides and silicates become fixed in the fibre in this operation.

*Re-oxidation of the Indigo.*—The blue and green colours may be re-oxidised by washing in running water. The colours thus treated, however, do not clear well in the subsequent operation. The tissues are, therefore, preferably submitted to exposure to moist air for 1—2 hours at a temperature of 20°—25° C. Frequently tissues are first aged in this manner and are then washed before being cleared. Deeper blues are thus obtained.

*Washing* is accomplished in tanks fitted with rollers. The tissues pass through full width and during their passage are treated with a spray of water. The upper rows of rollers are fixed above the tanks so that the tissues shall be freely exposed to the air, and the oxidation of the Indigo White thereby completed. To this end, it is useful to place a number of rollers above the tank, as in a continuous Indigo dyeing apparatus. The dimensions of the tanks are: length, 2—3·5 m.; height, 1·25—1·5 m. (width not given). The operation lasts 1½—2 minutes.

In washing, oxidation of the Indigo White is achieved, and various matters, as glucose in the prepared tissues and coloured alkali compounds, are removed. The clearing baths through which the tissues afterwards pass are less soiled and, consequently, last longer, when this preliminary treatment is adopted. Moreover, the red grounds of the patterns retain their brilliancy of colour better when they are thus treated prior to being cleared. On the other hand, the discharges ultimately produced, are less perfect than is the case when the operation is omitted. This is due to the formation in the printed parts of calcium-alizarates and silico-alizarates, from the lime salts present in the water.

It is, in all cases, advisable to wash tissues printed with full patterns before clearing them.

*Clearing.*—The tissues are treated at a temperature of at least 90° C. with a solution of sodium silicate. The operation is performed in a tank, which is commonly divided into three compartments, wide enough to admit two pieces of tissue side by side, the dimensions of each compartment being as follows: length, 3·5—4·5 m.; height, 1·25—1·5 m.; width, 1·25 (? 2·25) m. To increase the output, four pieces are sometimes passed back to back at the same time, two on each side of the machine, but this is practicable only in the case of reds dyed with Alizarin, since in other cases "marking off" occurs. For blue shades of red, the temperature of the bath must be maintained at the boil, for other shades a rather lower temperature suffices.

Reds dyed with Anthrapurpurin and Flavopurpurin are given a treatment of from ½ a minute to 1 minute, those dyed with Alizarin, one of 1¾—2 minutes' duration. In the former case a 0·5—1·75 per cent. solution of sodium silicate at 40° B., in the latter, one of 3—6 per cent. is used. The composition of the sodium silicate employed is of importance; it should approximate to the formula $2Na_2O,7SiO_2$.

When three tanks are used, it is advantageous to fill the first and last of them with the solution of sodium silicate, and to supply boiling water to the second. The solution in the first tank requires to be renewed after 150—300 pieces, 50 m. long, of yellow-shade reds or 500—800 pieces, of blue-shade reds, previously washed, have been passed through it. The consumption of sodium silicate in the latter case is 700—800 grms. of the solution at 40° B. per piece.

*Composition of Compounds on the Fibre.*—The colours containing lead, tin, and zinc oxides absorb silica in the operation of clearing.—E. B.

## PATENTS.

*Dyeing, Scouring, and Bleaching Hanks or Skeins of Yarn, Impts. in Machinery for.* W. H. Thorpe, Halifax. Eng. Pat. 25,564, Nov. 4, 1897.

An improvement on Eng. Pat. 19,226 of 1894 (this Journal, 1895, 34). In addition to the horizontal and reciprocating motion of the rollers, a vertical motion is imparted to them by causing the carriage bearing the hank-carrying rollers to ride over a semi-circular surface so that the hanks rise and fall in the vessel.

The roller carriage is also provided with feet upon which it may rest when taken from the machine for the purpose of changing the hanks.

A series of horizontal bars may also be placed between each section of hanks in order to separate the hanks and to prevent contact between them while they are moving up and down.—R. B. B.

*Dyeing, Sizing, Finishing or similarly treating Yarn or the like; Impts. in Machinery for.* J. Dean, J. W. Knowles, and H. Barker, Bradford, Yorks. Eng. Pat. 27,435, Nov. 23, 1897.

The machinery described is for the purpose of regulating the tension when yarn is stretched between rollers, *e.g.*, in the mercerising process, and is intended to obviate the use of rotating screws. The machine consists of a series of rollers upon which the yarn is placed and stretched by the operation of a hydraulic ram in such a manner that the horizontal centre line through the upper series of rollers is always exactly parallel with the centre line through the lower series. The machine is also provided with a series of nip rollers which squeeze the liquid from the yarn after the latter has passed over the upper roller and before it again enters the liquid in the vessel.—R. B. B.

*Dyeing* [*Jigger*], *An Improved Process of.* H. Laag, C. Rütler, and M. Rütler, Düsseldorf, Germany. Eng. Pat. 29,317, Dec. 11, 1897.

This invention refers to the dyeing of piece-goods in the jigger and is stated to be specially suitable for fabrics consisting of silk, or silk and cotton, in which defects are often caused by dyeing in the ordinary winch machine.

The dyebath is prepared in a special reservoir, and whilst the piece is being run through the machine the dye liquor is continuously run, preferably in sprays or jets, into the dye vat of the jigger.—R. B. B.

*Mordanting Cotton for Aniline Black, An Improved Process for.* A. C. Marot and A. Bonnet, Troyes, France. Eng. Pat. 26,538, Nov. 13, 1897.

At present the cotton is boiled in water and then treated with an aqueous solution of the mordant. According to this improvement the previous boiling is omitted and a portion of the water used for the mordanting bath is replaced by methyl alcohol. The advantages claimed are increased strength, fineness, and softness of the fibre; more rapid oxidation in consequence of more rapid drying, and the production during oxidation of formaldehyde which forms a natural protection to the fibre.—R. B. B.

*Black Shades on Cotton, A New Process for.* H. E. Newton, London. From the Farbenfabriken vormals F. Bayer and Co., Elberfeld, Germany. Eng. Pat. 28,090, Nov. 29, 1897.

Cotton is prepared with an alkaline solution of the sodium salt of $\beta_1$-$\beta_2$-dihydroxynaphthalene-$\beta_3$-sulphonic acid with the addition of Turkey-red oil, and is then dried. It is then passed through or printed with a solution of tetrazo diphenyl salt (diazotised benzidine) containing sodium acetate. The above mentioned substances may be replaced by certain homologues or analogues. It is claimed that in this manner a pure and intense black is produced which resists the action of acids, alkalis, washing, and light.—R. B. B.

*Fast Brown to Brown-Black Tints on the Fibre by Combining Naphthol with the Diazo Compound of Diamidocarbazol, Production of.* O. Imray, London. From The Farbwerke vormals Meister, Lucius und Brüning, Hoechst a/Main, Germany. Eng. Pat. 30,446, Dec. 23, 1897.

The browns produced on the fibre in cotton printing by combining diazotised benzidine or tolidine with β-naphthol, although fast to soap, are not so to light, and this defect cannot be improved by treating the dyestuff with copper salts. By employing *m*- or *p*-diamido-carbazol in place of benzidine or tolidine, the patentees produce a brown fast to light, which on treatment with copper salts is increased in fastness. The following quantities are given for the various solutions:—The diazo solution is made by dissolving 20 grms. of diamidocarbazol in 20 c.c. of hydrochloric acid (22° B.) and 100 c.c. of hot water. After cooling, 200 grms. of ice are added, then 30 c.c. of hydrochloric acid, and, finally, 52 c.c. of sodium nitrite solution (290 grms. per litre) are run in, the whole being finally made up to 500 c.c. The copper solution consists of 1 litre of water containing 160 grms. of crystallised copper chloride, 42·6 grms. of crystallised copper acetate, 80 grms. of crystallised sodium nitrite, and 66 c.c. of acetic acid (8° B.). Two recipes are given for the printing colour: (1) contains 500 grms. of tragacanth water (60 : 1000), 500 c.c. of diazo solution, and 50 grms. of crystallised sodium acetate; whilst (2) consists of 400 grms. of tragacanth water of the above strength, 500 c.c. of diazo solution, 100 c.c. of copper solution, and 50 grms. of crystallised sodium acetate. The naphthol grounding is made by dissolving 30 grms. of β-naphthol, 50 c.c. of caustic soda lye (22° B.) and 30 grms. of crystallised sodium ricinoleate in 1 litre of water. The printing colour, (1) or (2) is printed on the fabric prepared with the naphthol grounding, and the material is then washed. An alternative method is to pass the fabric grounded with β-naphthol through the diazo solution of diamido-carbazol, either mixed with copper salts or without, the fabric being finally washed as before.—T. A. L.

*Cloudy or Variegated Effects in Textile Fabrics, Production of; solely by Printing.* F. H. Schneider, Mylau, Saxony. Eng. Pat. 19,645, Sept. 15, 1898.

In this method of producing cloudy effects on fabrics by printing, the subsequent washing and milling operations are dispensed with, and printing cylinders are employed which are engraved with patterns in such a manner that the patterns appear when printed like undefined blended representations. The ground-work is also formed by printing with a cylinder so finely engraved that the drawing is undistinguishable in the print.—R. B. B.

## VII.—ACIDS, ALKALIS, AND SALTS.

*Alkali Chlorides, Electrolysis of.* F. Winteler. Zeits. f. Elektrochem. 1898, **5**, 10—15, 49—51, 217—221.

*Carbon Anodes.*—The author discusses the qualities of a good carbon anode. Attempts to close up the pores by boiling in paraffin or by enamelling have not been satisfactory. Although carbon resists pretty well the action of chlorine, it is comparatively easily attacked by nascent oxygen which gradually disintegrates the carbon and forms carbon dioxide and carbon monoxide. A dense carbon is less subject to disintegration than a light one; but the hardness is no criterion of its goodness, since a specimen of soft graphitic carbon was found to be very durable. The density of some carbons examined by the author varied between 1·113 and 1·790. If the density exceed 2·0 it may be concluded that the carbon is not pure. In order to test the durability of carbon for use as anodes, it should be used as anode in the electrolysis of a saturated solution of potassium chlorate or of a solution of sulphuric acid. Bad carbons soon disintegrate completely and colour the solution brown, whilst good carbons only produce a slight coloration, and their surfaces become covered with a fine velvety powder.

*Electrolysis of Potassium Chloride.*—The author's experiments were made in the apparatus described by Wehrlin (Zeits. f. Elektrochem. **3**, 450), with a solution containing 20 per cent. of potassium chloride. With a single diaphragm it was found that the current efficiency diminished as the concentration of the caustic potash increased, and that it was not possible to obtain a caustic potash solution containing more than 8 per cent. of KOH, unless the current efficiency were allowed to fall below 70 per cent. The introduction of another diaphragm, and the formation of a middle compartment, gave a higher current efficiency, but only at the expense of the additional electro-motive force required (increase from 2·9 V. to 4·4 V.).

The originally neutral solution of potassium chloride contained in the anode compartment; always became acid from the action of the chlorine on the water, oxygen being thereby liberated. It is already known that pure chlorine is evolved on the electrolysis of hydrochloric acid, provided the concentration of the acid be not allowed to fall below 10 per cent. The evolution of oxygen and the consequent destruction of the anodes might thus be prevented by keeping the anode liquor always acid to the extent of 10 per cent. This, however, would lower the yield of alkali, and of the two evils it is probably better, unless the production of specially pure chlorine were required, to choose the former and to employ the best possible carbon as anodes.

*Production of Hypochlorites.*—The author considers that Kellner's proposal to lead the electrolytic chlorine through a third vessel into which the cathode liquor has been introduced, is the most rational method by which to prepare hypochlorites, owing to the fact that they are decomposed both by nascent oxygen and nascent hydrogen.

*Production of Chlorates and Perchlorates.* — With reference to the electrolytic manufacture of chlorates, the author points out that, excepting some old investigations (also this Journal, 1898, 578—579), there have been no published accounts to show whether the chlorate formed is further oxidised to perchlorate under the conditions of manufacture. An examination of various samples of commercial chlorate prepared by electrolysis did not reveal the presence of a trace of perchlorate. In an experiment carried out in the electrolytic works at Vallorbes (Switzerland), 860 kilos. of crude chlorate were dissolved in just sufficient hot water to leave 10 kilos. undissolved. This residue, freed from ferric oxide, lime, and alumina, was then recrystallised and analysed, but no perchlorate could be detected.

The author then investigated the best conditions for obtaining a good yield of perchlorate by electrolysing solutions of sodium chlorate, and has arrived at the following conclusions:—

(1.) Neutral 20 per cent. solutions of sodium chlorate are invariably further oxidised to perchlorate with any current density, provided that a sufficiently low temperature be maintained.

(2.) The amount of oxidation increases with increase of current density, and is most favourable when the current density is 4—8 ampères and more per sq. dcm.

(3.) If the cathodic current density fall below 2 ampères, a slight reducing action takes place at the same time as the oxidation at the anode.

(4.) If the electrolyte be not artificially stirred, acid layers form around the anode and affect the process of oxidation. The time at which they are formed and their concentrations depend on the current density.

(A solution containing 50 per cent. of sodium chlorate behaved in a similar way.)

In these experiments the current efficiency (91—95 per cent. oxidation) remained constant so long as an excess of at least 10 per cent. of unchanged chlorate was present in the solution.

At higher temperatures, *viz.*, 50°—60° C., and with varying current densities, solutions of sodium chlorate behaved in the same way as solutions of potassium chlorate.

In general, experiments with the latter substance showed that the current efficiency gradually fell off to about 14—20 per cent., whilst even a slight reduction was noticed in some cases.

At 100° C., reduction of the chlorate takes place instead of the oxidation which occurs at low temperatures.—J. S.

*Potassium Para-Tungstate, The Action of Metallic Sulphates on.* L. A. Hallopeau. Comptes Rend. 1898, **127**, [17], 620—621.

The sulphates of the metals react with potassium para-tungstate, giving double tungstates analogous to those obtained with sodium tungstate; difficulties are, however introduced, owing to the smaller solubility of the potassium salt. The following double salts are described:—Potassium and magnesium para-tungstate, obtained by mixing solutions containing equivalent quantities of potassium tungstate and magnesium sulphate. It is nearly insoluble in cold water, but can be recrystallised from hot water. Analysis indicated the formula $12WO_3, 5(\frac{2}{3}K_2O + \frac{1}{3}MgO) + 24Aq$. This salt crystallises in prisms or hexagonal plates, acting on polarised light with extinctions parallel to the direction of the axes. It loses 17 molecules of water on heating at 100° C.

Potassium and manganese para-tungstate is obtained as a yellowish-white precipitate, quite insoluble in water. The microscopic crystals are in the form of rhombohedral prisms, with modified edges, very refractive, and acting strongly on polarised light. They have a composition corresponding to the formula $12WO_3, 3K_2O, 2MnO + 16Aq$, and lose 10 molecules of water at 100° C.

Sulphate of zinc gives a slightly soluble crystalline double tungstate, but it could not be purified from potassium tungstate.

Cadmium sulphate and the sulphates of most of the other metals give amorphous precipitates with potassium tungstate, insoluble in water; but these precipitates are often mixed with tungstic acid, especially when the solution of the sulphate is acid.—J. F. B.

*Alkali Nitrites, Preparation of Pure.* E. Divers. Proc. Chem. Soc. 1898, [199], 222.

Alkali nitrites are easily obtained pure and in concentrated solution by passing nitrous gases containing slight excess of nitric oxide into concentrated solutions of pure potassium or sodium hydroxide or carbonate, out of contact with air. With ordinary care, no nitrate is formed; it is a current mistake to suppose that it is.

Both nitrites are slightly yellow, and their concentrated solutions markedly yellow. The sodium salt melts at 213° C.; at 15°, 6 parts of water dissolve 5 parts of it. Its crystals are often very large, but very thin, flattened prisms, moderately deliquescent. Sodium nitrite can be readily purified by recrystallisation; potassium nitrite cannot. Potassium nitrite occurs only in minute, short prisms, and is soluble in one third of its weight of water. It is exceedingly deliquescent, but its crystals are actually anhydrous, although Lang and Hampe independently found them to contain $\frac{1}{2}H_2O$.

*Sodium Chromate.* Leipziger Färber und Zeugdr. Zeit. 1898, **47**, [11], 464.

*See under* VI., *page* 1112.

*Sodium Bicarbonate, Detection of Monocarbonate in.* M. Kohn. Archiv. der Pharm. **236**, 321.

*See under* XXIII., *page* 1182.

## PATENTS.

*Salt, Improved Means Applicable for Use in the Electrolysis of.* [*Diaphragm Electrode.*] J. Hargreaves, Farnworth-in-Widnes. Eng. Pat. 17,161, July 20, 1897.

Strengthened electrode diaphragms are formed by covering wire cloth or perforated plate with a thin layer of temporarily retentive material, such as paper or clay. A superposed coating of hard or dense material, such as Portland cement, is applied, which is then covered with a soft or porous material, such as asbestos cloth, slag wool, clay, or the like. The temporarily retentive material is then dissolved or removed, leaving a narrow space between the cathode and the hard casing. The side of the diaphragm remote from the cathode is given a porous covering of asbestos and lime, indurated with a solution of silicate of soda. The electrodes may be arranged either vertically or horizontally, and the diaphragm separates the anode chamber from the rest of the cell. The chamber is formed of two lead plates coated on their inner sides with pitch. The anodes are inserted through perforations in them, and the walls are formed by casting cement against the layer of pitch. The anodes may form the grid on which the salt rests, and a charging orifice, with a sealed cover, is provided for the admission of the salt to the saturating chamber. The weakened brine from the electrolytic cell, which may be injected by steam, enters by an inlet beneath the grid, and at the upper end of the vessel are outlets for the strong brine and the chlorine gas.
—G. H. R.

*Solutions of Carbonate of Soda resulting from Electrolysis of Chloride of Sodium; Impts. in and relating to the Treatment of, and in Apparatus applicable for Use therein.* J. Hargreaves, Widnes. Eng. Pat. 21,178, Sept. 15, 1897.

One object of the invention is to enable porous diaphragms, permitting the passage of sodium chloride from the anode to the cathode compartment of electrolytic cells, to be efficiently employed. Solutions containing varying proportions of sodium chloride and of sodium carbonate are thus obtained, and the cells are so constructed that the liquid may be drawn off at need from the different compartments, and conveyed regularly by ducts to a series of conveniently communicating evaporating pans. At a certain stage of concentration of portions of the liquid, a solution is obtained containing two equivalents of sodium chloride to one of the carbonate, and the salts removed, contain sodium chloride and monohydrated sodium carbonate, other portions of the liquid giving crystals of the latter only. The methods of treating such solutions of varying constitution, in series, so as to obtain "pure" and "impure" sodium carbonate solutions, are described.

The summarised process as regards the solutions generally, consists, according to the second claim, in separating monohydrated sodium carbonate; then, after concentration, "cooling the mother-liquor, and further separating therefrom carbonate of soda in the form of crystals, treating the resulting mother-liquor with carbonic acid gas to obtain bicarbonate of soda; and then neutralising the resulting solution of chloride of sodium" with brine mother-liquors, containing magnesium and calcium chlorides.—E. S.

*Oxalic Acid and Oxalates, An Improved Method or Process of Preparing.* Dr. M. Goldschmidt, Charlottenburg, Germany. Eng. Pat. 26,172, Nov. 10, 1897.

The inventor has previously described a method of obtaining sodium formate, consisting in heating sodium carbonate in a current of carbonic oxide gas under pressure, in Eng. Pat. 17,066, 1895 (this Journal, 1896, 652), and he now shows how from sodium formate, the oxalate may be quantitatively obtained. The transformation has been previously partially effected with very small quantities, by heating sodium formate rapidly to 440° C.; but with the disadvantage that at a lower temperature a carbonate is formed, so that the yield of oxalate is small. This difficulty is overcome by heating a mixture of 4 parts of crystallised sodium formate with 5 parts of anhydrous sodium carbonate, contained in a glass or iron vessel, in a bath of molten lead to from 400° to 410° C. Air is preferably excluded, and provision is made for collecting the hydrogen evolved in the reaction. The product consists of sodium oxalate and carbonate, which is treated with water at 33° C. to dissolve the carbonate.
—E. S.

*Chlorine, Impts. in Obtaining.* A. R. Scott, Kirkintilloch, N.B. Eng. Pat. 27,221, Nov. 20, 1897.

The apparatus is substantially the same as that employed in the process described in Eng. Pat. 12,074, 1893 (this

Journal, 1893, 833), in which hydrochloric, nitric, and sulphuric acids are made to react together in a continuous manner; but instead of re-forming nitric acid from the nitrogen compounds, the nitrosyl sulphate produced in the operation is denitrated by steam, and the nitrous anhydride thereby liberated, is mixed with air in proportion to form nitric oxide, which is then used in continuing the process.
—E. S.

*Acetic Acid, Impts. in the Manufacture of.* E. G. Scott, Liverpool. Eng. Pat. 223, Jan. 4, 1898.

"Pyroligneous salts" [crude acetates] are introduced to a vacuum pan provided with a stirrer and a steam jacket, by a valved hopper, and after a vacuum has been formed, a mineral acid is added through another inlet. The stirrer is then set in action and heat applied. The distillation of the acetic acid and its condensation and collection are effected in the usual manner.—E. S.

*Alkali Salts, especially Alkali Chlorides; Impts. in the Electrolysis of Solutions of.* O. Imray, London. From The Oesterreichischer Verein für Chemische und Metallurgische Production, Aussig-on-the-Elbe, Bohemia. Eng. Pat. 16,129, July 23, 1898.

In this process for the electrolysis of solutions of alkali salts, the electrodes are placed side by side, and, if necessary, at different levels. The anode is covered by a bell which extends below it, and fresh electrolyte is fed into this compartment continuously, while the alkaline lye produced is continuously removed from the cathode compartment, and the layer formation of the anode and cathode liquids is maintained without the use of a diaphragm, so that the products formed at the electrodes are continuously separated.—G. H. R.

*Caustic Alkalis, Impts. in the Manufacture of, from Alloys of Lead with Alkali Metals.* C. E. Acker, East Orange, U.S.A. Eng. Pat. 14,269, June 28, 1898.

The apparatus consists essentially of a cast-iron cylindrical pot set in a fireplace, having a pipe terminating in a bell reaching to near the bottom of the pot and having nearly the same diameter. The pipe attached to the bell is adapted to receive a current of steam, and has mounted on it a hood, the sides of which extend downwards into a lute, which lute is sealed by molten caustic alkali. The pot is charged with an alloy of lead and an alkali metal in a fused state, and steam being passed in, the alloy is depressed to the mouth of the bell, where the steam acts continuously on its molten surface. The hydrogen disengaged by the reaction escapes upwards and then through the lute of melted caustic alkali. Channels are provided in the outer shell of the lute, whereby the excess of the alkali hydrate can be discharged. The hydrogen passes out by apertures in the cover. There is no access of air to the contents of the pot, which are acted upon solely by the steam.—E. S.

*A Portable Spout to attach to Drums containing Ammonia or other Strong or Obnoxious Chemicals.* E. Beckton, London. Eng. Pat. 25,898, Nov. 8, 1897.

The invention is described as "a conical, curved spout, fitted with a small air-tube fixed to the back, and so constructed as to pass into the drum when in use. The spout has a rubber seating to make a joint on the neck of the drum, and a slotted lug fixed to each side to receive bolts." The spout is adapted to prevent the efflux of noxious or irritating vapours.—E. S.

## VIII.—GLASS, POTTERY, ENAMELS.

*Ceramic Pastes, Study on the Dilatation of.* M. Coupeau. Bull. de la Soc. d'Encouragement, 1898, **3**, [10], 1274—1309.

The addition of quartzose sand to clay, augments dilatation in proportion to the quantity added. With a high proportion of quartz, the dilatation of products baked at 1,000° C. appears to be very similar to that of pure quartz. The curve of dilatation is also the same, increasing rapidly towards 500° C., but ceasing to increase and even somewhat decreasing above 600° C. The absolute dilatation value, however, is less than that of pure quartz, owing to the lower dilatation of the particles of clay which surround the sand and hinder its free expansion.

The addition of calcined flint to a clay augments dilatation much more than the addition of quartz. The greater part of the dilatation, in this case, occurs between 200° and 300° C., and this accords with one of the known properties of calcined flint, *viz.*, its sudden and enormous expansion at 200° C.

In the case of the sand of Decize, which is rich in alkalis, dilatation was found to decrease regularly in proportion as the temperature of the baking was increased. Decize quartz, under the influence of fluxes, is converted into amorphous silica. Quartzose sand, under certain conditions, undergoes the same change as calcined chalcedony, producing enormous increase in dilatation, with an irregularity at 200° C. The finer the quartz and the higher the temperature of the baking up to 1,370° C., the more readily does the change take place. This form of silica is, however, not stable, but becomes transformed into the amorphous variety in proportion to the temperature, quantity of flux, and the degree of fineness of the sand. Pure quartz fluxes towards 1,800° C., and yields a vitreous amorphous silica. Quartzose sand with the addition of 3 per cent. of lime is transformed into amorphous silica at 1,600° C. Decize sand, rich in alkalis, commences to undergo this change at 1,200° C. Very fine sand mixed with relatively pure clay, after showing enormous dilatation when baked at 1,270° C., exhibits a decreased dilatation when baked at 1,370° C.

The results on the whole indicate that the firing should be regulated according to the composition of the ceramic paste, the nature of the flux, the fineness of the ingredients, and the completeness of their intermixture.—H. H. B. S.

*Ceramic Pastes, Observations on Coupeau's Study on.* G. Vogt. Bull. de la Soc. d'Encouragement, 1898, **3**, [10], 1309—1316.

Coupeau's researches confirm the following points:—1. Quartz augments the coefficient of dilatation in proportion to the degree of fineness to which it is ground. 2. Felspar, and vitrifiable substances generally, decrease it. 3. The coefficient of dilatation increases for non-vitrifiable pastes with the increase of the temperature of the baking. The following new facts are established:—1. Clays, according to their composition, augment or decrease the coefficient of dilatation. 2. With vitrifiable pastes, the coefficients of dilatation decrease when the temperature of baking is raised.

It is suggested that the investigation should be completed by studying the influence of the usual colouring oxides upon the coefficient.—H. H. B. S.

*Ceramic Pastes, On some Physical Properties of.* M. Le Chatelier. Bull. de la Soc. d'Encouragement, 1898, **3**, [10], 1317—1321.

The results obtained are chiefly of interest in so far as they indicate the direction in which new researches should be undertaken. These should embrace, in the first place, the study of very aluminous pastes free from quartzose sand, the mechanical properties of which appear to be somewhat remarkable. In the second place, the precise conditions necessary for the vitrification of silica in porcelain should be defined, as it seems, from certain facts, that only the alkalis, properly speaking, are capable of producing this vitrification.—H. H. B. S.

*Red Glass.* Bull de la Soc. d'Encouragement, 1898, **3**, [10], 1329; from Sprechsaal, 1898, **31**, 456.

A red glass, similar to that produced by gold or copper, can be obtained by the use of a certain quantity of the following flux of oxysulphide of antimony (glass of antimony):—Silica, 100; carbonate of soda, 50; lime, 20; sawdust, 7·5; glass of antimony, 4. A smaller proportion of antimony gives a yellow.—H. H. B. S.

*Glasses Coloured Blue by Chromium Oxide.* A. Duboin. Ber. **31**, [13], 1977—1979.

On the basis of the observations of St. Clair Deville, Caron, and Gaudin, on the action of chromium oxide in colouring gems, the author attempted to apply the same to the production of blue glass. For this purpose he employed a mixture of $SiO_2$, 135 parts; $Al_2O_3$, 51 parts; $CaCO_3$, 150 parts; and $K_2CrO_4$, 9 parts, which, on fusion for several hours in a reducing atmosphere, produced a blue glass, though only of inferior lustre. Better results were obtained by replacing the calcium carbonate by 295·5 parts of $BaCO_3$ and reducing the chromate to 7 parts, the glass obtained being very handsome. A very fine glass of greater fusibility was prepared by modifying the proportions first taken, so as to contain 148 parts of barium carbonate and 75 parts of calcium carbonate.

The following mixture: $B_2O_3$, 4 parts; $Al_2O_3$, 1 part; $K_2CrO_4$, 1 part, gives a beautiful blue glass, but one readily attacked by acids. If borax be used to replace boric acid, the alumina is reduced, a metallic skin, chiefly aluminium, having been obtained in one instance, where the silica corresponded to two-fifths of the alumina.

A mass resembling that of Jena glass, and composed of $SiO_2$, 84 parts; $B_2O_3$, 39 parts; $Al_2O_3$, 16 parts; $BaCO_3$, 157·6 parts; $K_2CrO_4$, 7 parts, gives a blue glass.

Attempts were made to employ aluminium or calcium carbide as a reducing agent. In the former case the results were unsatisfactory, though probably at higher temperatures they would be more successful. The blue obtained in presence of carbide is inferior.

No good results could be obtained with glass masses of the ordinary type or with ready-made glass.—C. S.

## PATENTS.

*Potters' Glazes, Kilns for Calcining or Fritting Materials used in the Production of; Impts. in.* W. Owen, Burslem, Staffordshire. Eng. Pat. 27,841, Nov. 26, 1897.

This kiln is devised primarily with the object of enabling the potter to mix the lead used in glazing, with the other glaze materials prior to the fritting, with the view of rendering it less injurious to the workers. This is not possible with the usual form of kiln, owing to the firebrick bottom being attacked and destroyed by the frit. In the improved kiln, the bottom is formed of quarries or fireclay bricks resting upon an iron plate, having an upward turned flange, and built into the walls of the kiln. The quarries are, of course, still attacked by the frit, but the flanged plate prevents them from sinking, and the frit from being lost. As the quarries become destroyed they are chipped from off the plate and replaced by fresh ones. Beneath the plate is an air chamber, communicating with the outside air by openings made in the kiln walls, and with the firebox by openings in the fire-bridge. The roof of the kiln is made higher at the front, nearest the firebox, thus causing the furnace gases to reverberate on to the frit in their passage to the chimney. By a further modification the kiln can be adapted for the continuous production of silicate of lead. —H. H. B. S.

*Water- and Acid-proof Non-conducting Articles of Clay, An Improved Process for the Manufacture of.* W. van der Berg, Dulken, Germany, and T. Haass, Mutteuz, near Bâle, Switzerland. Eng. Pat. 12,852, June 8, 1898.

Clay articles are treated directly after burning or drying, and therefore before they can take up moisture, with molten asphalt or pitch, with or without the addition of resin, gum, fat, or oil.—H. H. B. S.

*Enamels, Glassware and Porcelain, Impts. in Furnaces or Kilns for Baking.* A. J. A. Berthelier, Lyons, France. (Under Internat. Convent.) Eng. Pat. 16,550, July 29, 1898.

This furnace has two muffles, placed one above the other, made of cast-iron or fire-clay, according to requirements. By means of a separating plate with apertures and a longitudinal partition, the hot gases are made to travel around the muffles before escaping by the chimney. Vertical flues are arranged at the corners to accelerate the draught, and to convey a portion of the heated gases directly to the top of the upper muffle. The furnace has also movable bars, to allow the fire to be dropped when the proper degree of baking has been reached.—H. H. B. S.

# IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

*"Phyllades" [Slate Material] of the Ardennes; Compared with the Slates of North Wales.—Part I.* T. M. Reade and P. Holland. Proc. Liverpool Geol. Soc. 1897–98, 274—293.

The chemical composition and physical characteristics of the rocks referred to have been studied and compared. The following are the results of the chemical analyses:—

| — | 1. Quartzite. | 2. Quartzite. | 3. Phyllade Ottrélitifère. Blue-Black. | 4. Phyllade Aimantifère. Green. | | 5. Phyllade Devillien. Violet. | 6. Penrhyn Slate. Cambrian. Purple. | 7. Penrhyn Slate. Cambrian. Purple. | 8. Egryn Slate. Cambrian Barmouth. Green. | 9. Llanllyfni Slate. Silurian Green. | 10. Velenhelli Slate. Cambrian Llanberis. Blue. | 11. Delabole Slate. Cornwall. Grey. | 12. Vermont Slate, U.S.A. Red. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Silica | 92·48 | 87·30 | 57·80 | 57·89 | 57·73 | 60·56 | 57·75 | 63·01 | 59·16 | 57·27 | 63·30 | 56·75 | 60·30 |
| Alumina | 3·11 | 3·61 | 23·96 | 20·40 | 20·17 | 19·72 | 16·44 | 16·28 | 18·42 | 17·60 | 14·99 | 21·81 | 13·75 |
| Ferric oxide | 0·45 | 1·40 | 3·10 | 4·93 | 5·28 | 6·57 | 10·84 | 8·42 | 7·67 | 4·70 | 8·75 | 1·36 | 5·03 |
| Ferrous oxide | 1·47 | 3·22 | 3·40 | 3·08 | 4·10 | 1·86 | 1·66 | 0·66 | 2·92 | 6·40 | 1·53 | 7·20 | 0·86 |
| Manganous oxide | Trace | 0·27 | 0·42 | Trace | .. | 0·11 | 0·25 | 0·42 | 0·14 | 0·46 | 0·25 | Trace | Trace |
| Titanic oxide | 0·41 | 0·37 | 1·01 | 0·89 | 0·81 | 0·55 | 1·27 | 0·58 | 0·17 | 1·20 | 1·04 | 0·71 | Trace |
| Lime | .. | .. | 0·43 | .. | .. | Trace | 0·23 | 0·72 | 0·04 | 0·17 | 1·08 | 0·84 | 3·62 |
| Magnesia | 0·13 | 1·10 | 1·59 | 2·12 | 2·42 | 1·87 | 2·36 | 1·95 | 2·54 | 2·28 | 1·63 | 2·48 | 5·20 |
| Potash | 0·84 | 0·22 | 3·02 | 4·71 | 4·79 | 4·61 | 4·23 | 2·98 | 4·01 | 3·79 | 2·84 | 3·72 | 3·84 |
| Soda | .. | 0·59 | 0·85 | 1·26 | 1·13 | 0·55 | 1·30 | 2·14 | 0·80 | 1·20 | 1·92 | 0·97 | 0·72 |
| Carbonic acid | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | 3·77 |
| Phosphoric acid | .. | .. | .. | Trace | .. | 0·09 | 0·21 | Trace | 0·07 | 0·11 | 0·07 | Trace | Trace |
| Approximate carbon | 0·25 | .. | 0·35 | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| Combined water | 1·13 | 1·85 | 4·22 | 3·08 | 3·08 | 3·85 | 3·35 | 2·92 | 4·00 | 4·47 | 2·88 | 4·23 | 2·91 |
| | 100·27 | 99·93 | 100·15 | 99·59 | 99·81 | 100·37 | 99·89 | 100·08 | 99·94 | 99·65 | 100·18 | 100·07 | 100·00 |
| Silica as quartz | .. | .. | 38·23 | 33·10 | | 35·36 | 34·06 | 42·26 | 36·45 | 33·71 | 43·03 | 33·24 | 38·76 |
| Specific gravity | 2·695 | 2·690 | 2·878 | 2·854 | | 2·846 | 2·852 | 2·852 | 2·840 | 2·838 | 2·883 | 2·841 | 2·828 |

The following is an abridged description of the rocks examined and their chief characteristics:—

#### *The Quartzites.*

No. 1 is a typical quartzite from the Cambrian rocks of the Ardennes, near Fépin.

No. 2 is a Devonian quartzite from the same locality.

#### *Phyllades of the Massif of Rocroy.*

No. 3. Phyllade Ottrélitifère - Revinien Monthermé. Taken on the right bank of the Meuse, between Monthermé and Deville. A coarse, blue-black, roughly fissile schistose rock; scales of white mica on the surface.

No. 4. Phyllade Aimantifère. Taken lower down the Meuse than No. 3. A true slate used for roofing purposes. It is of a light grey-green colour.

No. 5. Phyllade Devillien. Collected between Haybes and Fépin. A true slate of a violet colour, and extremely smooth and "satiny" on the cleavage planes. Used for roofing purposes.

#### *The Slates of North Wales.*

No. 6. A Penrhyn slate taken at a quarry at Bethesda, Carnarvonshire. This slate, which is of Cambrian age, is distinguished by the large proportion of titanic oxide it contains, which exceeds that found in any of the other specimens, whether from Wales or the Ardennes.

No. 7. Penrhyn roofing slate, Carnarvonshire (Cambrian). The colour is almost identical with No. 6. The surfaces of the cleavage planes have, however, that curvilinear ridgy grain which distinguishes these Welsh roofing slates from the phyllades. It is a very silicious slate, and considered to be the strongest and most durable for roofing purposes.

No. 8. Egryn slate. Quarry north of Barmouth, Merionethshire (Cambrian). A grey-green slate, darker in shade than No. 4, and having a soapy feel intermediate between No. 4 and No. 6. The microscopic characteristics and chemical composition approach those of a phyllade.

No. 9. From a slate quarry at Llanllyfni, near Pen-y-groes in the Nantlle Valley, Carnarvonshire (Silurian). This is not a true type of the slates of the Nantlle Valley, as it does not cleave truly. It was selected for comparison with the phyllades as approaching them in microscopic appearance.

No. 10. Roofing slate, known as Velenhelli Ton (Cambrian). A very strong, coarse slate of a dark-blue colour. It is very durable, but wanting in the artistic appearance of the glossy small-sized phyllade slates.

#### *Other Slates.*

Nos. 11 and 12. For the sake of comparison, the authors have added analyses of slates from Delabole, Cornwall, and Vermont, U.S. The latter resembles in structure the Welsh roofing slates, but the colour is a bright red.

#### *General Remarks.*

There is a strong family likeness between some of the Welsh slates and the phyllades, but the Welsh slates are, on the whole, richer in iron. It is interesting to find that a comparison of the phyllades and slates from such distant localities as the French Ardennes and North Wales shows that their physical qualities are largely dependent upon their chemical composition, and that, given the same chemical composition, the slates approximate to each other, whether from Wales or the Ardennes. The coarser character of the Welsh slates can be traced to the preponderance of silica in the form of quartz.—H. H. B. S.

### *Fire-Clays, The Influence of the Size of Particles on the Resistance of, to Heat and to Fluxes.* H. O. Hofman and B. Stoughton. Eng. and Min. J. 1898, **66**, 485.

The possibility of laboratory tests of clays, which are ground uniformly fine for the purpose, not being reliable for materials which are practically used in the raw lumpy state, led to experiments to ascertain if the inference that the results might be below the truth was correct. The size of the Seger cone chosen, was ¾ in. at the base and 2⅜ ins. high. The results of fusing and fluxing tests of the clay mixtures, both in the ordinary and powdered states, show only a slight difference, and prove that the samples, in testing, can safely be ground fine and compared with the small-size Seger cones in the ordinary Deville furnace.

—A. W.

### *Clays, Modified Methods for Determining Fusibility of.* H. O. Hofman. Eng. and Min. J. 1898, **66**, 485.

The author describes some experiments, made in conjunction with Messrs. J. L. Newell and G. A. Rockwell, wherein the method of testing was modified by changing the Bischof standard, corresponding to Seger cone No. 36, to Seger cone No. 26, so as to find out how far the non-refractory clay stood below the point of being a fire-clay, rather than to ascertain how much refractory material would have to be added to bring it up to No. 36 standard. The advantages are, the lower temperature of the test, the saving in time and gas-carbon, and the longer life of the furnace. 1 grm. of the clay to be tested was mixed severally with 0·1, 0·2, 0·3, &c., grm. of silica-alumina flux, made up of finely-ground quartz containing 99·88 per cent. of silica and an alumina containing 98·46 per cent. of the pure oxide. The various mixtures were worked in the usual way and heated with a No. 26 Seger cone until the latter was melted down, which required about 35 minutes.

This modification acts as a valuable guide in making up clay mixtures, although, of course, it does not altogether determine the suitability of a clay for a specified purpose. The results obtained, however, in some cases gave a true prediction of what afterwards happened on a large scale. The method is applicable in testing the action of basic charges on acid material, and the resistance of clays to corrosion by fusible salts, oxides, &c.—A. W.

### *Hydraulic Cement, Notes on.* A. W. Hale. Eng. and Mining J. **66**, [19], 544.

The author discusses the relative value of cement and other coatings for checking the percolation of water through brick or stone work, his observations being principally based upon the results of an investigation carried out in connection with the construction of the new Croton aqueduct of New York. The experiments embraced a wide range, including such substances as linseed oil, shellac, and iron varnishes, aluminium acetates, oleates and stearates, sodium silicate and calcium chloride, varnish, pitch, tar, and a number of proprietary compounds.

Hydrocarbons, usually regarded as most suitable for waterproofing brick or stone, are found, on exposure to air, to oxidise and disappear. Portland cement gave the best results, and the only coating which ranked with it in impermeability was a hydrocarbon compound, which after a time peeled off. Compounds of alumina with organic acids, and of lime with silicic acid, shared the same fate. The permeability of a brick, when coated with successive washes of cement applied with a brush, diminished much more rapidly than when coated with an increased thickness of cement applied with a trowel. The percolation through a brick coated with four washes of cement diminished under a water pressure of 80 lb. per sq. in., and at the expiration of two months ceased altogether, the brick then remaining impervious under a pressure of 200 lb. per sq. in.

—H. H. B. S.

### *Sheet Zinc, A Substitute for Wall Paper.* Papier Zeit. 1898, **23**, 3127.

Metallic zinc, rolled to the thickness of paper, is now being used in the States and Germany for the decoration of rooms in place of ordinary wall paper. The face is enamelled to resist moisture and to imitate stone or marble; and the material is applied to the plaster with paste, adapting itself readily to surfaces of any shape.—F. H. L.

### *Portland Cement, Detection and Estimation of Free Lime [$Ca(OH)_2$] in Set.* W. N. Ljamin. Zap. Imp. Russ. Tech. Obshtsch. 1898, **32**, 75.

*See under* XXIII., *page* 1183.

## PATENTS.

*Cement, Improved Means applicable for use in the Manufacture of.* F. D. Cummer, Cleveland, State of Ohio, U.S.A. Eng. Pat. 18,246, Aug. 5, 1897.

An improved method of treating liquid slurry by mixing it with sufficient dried and powdered slurry to bring the whole to the condition of a plastic mass, which is then dried and calcined. -H. H. B. S.

*Treating Wood for the Purpose of rendering it Non-Inflammable or for Preserving it, Impts. in.* H. V. Simpson, Upper Norwood. Eng. Pat. 28,938, Dec. 7, 1897.

The wood is first exhausted in a vacuum chamber under moist heat, and is then subjected to the "action of water spray or steam, or methylated spirit, with or without the addition of a saponifying medium," and preferably under pressure. It is then dried, placed under exhaust, the impregnating solution run in and pressure applied. The solution is subsequently drawn off, and the wood dried. For the purpose of preservation, the wood is first impregnated with ammonium sulphate or phosphate, or both, and then with a salt of zinc or aluminium, or both.—H. H. B. S.

*Ceramic Stone, Impts. in the Manufacture of.* L. A. Garchey, Lyons, France. Eng. Pat. 2008, Jan. 25, 1898.

This invention is based upon the principle that powdered glass becomes devitrified and porcelain-like on being heated nearly to fusion. Any waste glass may be used for the purpose, and it may be mixed with slag, scoriæ, clays, or any other materials that can be vitrified. The materials are pulverised and graded by sifting, so that by blending different grades, the appearance of certain rocks can be imitated. If the materials employed are not easily fusible, the particles are agglutinated by silicates or gum, so as to form a paste, which can be roughly shaped to the desired form. The roughly-shaped mass or the dry material, as the case may be, is then introduced into a highly-heated mould of refractory material "lined with sand and subjected to such heat as may be required to effect devitrification and agglomeration. (See also this Journal, 1896, 545.)

*Artificial Stone known as Reconstructed Granite, Impts. in and relating to, and in Process of Making the same.* W. Courtenay, New York. Eng. Pat. 17,158, Aug. 9, 1898.

This invention is an improvement on Eng. Pat. 7184 of 1891, and refers to the making of building blocks by burning a mixture of pulverised granite, fireclay, and felspar. In the present patent a more fusible mixture is used for the face than for the interior, and, whilst the former is fused, the latter is only baked. By this device a better result is produced at a lower temperature and at less expense than when the blocks are made of one composition throughout. The mixture for the interior is made of 100 parts of granite, 10 parts of felspar, and 40 parts of fireclay; that for the facing, of 100 parts of granite, 30 parts of felspar, and 20 parts of fireclay. Sufficient water is added to make the mass plastic. To prevent discoloration of the blocks during burning, they may be glazed with zinc-white, white-lead, or other suitable material, removing the glaze after burning by means of a sand blast or by treatment with hydrofluoric acid. An improved method of burning the blocks is also claimed, consisting in supporting them upon tiles and firebricks with their faces protected by asbestos paper.

—H. H. B. S.

*Calcining Cement Materials, A Furnace or Kiln for.* V. Kjeldsen, Ennigerloh, Westphalia, Germany. Eng. Pat. 19,465, Sept. 13, 1898.

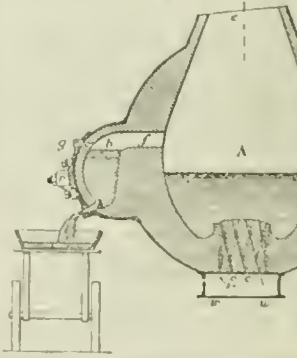
This is a modification of the common form of kiln, in which the fuel and charges are introduced in successive layers. The wall of the kiln is pierced by a number of holes, distributed over its entire surface, which can be opened or closed at will, thus enabling air to be admitted at any part where combustion may appear to be retarded.—H. H. B. S.

# X.—METALLURGY.

*Converter [Steel], The Paul David "Selecteur" or.* P. L. Barthe. Eng. and Min. J. 1898, **66**, 487—488.

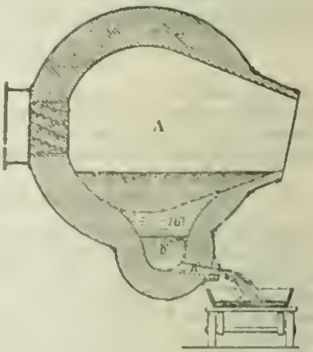
Figs. 1 and 2 illustrate the principle of the new form of Bessemer converter, or "selecteur," for smelting and reducing copper matte. The converter is spherical in outer form and suitably lined, with the twyers situated at the bottom of the vessel. It revolves on an axis inclined from the horizontal, and possesses a side pocket for the reception of the "copper bottoms." The original form is shown in Fig. 1, which can be used when the matte charge is consistent in composition, so that the pocket can be made the correct size. The matte is run in and blown to scorify the iron, which, in the form of slag, is run out at the mouth in the ordinary manner. The converter is then placed upright and blown again for the production of sufficient copper or "bottoms" to carry with it the gold, arsenic, and other impurities, when it is turned in the opposite direction with the pocket underneath. The pocket, which has been previously heated by burning coke with the orifices *g* and *h* open and the channel *f* temporarily closed, now receives the bottoms, which can be tapped therefrom when the vessel is again upright, and whilst the remainder of the matte is being converted into metal. The final copper is poured out of the mouth as usual.

Fig. 1.



When the metal charges are irregular, and the pocket is either too small or too large to exactly fill with the varying quantity of bottoms which it is necessary to produce, the converter takes the form shown in Fig. 2, wherein the bottoms are being tapped from underneath the remaining matte. The slag and final copper are run out at the mouth by tipping in the opposite direction. A saving of time is effected by the twyers in either case entering at the bottom of the charge. They are so arranged that, through holes *w* in the blast box, a rod can be passed up to clear them, and they may be either flush with the converter lining or slightly raised. The advantages put forward are, the economy, the saving of time, the greater purity of the copper for the refinery, and the more nearly perfect recovery of the gold.—A. W.

Fig. 2.



*Silicon and Chromium, Condition of, in Iron and Steel.* Carnot and Goutal. Oesterr. Zeits. Berg u. Hüttenw. 1898, **46**, 592. Through Chem. Zeit. Rep. 1898, **22**, [31], 261—262.

On treating silicon iron containing but little manganese for about 15 days with cold 5 per cent. sulphuric acid, a cohesive residue is left consisting of carbon, silicon hydroxide, and different combinations of iron and silicon. The silicon hydroxide and iron silicide (FeSi) can be removed by means of a solution of potassium hydroxide, and the hydroxides of manganese and iron by means of sulphuric acid. A residue is left, which in the author's experiments contained from 19·75 to 19·86 per cent. of silicon, and 79·63 to 79·84 per cent. of iron, figures corresponding to the formula $SiFe_2$, a compound which Moissan has prepared in the electric furnace. It differs from the mono-metallic silicide FeSi in being very resistant to the action of acids. The latter compound, which is formed in blast furnaces, is readily attacked by warm dilute acids.

From products rich in manganese and iron, a double silicide of manganese and iron can be isolated, with a composition corresponding to the formula $SiM_3$, in which $M$ represents the sum of the iron and manganese. From powdered ferro-manganese products, the author was able to withdraw about 3 per cent. of particles with a magnet. These were relatively poor in manganese, but contained much more iron and silicon than the original substance. Similar experiments were made in the case of chromium, with chromium steel and chrome ore, the latter containing 57—59 per cent. of chromium and 9·1 to 9·9 per cent. of carbon. By the long-continued action of hydrochloric acid, a residue was obtained with the composition $Fe_3Cr_9C_7$, which can also be written $CFe_3.3C_2Cr_3$. The substance $CFe_3$ has the same composition as *cementite*, and $C_2Cr_3$ is the formula of the chromium carbide obtained by Moissan. The author considers it probable that this residue is a compound of these two carbides. Different specimens of chromium steel, containing but little chromium, behaved differently in the solvent according to the amount of carbon they contained, but the residue invariably had a composition in agreement with the formula $Fe_9Cr_3C_5$, or $3CFe_3.C_2Cr_3$. A manganese iron containing a little chromium, left on treatment with a solution of copper potassium chloride, a residue with the same composition. From these results the author infers that all chromium in iron is present in the form of carbide, chrome iron containing 3 mols. of chromium carbide with 1 of iron carbide, and chromium steel 1 mol. of chromium carbide with 3 of iron carbide.—C. A. M.

### *Auriferous Quartz Veinstone in Victoria, The Reduction and Ore Dressing of.* H. Rosales. Trans. Australasian Inst. of Mining Engineers, **5**, 81—91.

The tailings from a number of reduction plants in Victoria have been collected, washed into sands and slimes, and assayed. The results show the great extent to which the gold carried away in the tailings is lost in the slimes portion as compared with that in the sands portion. Consequently the slime production should be minimised. In dealing with the pulp after it has left the amalgamating plates, great attention should be paid to its separation into sands (coarse and fine) and slimes, and the subsequent treatment of these different materials separately. In an outline of the general manipulation of an ore, the author summarises the treatment of the tailings after separation into the mixed sands and slimes as follows:—*Coarse and fine sand.*—(*a*.) Volumetric sizing, two grades, by a trommel; (*b*) two vanners; (*c*) tailings from vanners to be treated by the percolation system in the cyanide works. *Slimes.*—(*a*) Classification in a series of inverted pyramidal boxes (Spitzkasten); (*b*) Buddles and Lührig vanners; (*c*) tailings from these to be treated by the agitation method in the cyanide works; or (*d*) the slimes may be treated by the agitation process after gravitating direct from the separator to the cyanide works.—A. W.

### *Screening of Crushed Materials* [*Ores, &c.*], *Graphic Records of the.* C. de Kalb. Eng. and Mining J. 1898, **66**, 486.

Sizing curves have been plotted out by the author showing the quantities by weight of the different ores which, after crushing in 100-lb. lots and screening, remained on a series of hand sieves of diminishing size of mesh. The curves present practically the same characteristics for different ores under similar conditions of crushing, and they only vary by altering these conditions. Rock crushing may take place in three ways:—(1.) By forces acting in opposite directions along a diameter of the particle, which is exemplified in the stamp-mill; (2) by forces acting along radii making an angle with each other, or radial crushing, of which reciprocating jaw-crushers, gyratory crushers, and rolls are types; and (3) by forces acting in opposite directions along parallel lines respectively tangent to the ore particle at the two extremities of a diameter, which is represented by disc grinders, cone grinders, &c. Any or all of these principles may be employed in one mill, and it is hoped that it will be possible to test the effective action of crushers by comparing their sizing curves with those of known origin, when these data have been obtained. It is proposed to investigate the action on homogeneous materials, with definite angles and known power.

The author reiterates the wish that there should be some uniformity of scale, as to the size of the mesh and thickness of the wire, amongst manufacturers of screen cloth.
—A. W.

### *Metallic Oxides, New Method of Producing High Temperatures and Reducing Refractory.* H. Goldschmidt. Stahl und Eisen, 1898, **18**, 408.

The heat formed by reducing metallic oxides by aluminium is comparable with that of an arc furnace, and, under control, may be utilised for welding, brazing, or rivet-heating. The metal to be heated is covered with a mixture of powdered aluminium, ferric oxide, and sand, the reaction being started by a mixture of aluminium and barium or sodium peroxide, which is itself fired by magnesium ribbon. In these cases the aluminium need not be pure. Iron tubes may thus be brazed together with the consumption of 2·5 oz. of aluminium per joint.

For producing metals, pure aluminium is necessary. Chromium, in masses of 50 lb. and free from carbon, has been produced from a mixture of chrome oxide and aluminium in a magnesia-lined crucible, starting with a small quantity and gradually filling the crucible. The heat energy developed is considered equal to about 2,000 h.p. in a few minutes. The slag of alumina which is produced is similar to crystalline corundum, and may be used like emery as a grinding material. Other refractory metals may also be obtained in this manner.—A. W.

### *Dry and Wet Crushing* [*Gold Ores*]. F. Merricks. Proc. Inst. Mining and Metall., Nov. 1898.

At Waihi, New Zealand, the ore was at first wet-crushed, and 35 per cent. only of its value obtained with amalgamating plates. Later on, with pan amalgamation and both dry and wet crushing, the extraction was increased to 60 and 70 per cent., respectively, of the gold, and 25 and 45 per cent. of the silver; whilst in 1894 this process was replaced by cyanide treatment and dry crushing, with an average extraction of 90 per cent. of the gold and 50 per cent. of the silver.

The low results at first, were due to the fine state of division of the gold, and the fineness of the bullion was only 640. The author attributes the advantages of dry over wet crushing to its preventing the loss of floating sulphide of silver and fine gold. With wet crushing there is an increased output, which more than counterbalances the lower extraction, and favours its adoption. Furthermore, drying the ore for dry crushing has a tendency to melt the bullion into small pellets, which are not dissolved by cyanide.

In the treatment of West Australian telluride ore, the author suggests first hand-picking the rich telluride for smelting, crushing the remainder to a small size, stamping wet with the production of a minimum of slimes, concentrating the remaining tellurides for smelting, treating the tailings with cyanide, and collecting the slimes and extracting them in filter presses.—A. W.

### *Dry and Wet Crushing* [*Gold Ores*], *with Cyanide Treatment, in New Zealand.* J. McConnell. Proc. Inst. Mining and Metall., Nov. 1898.

This paper also deals with the ore treatment at Waihi (see preceding abstract), and three objections are stated as to dry crushing, *viz.*:—(1) The extra cost of drying the ore, which is 2*s.* to 2*s.* 6*d.* per ton, and in which operation, certain undesirable reactions take place; (2) the dust difficulty, in crushing and in charging the vats; and (3) the reduced output of the stamps. The dust is a serious question, as it penetrates everywhere and causes great wear and tear of machinery; moreover, it assays about twice the value of the remaining ore. In wet crushing, cyanide solution is added to the stamp box, and its consumption is greater than in the dry crushing process, although this difference should be reduced almost to *nil* after the solutions have been made up for their first extraction and the plant has become saturated. The quantity of solution used should be as little as possible—from 1·5 to 2 tons per ton of ore. Extraction by agitation is much higher than by percolation, and very much quicker. Experimental agitation in 2-ton lots with a 1·2 per cent. solution of cyanide, of an ore containing 0·3 per cent. of pyrites and some manganese and traces of copper, for periods ranging from 7 to 15 hours, extracted from 83 to 91 per cent. of the gold, and from 31 to 73 per cent. of the silver, the percentage of cyanide in the solution being reduced to 1·1 per cent.; whereas percolation with five or six days' treatment yielded only 65 per cent. of the value of the ore.

In some cases it might be found advantageous to restrict the output for a higher extraction by dry crushing, &c.; and the author thinks that each mine in a district should work out its own process before the plant is erected, instead of following one idea which might have been successful at one mine in that district, which is the present tendency in New Zealand.—A. W.

*Slimes, Process for Treating, without Filtration or Decantation.* A. James. Proc. Inst. Mining and Met. Nov. 1898.

This process has been communicated to the author by Messrs. Gilmour and Young, who have worked it out and successfully employed it in Nicaragua, where other methods have failed. The ore contains a large percentage of clay, and produces over 70 per cent. of slimes, which carry the greater proportion of the gold, and yield very little of it by amalgamation.

The crushed ore is charged into a pan in 2-ton lots, with 100 galls. of water to form a very thick pulp, six bottles of mercury are added and circulated until the globules can be seen in the pulp. The necessary quantity of cyanide is then mixed, and the whole run in for two hours, after which 10 lb. of mixed zinc and copper amalgam is added, and the pan run for four hours longer. By this means the precipitation of gold from the solution is nearly complete, only 10 grains or less per ton remaining soluble. The pulp and solution are discharged into a settler, and tho mercury retorted and dealt with in the ordinary way. The slimes show an extraction of 90 per cent., but the sands, in which the gold takes longer to dissolve, yield only 75 per cent., unless re-treated. The average extraction of the whole, without re-treatment of the sands, is 84 per cent. of the gold and 64 per cent. of the silver.

Copper sulphate and iron turnings are used in making the copper amalgam, and mercury is poured into molten zinc for the zinc amalgam, which is then kept under water to prevent rapid decomposition. The cost for chemicals and mercury lost is given as about 3*s.* per ton, labour, 3*d.*, and consumption of cyanide, ½ lb. per ton, with a small plant, whilst the process, if practicable, avoids the necessity of filter presses, electric precipitation, and the handling of gold slimes.—A. W.

*Slime Treatment [Gold], Discrepancies in.* J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 127—130

This is a discussion (on a previous paper by W. A. Caldecott; this Journal, 1898, 1052) as to whether by assaying slimes it is possible to obtain a result closely approximating to their value. It was shown that concordant results could hardly be obtained if the slimes, still wet with a solution containing dissolved gold, were dried in a pan and then sampled and assayed. The slimes should be washed, after a sample has been taken for the determination of moisture, and the solution and the washed residue should then be assayed separately. To obtain good results, the assays should not be made on smaller quantities than eight assay tons. The evaporation of the solution should be conducted with great care, first in porcelain dishes, and then in leaden capsules, which can be melted up with the residue. In the crucible-reduction of the gold from the slimes themselves, the weight of lead reduced must be large, or else gold will be lost in suspension in the slag. This gold may, however, be recovered by cleaning the slag by throwing into the crucible at the end of the fusion a mixture of soda, litharge, and charcoal, or tartar. No appreciable loss of gold occurs in scorification and cupellation, even with the minute quantities present in the lead buttons from slimes-assays; and there is no reason why the results of assays of slimes should not be as accurate as those of other gold-bearing products.—W. G. M.

*Slimes [Gold], Coagulation and Rapid Settlement of.* A. Prister. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 131—133.

In replying to the discussion on a previous paper of his (this Journal, 1898, 1051), the author quotes from a paper by T. Thoulet, in the *Annales des Mines*, 1891, page 1 (*cf.* this Journal, 1891, **10**, 730), on the falling-rapidity of pure kaolin in water and acids. The rapidity was determined in glass tubes 25 cm. long and 25 mm. in diameter, kept at constant temperature (56·5°) in a d'Arsonval stove. It was found that for 80 hours the falling-rapidity of kaolin in water is uniform, thus confirming Rittinger's formula. A rise of temperature causes an increase in the rapidity of fall at the rate of about 0·081 metre per degree per 80 hours. At 23° the clay used remained indefinitely suspended in pure water. The greater the quantity of sediment, the slower is the descent of the upper layer (of kaolin). But, within certain limits, the augmentation of sediment is without influence on the rate of falling of the upper layers, which becomes almost constant. The rapidity of falling from the upper to the lowest layers seems to decrease in arithmetical progression; and the rapidity is given, not by the highest, but by the lowest layer. The addition of HCl, under the conditions of the experiment, accelerated the settlement proportionately. Very little of the acid was sufficient to give a marked effect, and seemed to produce a tendency to agglomeration and to the formation of flocculent bodies. Dissolved air was without effect on the rapidity, except that, by adhesion, it retarded the fall of the smaller particles, and tended to produce "classification." When solids are immersed in a saline solution, a little salt is deposited on the surface, and there is, in consequence, a greater tendency to precipitation—as, for example, when clay, suspended in river water, comes in contact with sea water. For a like reason, the addition of dissolved lime facilitates the deposition of slimes. Variations in atmospheric pressure up to 15 atmospheres are without effect. Infusoria of 0·12 mm. diameter were found to deposit in sea water at tho rate of 0·7 cm. per second, and quartz grains of 0·5 mm. diameter at 7 cm. per second.

—W. G. M.

*Copper-Glucinum Alloys by the Electric Furnace.* P. Lebeau. L'Éclairage Electrique, 1898, **14**, 121—122.

When oxide of glucinum is heated with charcoal in an electric furnace, the carbide is produced and not the metal. However, when a mixture of the oxides of copper and glucinum, obtained by dissolving, evaporating, and igniting the mixed nitrates, is heated similarly with charcoal, an alloy of the two metals, free from carbide, is obtained. The alloy has a rose-red fracture, but is not homogeneous, and on heating yields a product containing 5 to 10 per cent. of metallic glucinum, which varies from a yellowish to a whitish colour. The 5 per cent. alloy does not tarnish in air, and only slightly in sulphuretted hydrogen. It is malleable both hot and cold, easily worked and polished, and readily dissolved by nitric acid. When the glucinum in the alloy is as low as or lower than 1·3 per cent., the metal becomes sonorous, and of a deep golden colour.

—A. W.

*Lead Bullion, Refining of Base, containing Silver and Gold.* G. H. Blakemore. Trans. Australasian Inst. of Mining Engineers, **5**, 221—258.

This paper is a general account of the purification of base lead through the following operations: The removal of the base impurities by oxidation and skimming, the extraction of gold and silver from the purified or softened lead, preferably together, by means of zinc, the elimination of the zinc from the desilverised lead by steam, the liquation of the zinc crusts containing the gold and silver to remove the lead and obtain the "dry" crust, the retorting of the dry crust to distil off the zinc and obtain retort bullion, the concentration of this bullion in water-jacket cupels up to 50 or 60 per cent., the refining to Doré bullion and the parting of the latter by sulphuric acid. It contains considerable detail of the practical points of these separate operations.—A. W.

*Lead and Zinc Sulphide Ores, The Ashcroft Process for the Treatment of Mixed.* W. R. Ingalls. Eng. and Mining J. 1898, **66**, 488—490.

This paper is an account, estimate, and criticism of the "Ashcroft" process (this Journal, 1894, 957; and 1896, 658), and its practical failure in Australia.

The process consisted of drying and crushing the ore very fine to assist in the complete roasting of the zinc to sulphate, re-grinding after roasting to facilitate leaching, dissolving in water and sulphuric acid, heating the leaching liquor to 80° C., and filtering through filter presses. The zinc was first recovered from the solution so obtained by electrolysis. But there were many difficulties in the way, the chief of which were the incomplete deposition of the zinc, and the accumulation of manganese, which prevented the working of the liquor over and over again. Consequently only 50 to 75 per cent. of the zinc could be obtained by this means, and the remainder, and the sulphuric acid of the sulphate, had to be otherwise recovered from the solution, since no economic method could be found to eliminate the manganese. Further difficulties were said to be the proper acidity, iron taken into solution, &c. Of course any lead, copper, silver, &c. was previously removed from the solution by the addition of zinc, after which, for some unexplained reason, the solution had to be subjected to oxidation, previous to electrolysis.

The present author cannot define the precise cause of failure, but it was probably due to a combination of circumstances. It would appear that extreme fineness of the ore was necessary for roasting, but the heat had to be carefully controlled to prevent sintering, and consequent re-crushing. Furthermore, this same fineness was probably the cause of some of the difficulties met with in later operations. It would appear, however, that the roasting entirely failed to produce any satisfactory quantity of soluble zinc sulphate, and of the total zinc present, 87·8 per cent. was extracted by water and sulphuric acid, 11·2 per cent. was insoluble, indicating unroasted blende, and 1 per cent. was lost in fume and dust. The formation of gelatinous silica by the action of the sulphuric acid created a great difficulty in handling and filtering the solution, and the leaching and washing were not very successful, only 70·24 per cent. of the total zinc in the ore being washed out through the filter press, although 87·8 per cent. was in solution.

In the original process it was proposed to leach the roasted ore with ferric chloride to form ferric hydrate and zinc chloride, the latter to be electrolysed so as to yield metallic zinc and ferrous chloride, this salt in turn acting on more zinc chloride to produce zinc and re-form ferric chloride; but ultimately this method was abandoned. The author thinks that the adverse experience at Cockle Creek has much metallurgical value, although it has been obtained at enormous cost. One conclusion drawn is that no progress has been made in the electrolysis of zinc sulphate solution, and this operation cannot be economical where coal has to be the source of power. With the present known improvements and devices, it is considered that it might, however, be worked with cheap water power.
—A. W.

*Mercury, A New Assay for.* R. E. Chism. Eng. and Mining J. 1898, **66**, 486.

*See under* XXIII., *page* 1183.

*Zinc, Electrolytic Estimation of.* H. Paweck. Zeits. f. Elektrochem. 1898, **5**, [18], 221.

*See under* XXIII., *page* 1186.

*Ferro-Tungsten, Analysis of.* A. G. McKenna. Eng. and Mining J. 1898, **66**, [21], 607.

*See under* XXIII., *page* 1185.

*Cyanide Solutions, Estimation of Oxygen in.—II.* A. F. Crosse. J. Chem. and Metall. Soc. of S. Africa, 1898, **1**, 125.

*See under* XXIII., *page* 1187.

*Iron and Steel, Rapid Methods of Determining the Principal Elements in.* M. L. Campredon. Monit. Scient. 1898, **12**, [4], 778.

*See under* XXIII., *page* 1184.

## PATENTS.

*Ores, An Improved Process relating to the Reduction of, and Impts. in Furnaces therefor.* J. E. Dowson, London. Eng. Pat. 27,015, Nov. 18, 1897.

Water gas or other combustible reducing gas, after being heated by passing through a channel in the roof of a long, flat-bedded furnace, is passed back again over the ore to be reduced, and is then burnt in chambers underneath the bed, with air which has been heated in passages situated below these combustion chambers and above the flues therefrom. Valves regulate the supply of air and gas, the latter of which, if desired, may be partly or entirely led directly into the combustion chambers, without passing over the ore.
—A. W.

*Metal [Al, &c.], An Improved, and Method of Producing it.* F. D. James and M. B. Fox, both of Gloucester. Eng. Pat. 27,881, Nov. 26, 1897.

The alloy is prepared by melting 100 parts of aluminium with from 5 to 25 parts of a primary homogeneous phosphoric alloy, previously prepared, which may contain from 50 to 90 parts of copper, 2 to 20 of nickel, 5 to 25 of zinc, and 2 to 10 parts of phosphorus.

A claim is made for the method of adding phosphorus to molten metals or alloys, by sheathing it in a case of the same or an analogous alloy, and holding it under the surface of the metal, so that the walls of the sheathing melt and become thin and porous, thus enabling the whole to be gradually absorbed in the bath.—A. W.

*Ores, Impts. in Furnaces for the Treatment of.* A. G. Wells, London. Eng. Pat. 28,894, Dec. 7, 1897.

The furnace consists of a long closed chamber, with the bed built upon and heated by a zig-zag flue leading from the fire near one end to the chimney near the other. At each end of the chamber is a hood, dipping into water tanks, so as to prevent access of air to it when the ore, which traverses the bed from end to end, is being reduced and discharged into the water. The ore is automatically fed in through hoppers at the cool end, and moved toward the exit at the fire end, on cross-bars laid upon endless chains, which also dip into the water and get cooled, previous to travelling back under the furnace-bed for re-entry. Near the exit is provided a means for the supply of hot reducing-gases. The air for the fire is also heated under the furnace-bed in longitudinal ducts, and the claims are set forth for all these things in combination.—A. W.

*Emery Wheels, Impts. in the Manufacture of a Substitute for.* [*Steel.*] C. M. Pielsticker, Wembley. Eng. Pat. 284, Jan. 5, 1898.

As a substitute for emery, New Zealand ironsand, after the removal of silica, is mixed and heated in a crucible with carbon to produce fine particles of steel, the whole being then quenched in cold oil, or oil and water, to increase the hardness. This quality may be further increased by adding a compound of phosphorus to the mixture in the crucible. The hard, fine particles, containing 5 per cent. or more of carbon and some phosphorus, are mixed with a binding material and made into wheels in the usual manner.—A. W.

*Titanium, Impts. in Alloys of, and Processes for Producing same.* A. J. Rossi, J. MacNaughton, and W. D. Edmonds, all of New York, U.S.A. Eng. Pat. 18,127, Aug. 23, 1898.

This patent is for the production of alloys of titanium and iron containing above 5 per cent. of the former and not less than 10 per cent. of the latter, together with some carbon, by supporting in a bath of iron at an intense heat, a mixture of carbon and titanic acid, titaniferous iron ore, or other suitable compound, or a rich slag of titanium. The charge is heated in a crucible, packed in coke, in a small electric furnace. In one charge, 50 to 100 lb. of an alloy may be obtained containing about 43·5 per cent. of titanium and 19 to 50 per cent. of iron.—A. W.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### (*A.*)—ELECTRO-CHEMISTRY.

*Dithiondisulphide, Electrolytic Preparation of a New.* C. Schall and S. Kraszler. Zeits. f. Elektrochem. **5**, 1898, [19], 225—226.

By electrolysing an aqueous solution of potassium ethylthio-carbonate, the authors have obtained a new dithiondisulphide $(C_2H_5SC)_2S_2$, as a yellow, easily decomposed oil.
—J. S.

*Accumulators, Sulphating of Negatives.* L. Juman. Ecl. Electr. 1898, **16**, 133—136.

On open circuit this is chiefly due to two causes:—(1) the chemical action of the sulphuric acid upon spongy lead; (2) the electrochemical action of the couple formed by the active material (spongy lead), and the plate which supports it (lead often containing antimony). The sulphating is increased by the use of acid of high density, and also by the presence of copper (from the connections), and lead peroxide (from the positives). Soft lead free from antimony should be used for the plates; if this cannot be done, an electrolyte of low density should be used. The antimony present is found almost exclusively in the superficial layers of the plates, and spoilt negatives may often be improved by removing these superficial layers.
—D. E. J.

### PATENTS.

*Electrical Heating Apparatus and Resistances; Improved Compositions [Nickel Powder and Clay] for Use in the Construction of, and the Processes for their Manufacture.* L. Parvillée, Paris. Eng. Pat. 25,388, Nov. 2, 1897.

For electrical heating apparatus and the manufacture of resistances in general, the following formula is given:—

| | Parts. |
|---|---|
| Nickel, in the form of powder | 60 |
| Plastic white clay, washed | 40 |

"These materials, after being intimately mixed and dried till they contain not more than 6 per cent. of water, are compressed in suitable moulds with a pressure of not less than 3,000 kilos. per sq. cm., and the moulded articles are then baked with charcoal in a suitable closed receptacle, at a temperature a little below the melting-point of nickel." "Those parts of the articles to which the contact devices are connected are reinforced by the application to them at the time of compression of the following special composition (in place of which copper or other metal may be electrically deposited):

| | Parts. |
|---|---|
| Nickel, in the form of powder | 90 |
| Plastic white clay, washed | 10 |

—J. C. R.

*Accumulators or Secondary Batteries [Forming, &c.], Impts. in the Construction of.* H. Leitner, London. Eng. Pat. 27,725, Nov. 25, 1897.

The peroxide employed is prepared preferably according to a prior Eng. Pat. 780 of 1896 (this Journal, 1896, 361). "The essential feature of the invention consists in the utilisation . . . . of the pressure produced by the expansion of the peroxide plates reacting against highly resilient material, so that the peroxide itself is formed under great pressure depending upon the amount of expansion. The result of this pressure is, that the peroxide is rendered very dense and of great strength, and is also kept tightly against the conducting parts under all conditions."

Claim I. is: "Forming the peroxide plates under great pressure, produced by the expansion of the plates during the conversion into peroxide, reacting against highly resilient material," celluloid, vulcanite, wood fibre, or other similar material, "the whole process taking place in the cell itself."—J. C. R.

*Electric Batteries, Impts. in.* E. Edwards, London. From P. A. Emanuel, Aiken, South Carolina, U.S.A. Eng. Pat. 12,322, June 1, 1898.

"The objects of my invention are to produce a wet voltaic pile with a current of the electrolyte steadily flowing through it; to provide means for causing this steady flow, and to cause the current of electricity made by said pile to be reinforced by electricity, produced by a current of wet steam passing through restricted passages, and afterwards expanding." "In order to obtain this supply of electrified steam, I have invented apparatus therefor, dependent upon the same principle as Armstrong's hydro-electric machine." This apparatus is shown and fully described, in the course of twenty claims, in considerable detail.—J. C. R.

*Primary Electric Batteries [Zinc Carbon], Impts. in or relating to.* A. J. Boult, London. From V. Busson, Paris. Eng. Pat. 17,588, Aug. 15, 1898.

For a battery (carbon zinc) agglomerate electrode, the following composition is given:—

Plumbago, 1 part by weight.
Dioxide of manganese, 4 parts by weight.
Sulphate of zinc, 1 part by weight.

"placed in a receptacle containing the electrolyte," ammonium chloride.—J. C. R.

### (*B.*)—ELECTRO-METALLURGY.

*Zinc, Electro-Deposition of.* S. Cowper-Coles. Electrician, 1898, **41**, 382—383.

The author gives the results of his experiments on the formation of spongy zinc in the electro-deposition of this metal. A special cell was used with a revolving aluminium cathode; upon this the zinc was deposited from a zinc-sulphate solution, various gases (*e.g.*, oxygen, air, coal-gas, carbon dioxide) being formed over the cathode during deposition. The best deposits were obtained when coal-gas and carbon dioxide were used.—D. E. J.

### PATENTS.

*Electro-Deposition of One Metal on Another, Impts. in Apparatus for the.* J. E. Bryant, Redditch. Eng. Pat. 23,267, Oct. 11, 1897.

Relates to the employment of a rotating cylinder or barrel, of wood or other material, either hermetically sealed, to contain the electrolyte, anodes, and the articles to be coated; or perforated, and placed in separate vessels which may contain the electrolyte.

The claims relate to the anode suspended to a conductor immediately over the work, or articles to be coated; moving the anode nearer to or farther from the articles to be coated, and other details.—J. C. R.

*Electro-Deposition Apparatus [Revolving Cage, &c.], Impts. in.* A. F. Harris, Walsall, Stafford. Eng. Pat. 24,884, Oct. 27, 1897.

Apparatus for electro-deposition more particularly intended for small articles, which takes the form of a revolving cage or drum in the bath. The arrangement of electrodes, mechanical parts, and driving mechanism are fully illustrated.—J. C. R.

*Electro-Metallic Depositing Apparatus, Impts. in.* J. E. Hartley and H. E. Hartley, Birmingham. Eng. Pat. 27,707, Nov. 25, 1897.

The apparatus is principally intended for use where only a small number of articles are required to be plated at one operation, and consists of a basket or cage suspended vertically in the bath by means of cords, and carrying an anode which rotates with it. The bottom of the basket has a cam surface, which oscillates the work when it is rotated horizontally over a controlling roller fixed in the side of the bath. The suspension rod which carries the cathode connections is surrounded by a revoluble non-conducting sleeve, carrying a conducting collar, which is electrically connected to the anode, through a loose collar encircling the sleeve. The anode may be suspended centrally and vertically, instead of horizontally, in which case the electrical connections to the collars and sleeve are suitably altered.
—G. H. R.

*Iron, Steel, and their Alloys with Chromium, Tungsten, Nickel, and Manganese, and the like; Impts. in and connected with the Electro-Metallurgic Production of.* E. Stassano, Rome, Italy. Eng. Pat. 11,604, May 23, 1898.

This process for the reduction of oxide of iron and the metals to be combined therewith by the heat of the electric arc is carried out in a furnace having the form of a vertical double cone, the larger bases of which abut against each other. The smaller cone abuts against another slightly conical chamber which forms the pot, and at their junction are inserted two carbon electrodes whose distance apart can be regulated. The metallic fluid can be drawn off from the pot, and the top of the furnace is closed by a hopper through which the ore is fed, and its lower end is closed by a lever valve, which prevents the ingress of air. The furnace gases are carried off by two tubes closed with hydraulic valves. The ore is carefully selected by hand, pulverised, and passed through a magnetic separator. It is then analysed, and the materials necessary for its complete reduction added to it in a finely pulverised state, and the mixture so formed is moistened and compressed into briquettes, which are broken up and fed into the furnace.
—G. H. R.

*Electro-Deposition of Copper and other Metals on Rotary Cathodes [Insoluble Soft Rubbers, "Impregnators"], Impts. in and connected with the.* W. E. Heys, Manchester. From E. Dumoulin, Paris. Eng. Pat. 13,861, June 22, 1898.

Eng. Pats. 16,360 of 1895 and 2709 of 1897 (this Journal, 1898, 54) describe the use of certain organic membranes for the purpose of ensuring smooth, uniform, and homogeneous deposits. According to the specification, the advantages are obtained by the use of such rubbers or "impregnators," of a rapid and sound deposit, &c.; but, owing to the high density of the current employed, a rapid heating of the electrolytic bath ensues, which may suffice to bring about the softening or solution of some of the albuminous or gelatinous constituents of the "impregnators," which soften at about 30° C., and even begin to disintegrate and dissolve in the bath. To obviate this defect, the "impregnators" are thoroughly impregnated with a solution of formic aldehyde ("within the limits of 10 per cent. and 40 per cent."), after which they are copiously washed in water to remove the formic aldehyde, or this substance is neutralised or decomposed, as, for example, by means of acetic acid. Impregnators so treated may be employed "with equal facility in a hot or in a cold bath."—J. C. R.

*Metals from their Halogen Combinations, Electrolytic Process for Extracting.* Dr. E. Hilberg, Berlin, Germany. Eng. Pat. 16,569, July 30, 1898.

This process for the extraction of metals from their halogen combinations by electrolysis consists in carrying out the operation in a bath which is covered by a layer of refractory non-conducting material, such as asbestos. A large number of electrodes are employed, and the decomposition is effected with the simultaneous production of a vacuum above the molten material, or with the introduction above the same of an inert gas. The melting and the decomposition may be effected in the same or in separate vessels.—G. H. R.

## XII.—FATS, OILS, AND SOAP.

*Fatty Seeds during Germination, The Changes in the Composition of.* L. Maquenne. Comptes Rend. 1898, **127**, [17], 625—628.

That the glycerin of the oil in the seed is converted into sugar during germination has long been known, and the author, in the present paper, gives the results of his investigations on the behaviour of the fatty acids. The oil-seeds selected were *Arachis* (earth-nut), containing the saturated fatty acid, arachidic acid, $C_{20}H_{40}O_2$, and *Ricinus* (castor-oil seed), containing the unsaturated, ricinoleic acid, $C_{18}H_{34}O_3$. The seeds were grown in sand in an incubator at a temperature of 20°—25° C., and complete analyses were made at different periods during germination. The determinations included oil, saccharifiable carbohydrates, insoluble carbohydrates, as cellulose, nitrogenous matter, ash, and total weight, and were calculated on the dry substance. Starting with 51·4 per cent. of oil in both cases, *Arachis* still contained 12·16 per cent. after 28 days, whilst *Ricinus* only contained 3·08 per cent. after 18 days. The maximum percentage of total carbohydrates was attained in *Arachis* after 18 days and in *Ricinus* after 10 days, the increase from the start being 5·6 and 16 per cent. respectively. The following selection from the analyses shows the proportions of saccharifiable and insoluble carbohydrates and of oil at the start and at the periods of maximum :—

| Constituent. | Arachis (Earth-nut). | | Ricinus Castor-oil Seed. | |
|---|---|---|---|---|
| | Original Seed. | After 18 Days. | Original Seed. | After 10 Days. |
| Oil .................... | 51·39 | 21·45 | 51·40 | 5·74 |
| Carbohydrates dissolved by boiling in dilute acid. | 11·55 | 12·34 | 3·46 | 24·14 |
| Insoluble carbohydrates and cellulose. | 2·51 | 7·29 | 16·74 | 11·98 |
| Total carbohydrates | 14·06 | 19·63 | 20·20 | 36·12 |

In the case of earth-nut there was an initial decrease in the soluble carbohydrates with a subsequent increase, whilst in the case of *Ricinus* there was an initial decrease in the insoluble carbohydrates, also with a subsequent increase. The nitrogen remained nearly constant throughout the whole period of germination.

The amount of total carbohydrates derivable from the glycerin alone in the oil of these seeds is about 5 per cent.; hence the author concludes, with reservations, that the unsaturated ricinoleic acid is capable of being utilised for carbohydrate formation, whilst the saturated arachidic acid is not so suitable for this purpose. Probably this relation may be extended to acids of the oleic type and the saturated type generally, the latter taking part in respiratory functions alone, whilst only the two extremities of the chain in the former acids are consumed in respiration, leaving the allyl C : C grouping in the middle to be utilised for the formation first of glycerin and subsequently of carbohydrates. If this be so, one molecular proportion of tri-olein would be capable of furnishing 40 per cent. of glucose, equivalent to an increase of 22 per cent. in the weight.—J. F. B.

*Wheat Oil.* G. de Negri. Chem. Zeit. 1898, **22**, [92], 976.

By the usual methods of grinding wheat, 1 per cent. of germs is obtained. 1 kilo. of the latter actually contains 125 grms. of fatty matter and of this 80 grms. can be obtained by extracting with petroleum spirit.

After distilling off the solvent in a partial vacuum, a clear yellow-brown mobile oil is obtained which has a peculiar smell, resembling wheat.

The oil solidifies at 15°. It is soluble in ether, petroleum ether, chloroform, and carbon bisulphide, but insoluble in cold absolute alcohol. It is soluble, however, in 30 parts of hot alcohol. Glacial acetic acid dissolves at 65° an equal volume of oil. It is only slowly saponified by alcoholic potash.

The following are some of the important constants of wheat oil :—

| | |
|---|---|
| Specific weight at 15° | 0·9245 |
| Solidification point | 15° |
| Melting point of fatty acids | 30·5° |
| Solidification point of fatty acids | 29·7° |
| Saponification value | 182·81 |
| Iodine value of oil | 115·17 |
| Iodine value of fatty acids | 123·27 |
| Refractometer value (Zeiss-Wolloy) | 74·5 |
| Free acid per cent. calculated as oleic acid | 5·65 |

Colour reactions :—Haydenreich's reaction, orange-yellow with violet spots. Brullé's reaction, red tinge becoming blood-red. Schneider's, and also Baudouin's reaction gave no colour. Becchi's as well as Millian's reaction gave a pale brown colour.

The oil easily turns rancid. After standing a year a sample contained 13·86 per cent. of free acid calculated as oleic acid. Germs of different origin were found to give oils with varying constants.—J. D. G.

*Carapa Oil from Trinidad.* W. H. Deering. Imp. Inst. J., Nov. 1898, 313.

Carapa oil (crab oil, andiroba oil) is extracted from the nuts of *Carapa Guianensis*, a tree which exists more or less commonly throughout tropical America and the West Indies. The yield of oil from the seeds is said to be 70 per cent. by weight. It is used locally, admixed with pigments or tar, for preserving timber, and it has been found to be well suited for the manufacture of a good oil-soap (like olive-oil soap). A sample examined by the author consisted of a yellow, non-drying, glyceridic oil, slightly soluble in alcohol, and with an acid but not unpleasant vegetable odour, and an intensely bitter flavour. On cooling for a day to 40° F., it solidified to a whitish solid of the consistency of tallow, at a higher temperature, it showed a tendency to the separation of solid fat. Its viscosity at 70° F. is about that of rape oil, specific gravity at 60° F., 0·9225. It is saponified readily and completely by caustic potash, requiring 2·26 per cent. of KOH to neutralise the free acid, and 19·56 per cent for complete saponification. The free acid amounted to about 12 per cent. The oil gave 93·7 per cent. of fatty acids, insoluble in water, and an appreciable trace (equivalent to 0·25 per cent. of KOH) of volatile fatty acid soluble in water; it also yielded 10·4 per cent. of crude glycerin. The fatty acids form a very firm crystalline solid, of pale buff colour, melting at 102° F. The amount of bromine absorbed by the oil dissolved in carbon bisulphide was about 41 per cent. (40·84 and 41·06 per cent.) ; this low number indicates a comparatively high percentage of saturated glycerides, and a low percentage of olein.

—A. S.

*Paradise-Nut Oil.* G. de Negri. Chem. Zeit. 1898, **22**, [90], 961.

This oil was obtained by extraction with petroleum spirit (ligroin) from the kernels of the fruit of the *Quatelé Zabucajo* (*Lecythis Zabucajo Aubl*), a tree belonging to the natural order *Myrtaces*, which occurs in the forests of Brazil and Guiana. The kernels yielded from 50 to 51 per cent. of a mobile, clear oil which was almost colourless or at most light yellow. It readily became rancid, though not so rapidly as Brazil-nut oil. Cooled down to 12° C. it began to form white lumps and at 8° C. was completely solid. It was soluble in ether, chloroform, and benzene, insoluble in the cold in anhydrous alcohol and scarcely soluble in hot water. It was insoluble in cold glacial acetic acid but dissolved in an equal volume of the hot acid. The constants given by this oil and for the purpose of comparison those of Brazil-nut oil are shown in the subjoined table.

| — | Brazil-nut Oil. | Paradise-nut Oil. |
|---|---|---|
| Specific gravity | 0·918 | 0·895 |
| Solidification point | 0° | 4° |
| Melting point of fatty acids | 28°—30° | 37·6° |
| Solidification point of fatty acids | .. | 28·5° |
| Saponification value | 193·36 | 173·63 |
| Iodine value of oil | 106·22 | 71·64 |
| " " fatty acids | 106·04 | 72·33 |
| Acid value (calculated as oleic acid) | 1·433 | 3·19 |
| Acetyl value | .. | 44·08 |
| Refractometer value (Zeiss-Wollny) at 15° C. | .. | 61·3—61·5 |

—C. A. M.

*Butter and other Fats, Relation between Specific Gravity and Insoluble Fatty Acids of.* N. Leonard. Analyst, 1898, **23**, [272], 282.

It is stated that the correspondence often noted between the specific gravity and the percentage of insoluble fatty acids of butter and margarine is due to the fact that the usual adulterants (beef and mutton fat, &c.) have approximately equal densities and yield the same proportion of insoluble fatty acids. The presence of a vegetable oil, such as cotton-seed, "cocoanut," or sesamé oil, having a higher density than animal fats, but yielding nearly the same proportion of insoluble fatty acids, is indicated by a deviation from the usual relation.

Some years ago the author found the relation might be represented by the formula $y = k\,(1 - x)$, where $y$ = percentage of insoluble fatty acids, $x$ = specific gravity at 100° F. (water at 60° F. = 1), and $k$ = a constant, the mean value of which was found to be 951 ± 1·6. More recent experiments gave $k = 951 \pm 1{\cdot}8$. For practical purposes the formula $y = 951\,(1 - x)$ is sufficient. Of 63 samples examined, the percentage of insoluble fatty acids found by experiment differed from that calculated from the formula by not more than 0·5 per cent. in 35 cases and not more than 1·0 per cent. in 54 cases, whilst in only three cases was there a difference of more than 1·4 per cent., and in each of these the amount of insoluble fatty acids was abnormally high.—A. S.

*Curcas Oil ("Physic Nut" Oil).* O. Klein. Zeits. angew. Chem. 1898, [44], 1012—1015. See this Journal, 1898, 1009.

This oil is obtained from the seeds of the *Jatropha Curcas* (the physic nut tree), which grows to a height of 3 or 4 metres, and has considerable resemblance in appearance to the castor-oil plant. It is cultivated in all the Portuguese colonies, but especially in the Cape Verde Islands, which supply about three-fourths of the total quantity shipped to Portugal. There are two factories in Lisbon where the oil is extracted, the finely-divided seeds being subjected to hot pressure. The oil is used for lubricating and lighting purposes, and in the soap and candle manufactures, and the pressed residue fetches a high price as a manure.

The physical and chemical constants of the oil extracted in different ways by the author, and of a commercial specimen, were :—

| — | Cold Pressed. | Hot Pressed. | Cold Pressed. | Extracted with Ether. | Commercial, Hot Pressed. |
|---|---|---|---|---|---|
| Specific gravity | 921·0 | 924·0 | 919·9 | 920·7 | 920·8 |
| Refractive index at 25° | 1·4686 | 1·4689 | 1·4687 | 1·4687 | 1·4681 |
| Iodine value | 109·8 | 109·1 | 110·4 | 109·1 | 107·9 |
| Saponification value | 203·5 | 203·6 | 197·5 | 197·0 | 198·1 |
| Free acid | 4·90 | 4·65 | 0·88 | 0·57 | 0·68 |
| Volatile acid | 7·10 | 5·5 | .. | .. | .. |

The oil has a yellowish-red colour, which varies according to the degree of pressure and heat to which the seeds were subjected. It has a characteristic smell, which cannot readily be mistaken. The acetyl values determined by the original method of Benedikt and Ulzer were :—

| — | Hot Pressed. | Extracted. |
|---|---|---|
| Acetyl acid value ............ | 183·8 | 172·0 |
| Acetyl saponification value .. | 218·5 | 227·7 |
| Acetyl value.................. | 34·7 | 51·7 |

A subsequent determination of this constant by Lewkowitsch's method (this Journal 1897, 503—506) gave 17·6 and 25·3 as the respective values for the two oils—a difference which, in the author's opinion, can only be accounted for by the formation of oxyacids in the preparation of the free acids.

The melting points of the fatty acids determined in a capillary tube were for No. 1 and No. 2, 29·5° and 30·5°, and the solidifying points were 25·75° and 26·5° C. The oil dissolved in alcohol to the extent of 24·12 per cent. It was not soluble in cold glacial acetic acid, but dissolved readily in the hot acid. The viscosity determined in Engler's apparatus at 20° C. was 9·45, so that in this respect curcas oil lies between crude rape-seed oil and olive oil, which, according to Künkler, has a viscosity of from 9·03 to 10·3. The phytosterol was determined by Bömer's method, the mean of three results being 0·58 per cent. (Zeits f. Untersuch. Nahr. u. Genussmittel, 1898, I. [i.], 21—49).

According to Hiepe (Rep. de anal. Chem. **5**, 326), curcas oil is frequently used to adulterate olive oil in Portugal, but the author states that he has never yet met with such an adulteration, although he has examined hundreds of samples of suspicious olive oil. He also considers such an admixture extremely improbable, taking into account the fact that 10 drops of curcas oil have as strong a purgative effect as a table-spoonful of castor oil, and that an addition of 10 per cent. of curcas oil to olive oil gives to the latter its marked odour. For its detection in olive oil, Hiepe recommends treating the oil with nitric acid and copper filings, which produces an elaidin mass of a reddish-brown colour. The author confirms this, but shows that the reaction is not characteristic, since earth-nut oil and cotton-seed oil give similar colorations. Other colour reactions are also inconclusive. An ethereal solution of silver nitrate is faintly reduced by the commercial oil, but the fatty acids cause no reduction. In the author's opinion, the best tests for curcas oil in olive oil are the characteristic odour, and the increase in the specific gravity, refractive index, and the iodine value.

The oil was found to contain 9·59 per cent. of solid fatty acids melting at 57·5° and with a molecular weight of 269·5, from which data they were judged to consist of 80 per cent. of palmitic acid and 20 per cent. of stearic acid. No myristic acid could be detected. The liquid fatty acids (80 per cent.) separated as lead salts with ether, yielded on oxidation, by Hazura's method, equal quantities of sativic and dihydroxy-stearic acid, from which it was concluded that they consisted of equal parts of oleic and linolic acids. No ricinoleic or linolenic acid was found.—C. A. M.

*Oils, Lubricating, and more especially Oleo-Naphthas; Contributions to the Study of.* A. Chenevier. Monit. Scient. **12**, [4], 1898, 785.

*See under* XXIII., *page* 1181.

### PATENTS.

*Soap for Toilet and Laundry Purposes, Impts. in the Manufacture and Composition of.* E. G. Hughes, Manchester. Eng. Pat. 4153, Feb. 19, 1898.

Claim is made for incorporating with the soap of the oxalate of some base, such as soda, potash, or ammonia, preferably the first. Suitable proportions for use with hard water are 5 parts of the oxalate to 95 parts of soap.
—C. A. M.

*Liquid Fats and Oils, Heated; Impts. in Apparatus for Treating and Cooling.* C. G. Hepburn, Sydney. Eng. Pat. 18,730, Sept. 1, 1898.

The object of the apparatus claimed is to obtain thorough aëration of the oil whilst in a fine state of division. It consists essentially of a sprayer which distributes the oil on to surrounding vertical louvres, through which is forced a current of air. In place of the sprayer a rotating filter may be used as a distributor and "air-whirler," sucking the air in through the louvres, which may be either stationary or may move with the spraying and aërating apparatus. Various modifications of these devices are claimed.
—C. A. M.

## XIII.—PIGMENTS, PAINTS; RESINS, VARNISHES; INDIA-RUBBER, Etc.

### (A.)—PIGMENTS, PAINTS.

*White Paint or Varnish, A New or Improved Non-Poisonous and Permanent.* L. Hunt, Manchester. Eng. Pat. 22,792, Oct. 5, 1897.

The base consists of about 75 parts of barium sulphate and 25 parts of "sulphite of zinc" intimately mixed together by precipitation. It is ground into linseed oil, and diluted either with oil, turpentine, and "terebine" to make a white "paint," or with pale copal varnish to make a white "varnish."—F. H. L.

### PATENTS.

*Paints, Anti-Corrosive Compositions, and Compositions for like Uses, Impts. in the Manufacture of.* C. J. Grist, London. Eng. Pat. 26,800, Nov. 16, 1897.

Jute, hair, or similar fibrous material, is cleaned, dried, and loosened, then dipped into linseed oil which has previously been boiled for 24 hours with litharge or other suitable drier. The excess of oil is removed in a hydro-extractor, the fibres are passed through a carding machine, and the residual oil is allowed to harden in the air. This operation is repeated twice till the jute is thoroughly covered with dry oil, when it is ground on a roller mill and crushed cold with 5 or 10 per cent. of sulphur. 100 lb. of the product are again ground with 7 to 10 lb. of any desirable pigment, mixed with 33 to 45 lb. of "ketone" or other solvent, and finally diluted with the necessary proportion of a varnish compounded as follows:—8 to 16 lb. each of shellac, a "gum," and "resin," are dissolved in about 2 to 4 lb. of methylated spirit, adding in some cases 2 to 4 lb. of "barytes" and a like quantity of manganese borate. For anti-corrosive purposes the pigments are omitted, and 1 to 5 per cent. of an arsenical salt introduced to render the whole toxic. The material is claimed to dry quickly, to have a brilliant surface, to withstand wear and weather, not to crack or perish, and to be impervious to moisture.—F. H. L.

*Water-proof and Weather-proof Paint, Improved Manufacture of.* H. Loesner, Leipzig-Lindenau, Germany. Eng. Pat. 28,484, Dec. 2, 1897.

30 or 40 parts of varnish or drying oil are mixed with 40 parts of cement, or with 30 parts of cement and 10 parts of silica (*e.g.*, sand or quartz), and finally 20 to 30 parts of some colour (lamp-black, iron oxide, barium sulphate, &c.) are added. The cement and silica must be very finely powdered, and each ingredient free from moisture, lest the cement begin to set prematurely. When the composition is applied to any surface, the cement gradually obtains sufficient water from the atmosphere to enable it to harden, thus rendering the coating air-tight as mentioned. The material has been tested by means of the author's "steam-test" (this Journal, 1898, 359), and it has, it is stated, been found to withstand the action of the steam for 15 hours. Loesner calculates that one hour of his hot test is more severe than two or three months' exposure to rain. (See also this Journal, 1898, 608.)—F. H. L.

*Linseed Oil Body for Paint, Printing Ink, Floor Cloth Making, and the like; Improved Compound applicable for Making a Substitute for.* S. Banner, Liverpool. Eng. Pat. 29,728, Dec. 15, 1897.

In this invention, rosin, or a compound of rosin with hydrate of lime (Eng. Pat. 11,805, 1887), is dissolved in a suitable solvent, and saponified by means of caustic soda or potash, silicate of potash, or mixtures of these substances. Suitable proportions mentioned are about 18 parts of caustic potash for each 100 parts of colophony or its lime compound.—C. A. M.

## (*B.*)—RESINS, VARNISHES.

*Resins, Note on Certain.* Rowland Williams. Analyst, 1898, **23**, [271], 254.

The author has examined representative samples of kauri, copal, and dammar in a manner similar to that which he employed on a previous occasion (Chem. News, **58**, 224—225). The kauri and one of the copals were also examined after being subjected to the melting or "running" process, which is applied to certain resins previous to converting them into varnish. "The hot KOH determinations" were made according to Koettstorfer's directions, except that in some cases a very large excess of alcoholic potash was added, and the boiling continued for a much longer time than usual. The cold KOH determinations were made by digesting the finely powdered resins with a mixture of ether and alcohol, and titrating with semi-normal KOH, using phenolphthalein as indicator. The iodine absorption was determined by allowing a large excess of Hübl's reagent to act all night (16 hours). The following results were obtained:—

| — | Kauri. | | Copal. | | | Dammar. |
|---|---|---|---|---|---|---|
| | | | Fine Quality. | | Finest.* | |
| | Ordinary Condition. | After "Running." | Ordinary Condition. | After "Running." | Ordinary Condition. | Ordinary Condition. |
| | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. | Per Cent. |
| KOH absorption, hot | 6·42 | 5·16 | 18·87 | 13·78 | 12·87 | 2·85 |
| " " cold | 6·19 | 4·76 | 14·36 | 10·99 | 11·56 | 2·63 |
| Iodine absorption | 130·62 | 126·52 | 124·32 | 129·60 | 95·40 | 99·40 |
| Loss or gain on heating at 100° C. for 15 hours. | 11·81 loss | 0·80 gain | 0·82 loss | 2·26 gain | 0·74 loss | 0·84 loss |
| Mineral matters | 0·15 | 0·14 | 0·14 | 0·16 | 0·03 | 0·05 |

* An exceptionally fine quality of copal, used only in the manufacture of the palest and highest class of varnishes.

—A. S.

## PATENTS.

*Oils, Solid Elastic, Suitable for the Manufacture of Linoleum, Floor Cloth and similar Articles; Impts. in the Manufacture of.* J. E. Bedford and C. S. Bedford, both of Leeds. Eng. Pat. 29,656, Dec. 15, 1897.

The inventors claim the use of oxidation products prepared from rosin oil or mixtures of rosin oil and linseed oil in the following ways:—(1.) The rosin oil is oxidised alone (with suitable driers) in an apparatus like that described in Eng. Pat. 7742, 1893 (this Journal, 1893, 406) until it becomes a viscous mass, and is capable of being used as a substitute for the ordinary colophony and kauri gum usually incorporated with the solid oxidised linseed oil. (2.) The mixture of rosin oil and linseed oil, in the required proportions, is oxidised in the presence of driers in the apparatus until it attains a spongy consistency, when it is transferred to an open pan heated by steam. Here it is further oxidised by the increased heat, assisted by a mechanical agitator, and when it becomes sufficiently elastic and has lost its greasiness, it is discharged on to an iron or concrete floor and left to cool.—C. A. M.

*Distemper, Permanent Washable.* F. C. Goddard, London. Eng. Pat. 4884, Feb. 28, 1898.

Two lb. of white or coloured pulp (whiting softened in water or a pigment ground therein) are warmed and thoroughly mixed with ¼ lb. of beeswax, previously dissolved in ¼ pint of turpentine. When required for use a little water-size is added, and after straining, the whole is ready. The beeswax is said to give the distemper a dull surface, and to fix it on the wall so that it can be washed without injury.—F. H. L.

## (*C.*)—INDIA-RUBBER, &c.

*India Rubber Milk, On.* A. Girard. Bull. Soc. d'Encouragement, **3**, [7], July 1893, 858—866.

The milky juice obtained from any of the many india-rubber producing plants, presents in every case one similar aspect. It has very much the appearance of animal milk, though being of a duller white, and always characterised by a certain viscosity, which is frequently very considerable. Under the microscope rubber juice presents exactly the same aspect as ordinary milk, the rubber being distributed in the shape of spherical globules throughout an aqueous serum. The coagulation of the juice is determined by the fusion of these globules into larger masses. The processes employed to induce coagulation, differ very greatly in different districts, and *a priori* show no connection with any common scientific principle. Nevertheless, they can be shown to be determined by the physical properties and the chemical composition of the various juices. The following samples of latex were examined.—(1.) The milk of *Hancornia speciosa*, from Pernambuco (Brazil). (2.) The milk of some creeper from Central Africa. (3.) The milk of one of the rubber trees of Para, *Siphonia elastica*, or *Hevea Guianensis*. (4.) A milk of unknown origin, probably from Central Africa. (5.) The milk from *Castilloa elastica*, Nicaragua. (6.) The milk of *Siphonia elastica*, Para. (7.) Several samples of milk, obtained from various specimens of *Ficus*, in the botanical gardens at Hamma, Algiers. (8.) Two samples from *Kickxia Africana*, New Caledonia.

The specific gravity of rubber milk was found to be 1·017 and 1·041 by Ure, 1·012 by Muspratt. The above-named samples, however, showed a density varying between 0·980 and 0·990. Higher values were indeed obtained with some samples of milk from various kinds of *Ficus*, but these, at the time of their investigation, were found to contain the rubber in an already resinous form. The rubber globules in the milk are of absolutely the same size as the fat globules in animal milk. The amount of rubber in the milk from Para (*Siphonia*), was found to be 42 per cent., whereas the milks from other sources gave a yield smaller by about 10 per cent. All the various processes, employed in different parts for the coagulation of the rubber milk, depend upon the employment of heat, in one form or another. Any reagents, added to the milk, to induce coagulation, must be used in conjunction with heat. In a number of experiments, highly satisfactory results were obtained, by heating the rubber milk in a churn to about 50° C. In most cases a few turns of the churn were found sufficient to cause complete coagulation.—C. O. W.

*India Rubber Substitutes, A Source of Error in Henriques' Method of Estimating.* R. Thal. Mag. Pharm. through Chem. Zeit. 1898, **22**, [92], 974.

*See under* XXIII., *page* 1191.

### Errata,

This Journal, 1898, 1058 :—

Col. 1, 23 lines from bottom, for "*Kixia*" read "*Kickxia*."
" 5 " " for "*Carpodrinus*" read "*Carpodinus*."
" 4 " " for "*henriqueriana*" read "*henriquesiana*."
" 2, 4 " top, for "*obizomes*" read "*rhizomes*."
" 32 " bottom, for "Adriatic" read "Asiatic."

Page 1059, col. 1, 12 lines from top, for "*almoides*" read "*ulmoides*."

## XIV.—TANNING, LEATHER, GLUE, SIZE.

*Bates and New Bating Materials.* W. Eitner. Der Gerber, **24**, 577—560. (See also this Journal, 1898, 1010—1013.)

Formerly, a bate liquor, made by steeping oat straw in hot water, was used, but so mild was its action, that it was only suitable for such leathers as cow hide and calf skin, where only a mild bateing is desired.

*Combined Bates.*—In the manufacture of glacé leather, the bates play a much more important part than in the manufacture of other classes. In 1878, Knapp patented a combined bate of dung and bran, in the preparation of which, the dung bate, which had been previously dried, was mixed with cold water, and was thickened to a paste with bran. After standing some hours, a strong fermentation took place. This semi-liquid mass was then filtered through linen, and to this liquor was added small quantities of sodium carbonate and a few drops of sulphuric acid. The whole was now mixed with tepid water, and used for bating the skins. The object of the sodium carbonate was to act upon the natural grease of the skin. This combination of two bates has, however, gone out of use, and at the present time it is usual to first of all bate the skins in a dung bate, and afterwards pass them through the bran bate, carefully cleaning them on removal from the dung bate.

Great progress has recently taken place in connection with this subject, especially in the manufacture of artificial bates, or, at least, in the study of the precise action of the bate, with the result that it is now possible to check or increase the correct ferment at will. A recent advance, is the invention by Messrs. Popp and Becker, of Frankfort, who bate from the animal hide by treating the raw skin for between four and five hours, with coatings of dog or bird excrement and cultivating therefrom the special bacteria suited for further bating purposes.

A second patent, by H. Noerdlinger, is also of interest. This chemist prepares a bate by using potatoes as a culture material for the generation of bacteria. The whole is then dried, ground to a powder, and, on being treated with warm water forms a bating material for skins. It is claimed that this dried material may be kept for any period in a dry place undamaged. The potatoes are dried at a temperature of between 45° and 50°.

From long observation, both in the factory and in the laboratory, the author states that the utility of the bate depends essentially on :—

(*a.*) The direct action of the micro-organisms on the pelt.

(*b.*) The action of the decomposition products formed by the action of the micro-organisms on the nutritive material (enzymes).

The decomposition products produced are :—

(*a.*) Peptones, formed by decomposition of the proteids.

(*b.*) Gases (hydrogen, marsh gas, sulphuretted hydrogen).

(*c.*) Acids formed by fermentation of the byproducts.

In order to produce a bate which should possess the above requirements, the following was tried with good results :—

Soja beans (or any cheap bean) are boiled with water, to this is added 0·2 per cent. of their weight of soda, and filtered, 10 per cent. of dry dog-dung is now dissolved in hot water, filtered, and added to the bean water. The whole is kept in a warm place until in active fermentation. This was found to make a very suitable bate for calf or hides for dressing leather, but although it produced a fine smooth and tough grain, it was not sufficiently energetic in its action for glacé leather, it lacked the full effect produced by a dung bate, followed by a bran drench.

The following combined bate was, however, found to fully supply the deficiency, and it has been worked practically for some time in many yards with unvarying success.

Five kilos. of limy fleshings, fresh from the fleshing beam, are boiled with 40 litres of water until the gelatin is dissolved. This clear solution is now run into a tub containing 10 litres of bran. 100 grms. of sodium hyposulphite is now added (to produce $H_2S$ which forms soluble salts with the lime in the skins); 50 litres of hot water are then added, and the temperature maintained above 50° C. To start the fermentation, hay-water is added. This is made by heating a handful of fresh hay with water. The neck of the flask should be closed with a loose plug of cotton wool, and put in a warm place for a few days. About half a litre of the fermenting hay water is added to the bran and gelatin solution, and kept in a warm place for two days. At the end of that time the solution is ready for use in bating, either filtered or as it is. The bate is found to work best at 25° C., and must never be allowed to go below 15° C. This bate thoroughly replaces both the dung and bran bate, combining the characteristic action of both without the objectionable and variable effects of the former.

The above may be used to substitute the dung bate only by leaving out the bran and the hyposulphite. In either case the author has always obtained results, superior to those obtained by other methods.—J. G. P.

### PATENTS.

*Leather, Impts. in and relating to the Manufacture and Treatment* [*Formaldehyde*] *of.* H. H. Lake, 43, Southampton Buildings, London, on behalf of Dr. Carl Dreher, Höchst-on-the-Main, Germany. Eng. Pat. 25,871, Nov. 6, 1897.

By treating tanned leather with dilute solutions of formaldehyde, the tannin is fixed or bound in the fibre so that it cannot be washed out, and it may even be boiled without damage. For light leathers the formaldehyde may be added to the ooze, for medium leather only added to the last pit. For heavy sole leather it is best taken as it comes from the last pit and allowed to drip, and while still moist suspended for four hours in a solution of, approximately, 2 parts of formaldehyde to 100 parts of water; the leather is then removed, allowed to drip, and while still moist rubbed on the grain surface with glycerin or other suitable oil, to prevent the formaldehyde destroying the grain, and dried in the ordinary way. Leather so treated can be boiled without damage or the tan oozing out. As far as outward appearance goes, it cannot be distinguished from untreated leather. The leather may afterwards be dyed, dressed, or finished in any way, just as with ordinary leather. Such leather is claimed to be specially suited for belting, hydraulic, harness, or any leather which is exposed to moisture or steam; it is impermeable to wet, and does not shrink or become hard. —J. G. P.

*Enamelled Leather, Impts. in the Manufacture of.* W. F. Reid, Fieldside, Addlestone, and E. J. V. Earle, 139, Queen Victoria Street, London. Eng. Pat. 26,677, Nov. 15, 1897.

In this process the surface of the leather is impregnated with a weak solution of a mixture of nitrocellulose and nitrolinolein or nitroricinolein, after which a stronger solution is applied on the surface so prepared, afterwards evaporating the solvent. It may be applied either on the flesh or grain side of the leather. A suitable mixture is that consisting of 11 parts by weight of nitrated castor oil and 5 parts of soluble nitrocellulose. Five to 10 parts of this mixture are dissolved in acetone. Any colouring matters may be used soluble in the solvent.—J. G. P.

*Tanning of Skins and the like, and to Apparatus therefor; Impts. relating to the.* G. Fettweis, Vervier, Belgium. Eng. Pat. 5003, March 1, 1898.

For the purpose of producing rapid and regular tanning, the skins, after a slight preliminary tanning by any ordinary method, are introduced into a special rotary polygonal drum, provided with inclined battles and with recesses or grooves, which retain the skins as they slide on the baffles and force them to spread out.—J. G. P.

*Preserving and Tawing Skins, An Improved Method of.* E. de Günzburg, Vitry-sur-Seine, France. Eng. Pat. 6638, March 18, 1898.

A process for treating skins without the help or the formation of any soluble and crystallisable salt, essentially characterised by the fact that the skins treated may be kept indefinitely without returning to their natural state and without losing any suppleness or strength, being likewise unaffected by the action of hot or cold water. The skins to be treated are immersed in water and then submitted to a bath consisting of 3 parts of water to 1 part of "sulphhydrate of calcium sulphide," depilated and fleshed, and tawed, if necessary. They are then submitted to a bath containing 10 parts of water, and 1 part of bisulphide of alumina; a small quantity of hydrochloric acid may also be added. They are now allowed to drain, and then immersed in a bath containing liquid ammonia, together with a small quantity of neutral chromate of soda. The skins are then stocked in a fulling mill, with a firm paste, consisting of 8 parts of wheaten flour, 1 part of glycerin, and the residues of precipitation from the ammonia-bath. The skins may be dyed and finished as usual.

The special claim is the use of "hyposulphate of sulphide of calcium" in the natural state and of the "bisulphite of aluminium" with the addition of hydrochloric acid and liquid ammonia, with the addition of neutral chromate of potash, together with the special paste of wheaten flour and glycerin for tawing.—J. G. P.

*Skins, An Improved Process for the Preparation of.* F. Grognet, 6, Boulevard Nanterre, Dept. of Seine, France. Eng. Pat. 12,274, May 31, 1898.

The fresh skins are stretched on suitable frames and coated with a mixture of equal parts of clay (hydrated silicate of alumina), talc, and baryta. They are now placed in a dry room, and, by means of a ventilator or aspirator of any suitable kind, dry air is drawn through the chamber until the skins are dry.—J. G. P.

*Tanning Hides and Skins, Impts. in.* Jesse H. Curling, Trowbridge, Wiltshire. Eng. Pat. 16,369, July 27, 1898.

The improvement consists of suspending the hides to be tanned in a large drum, in which is a relatively small quantity of tanning liquor. The drum is so constructed that each time it revolves, the hides are lifted out of the liquor, and are hung so far apart in order to avoid rubbing; and, further, in the introduction into the drum from time to time of a relatively small quantity of turpentine as a means of discharging the air and generated gases in the drum, thus substituting an atmosphere of, or saturated with, turpentine vapour, thus submitting the suspended hides, while out of the tanning liquor, to the mellowing influence of such vapour alternately with the tanning action of the liquor. Weak liquors are used to commence with, and may be gradually raised to any desired strength. It is set forth that fermentation of the liquor is prevented, and the hides are rapidly converted into leather of high quality.—J. G. P.

## XV.—MANURES, Etc.

*Denitrification.* G. Ampola and C. Ulpiani. Gaz. chim. ital. **28**, [1], 410—438; Chem. Centr. 1898, **2**, [17], 896.

The authors have succeeded in isolating two kinds of denitrifying bacteria from the earth and from the air, that from the earth being obtained by means of anaerobic culture, and the other by directly exposing to the air a solution of sodium nitrate, sodium acetate, and sodium phosphate, according to the method of Béchamp. The behaviour of these bacteria towards sunlight, heat, drying, alkalis, acids, and the like are described, and also the conditions of growth on alimentary substrata containing, besides nitrates, fatty acids and amido acids. The denitrifying action of the bacteria was studied under anaërobic conditions, and also by use of an alimentary soil containing 1 grm. of grape sugar, 0·625 grm. of common salt, 0·002 grm. of tricalcium phosphate, and 0·6006 grm. of sodium nitrate in 100 c.c. of water. When the completion of the reduction of the nitrate was indicated by the disappearance of the foam on the surface of the liquid (after 10 days), the gases formed were examined. They consisted of carbon dioxide and nitrogen. A determination of the sodium nitrate remaining in the alimentary soil showed that 94·58 per cent. of the total nitrogen had been set free. Since the decomposition of the saltpetre proceeds according to the equation—

$$2NaNO_3 = Na_2O + N_2 + 5O,$$

the author calculated that all the sugar decomposed had been oxidised by means of the oxygen set free from the nitrate, this view being also confirmed by a determination of the sugar. The authors consequently express the denitrification by the equation—

$$5C_6H_{12}O_6 + 24NaNO_3 =$$
$$24NaHCO_3 + 6CO_2 + 18H_2O + 12N_2.$$

Denitrification ceases as soon as the whole of the sugar has been consumed, and inversely, the sugar is no longer destroyed after complete decomposition of the nitrate. It was concluded that denitrification does not proceed by stages, but that nitrogen is formed at once, since nitrite could neither be detected in the course of the reduction, nor could the bacteria be developed on alimentary soil containing nitrite.—A. S.

*Manures, Effect of, on the Quality of the Experimental Grain Crops Grown at Woburn.* J. A. Voelcker. J. Roy. Ag. Soc. Eng. 1898, **9**, [3], 551—555.

Wheat and barley have been grown at Woburn for 21 years on the same land without manure, and with the same manures applied year by year to the same plots. The amount of produce and the weight per bushel have been recorded each year, whilst in the case of last year's crop, the comparative market values of the grain have also been estimated.

The wheat was, on the whole, of fair quality, the best plots—those which received minerals and ammonia—being decidedly above the average. The most striking result was the marked difference between the effect of ammonium salts and nitrate of soda, the nitrate having produced the poorest quality of all, thin and shrivelled grain of much lower weight per bushel. There was also a great increase in offal grain under the influence of nitrate of soda. Both dung and rape cake produced grain of inferior quality as compared with the unmanured and exclusively mineral plots.

The barley was badly weathered, and of inferior quality. The results, which are therefore given with some reserve, seem to indicate that nitrate of soda, in conjunction with minerals, does not produce the same marked deterioration which was observed in the case of wheat. Ammonium salts gave the worst results, owing, perhaps, to the impoverishment of the soil in lime. Dung stands high as regards the quality of the dressed grain, but produced, like rape cake and nitrate of soda (= 100 lb. of ammonia per acre), a high proportion of offal.—N. H. J. M.

*Chili Saltpetre, Allowable Quantity of Perchlorate in.* Maercker. Landw. Vers.-Stat. **51**, 39—43; Chem. Centr. 1898, **2**, [18], 935.

A description is given of the reports from the different branches of the German Union of Agricultural Experimental Stations with regard to the occurrence of perchlorate in Chili saltpetre.

In Halle, 107 samples were examined, with the following results :—

| —— | Nitrogen. | $NaNO_3$. | Perchlorate. |
|---|---|---|---|
| | Per Cent. | Per Cent. | Per Cent. |
| Maximum ............ | 15·6 | 94·7 | 5·64 |
| Minimum ............ | 13·8 | 83·8 | 0·27 |
| Mean................ | 15·1 | 91·6 | 0·94 |

From these figures it is proposed not to object to 1·5 per cent. of perchlorate.

In Pommritz the following method of examination was used :—(1.) The sample is mixed with a small quantity of alkali hydrate or carbonate, ignited, and the total chlorine determined. (2.) 5 grms. are gently boiled for ½ hour with 10 grms. of washed (free from chlorine) zinc dust and 150 c.c. of a 1 per cent. acetic acid solution. In the filtrate, the chlorine present as chloride and chlorate is determined. (3.) The chlorine present as chloride is determined in the aqueous solution of the sample. The amounts of chloride, chlorate, and perchlorate may easily be calculated from the three values thus obtained. Of 24 samples of saltpetre containing perchlorate, 20 were found to also contain chlorate (0·1—1·0 per cent.).

In Darmstadt, in the spring of 1897, experiments were carried out with oats and spring rye concerning the injurious effect of perchlorate and chlorate. The plants were manured on the one hand with a quantity of pure sodium nitrate corresponding to 10 cwt. (Centner) of Chili saltpetre per hectare, and on the other hand with the same quantity of nitrate, but with increasing amounts of potassium chlorate and perchlorate added. The manuring was given with the seed-corn of the plants. From the results obtained, the following relative values were calculated :—

| —— | Oats. | Rye. |
|---|---|---|
| With pure $NaNO_3$ .............. | 100 parts more produce. | 100 parts more produce. |
| „ $NaNO_3$ + 1·5 per cent. of perchlorate. | 105 parts more produce. | 99 parts more produce. |
| „ „ + 3·0 per cent. of perchlorate. | 101 parts more produce. | 78 parts more produce. |
| „ „ + 4·5 per cent. of perchlorate. | 84 parts more produce. | 66 parts more produce. |
| „ „ + 6·0 per cent. of perchlorate. | 77 parts more produce. | 41 parts more produce. |
| „ „ + 9·0 per cent. of perchlorate. | 24 parts more produce. | 7 parts less produce. |
| „ „ + 12·0 per cent, of perchlorate. | 8 parts less produce. | 0 parts produce. |
| „ „ + 15·0 per cent. of perchlorate. | 10 parts less produce. | 0 parts produce. |
| „ „ + 18·0 per cent. of perchlorate. | 0 parts produce. | 0 parts produce. |

Chlorate does not act quite so injuriously as perchlorate.

It is not considered advisable, in the meantime, to fix a limit for the allowable amount of perchlorate, until a standard method has been agreed upon for its determination.—A. S.

*Phosphates, Reynoso's Method for the Analysis of,* U. Antony and G. H. Mondolfo. Chem. Centr. 1898. 2, [18], 941.

*See under* XXIII., *page* 1186.

*Basic Slag, Estimation of Citrate-soluble Phosphoric Acid in.* J. Freundlich. Chem. Zeit. 1898, 22, [92], 974.

*See under* XXIII., *page* 1186.

*Phosphates, Mineral and Animal; Method of Distinguishing.* F. Martinotti. Staz. sper. agr. ital. 1897, 663.

*See under* XXIII., *page* 1182.

## XVI.—SUGAR, STARCH, GUM, Etc.

*Beetroot Juice [Sugar], Raw Diffusion; Action of Lime on.* H. Pellet. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, 16, [3], 257—263.

If to 100 c.c. of cold diffusion juice, containing 10 to 12 per cent. of sugar, there be added 10 to 12 per cent. of milk of lime at 20° B., containing about 20 per cent. of pure lime, and the mixture be then heated to 70° C., more or less insoluble sucrate is formed, enclosed in the precipitate of organic calcium matters, and it is only partially decomposed on carbonating. The filtered juice polarises less than the original by 0·40 to 0·80 per cent.

The lowering of the polarisation is represented by a corresponding quantity of sucrate found in the scums, so that if there are losses of sugar during carbonation, they are determinable.

If the juice be heated to 70° before lime is added, the formation of sucrate does not occur, nor is it formed in pure sugar solutions of the same strength, unless some substance is added, such as pectic acid or albumin, capable of forming a precipitate with lime.

These facts are dependent on the varying solubility of lime in sugar solutions at different temperatures.

The purification appears to be the same whether the juices are limed hot or cold. The filtered juice obtained by adding lime to the cold juice and then heating to 70° C. for two hours, is more coloured than that from juice heated to 70° C. before being limed and heated for the same length of time.

Cold press juice shows the same phenomena as diffusion juice; in fact, so do all the liquors of the sugar works from a purity of 88 or 89 downwards; the diminution of polarisation being greater with products of low purity owing to the greater precipitate formed, although not exactly in proportion to it, the impurities precipitated by the lime seeming to enclose the insoluble sucrate and prevent its resolution. In pure sugar solutions the precipitated sucrate, if formed, is entirely redissolved on cooling.

From the point of view of the action of lime, there are no exceptional juices, as has been assumed, to explain differences in the results of various chemists; it is only the conditions of experiment which have varied.

—L. J. de W.

*Sugar Solutions, Action of Lime on.* J. Weisberg. Bull de l'Assoc. des Chim. de Sucr. et de Dist. 1898, 16, [3] 263—280.

The treatment of a diffusion juice by lime at a temperature approximate to that at which it leaves the battery, and therefore the temperature at which the defecation is carried out in practice, generally gives rise to a diminution of polarisation. The average diminution in the author's experiments was 0·16 per cent. of sugar. This relatively small loss of polarisation, as he has previously shown, is a loss of sugar which may be easily recovered from the calcium precipitate, where it exists as sucrate, and it is readily recovered during carbonation on the large scale. Pure sugar shows no diminution of polarisation under the same conditions unless a substance like pectin is added. This does not mean that pectin is the only substance among the numerous organic and inorganic matters in raw juice which has this effect; probably all the impurities contribute in some measure.

When lime is added to cold juice (contrary to the practice on the large scale), and the limed juice heated to about 70° C., there is a considerable diminution of polarisation, and the filtered juice is darker in colour. If powdered quicklime be used instead of slaked lime, the colour is darker still. The alkalinity is found to be greater in those cases where lime was added to the cold juice, and more lime is dissolved when it is added as a dry powder. The sugar lost here also may be recovered in the scums.

The results obtained by Deltour (La Sucrerie Belge, 1896, [4], Oct. 15), who worked on limed juice coming from the rasping works of the central factory at Wauze, Belgium—that is, on juice from an exceptional source—showed a loss of sugar. Now this juice, according to Pellet, was at

25° C., and it remained a long time in contact with lime before being carbonated. The explanation of the loss in this case is to be explained by the relatively low temperature of the limed juice.—L. J. de W.

*Caramel [Sugar], Chemical Nature, Occurrence in the Sugar Factory, and Determination of.* N. Fradiss. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 280—286.

Sugar heated to 200° C., or treated with concentrated sulphuric acid, or boiled with caustic soda, becomes of a reddish-brown colour. When treated with ammoniacal lead acetate, and the precipitate decomposed by hydrogen sulphide to remove lead, and the solution evaporated, the substance obtained in all three cases shows all the characters of caramel, although more or less masked by impurities. According to Mulder, the action of heat on sugar generates humic products; it is also probable that caramel and assamar are only modifications of humic acid, *i.e.*, more or less dehydrated carbohydrates. Jackson and Tollens have likewise recognised the formation of humic acid with disengagement of formic acid when sugar solutions are heated to 160° C. The last condition is of importance, for syrups are frequently in contact with the coils heated to 140°—150° C.

Adopting Berthelot's formula, $C_{18}H_{14}O_6$, for humic acid, the author calculates that 1 grm. of caramel requires 2·10 grms. of saccharose for its production.

The properties of caramel are:—

It is soluble in methyl alcohol and in water; insoluble in amyl alcohol of ether, and chloroform, and slightly soluble in ethyl alcohol. It reduces Fehling's solution and ammoniacal nitrate of silver. Caramel is precipitated by amyl alcohol from its solution in methyl alcohol. It is completely precipitated by an excess of ammoniacal lead acetate. Caramel does not crystallise.

To estimate caramel, the dry extract is treated with methyl alcohol of 95°, and the solution precipitated with amyl alcohol, the operation being repeated to get rid of traces of sugar. The solution is then evaporated, dried at 90° C., and weighed. To make a quick estimation in a product of the same manufacture, the Fehling or Violette solution should be titrated with the caramel obtained. It is evident that if the product contains glucose, two tests must be made, one before, and one after, defecation with a large excess of acetate of lead.

The following are the amounts of caramel found:—

| | Per Cent. |
|---|---|
| 1st jet syrup | 0·05—0·08 |
| 1st jet massecuite | 0·50—0·90 |
| 2nd jet massecuite | 1·10—1·50 |
| 3rd jet massecuite | 1·50—2·10 |
| Molasses of 44 per cent. sugar | 1·10—2·50 |

This quantity of caramel found in the molasses, if part has not been eliminated by any decolorising process, calculated to the whole of the molasses, may account for a loss of sugar about equal to three-fourths of the unknown losses of manufacture.—L. J. de W.

*Sugar Products, Qualitative Test of Alkalinity or Acidity of.* J. Zamaron. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 295.

*See under* XXIII., *page* 1188.

*Indicators, Study of, on Sugar Juices and Syrups.* Gubal. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 288.

*See under* XXIII., *page* 1188.

### PATENT.

*Sugar in the Form of Bars, Cubes, and other Shapes; Improved Process and Apparatus for Producing.* E. Kommerell, Linteranskja, Russia. Eng. Pat. 11,813, May 21, 1898.

This invention relates to the employment of moulds for the formation of sticks of sugar, constructed of oblong, flexible, perforated plates, having ridges attached to them at right angles to their length, so that a number of them, when placed and wedged together in a frame, form a series of rectangular spaces into which the massecuite is poured. The frames are placed in a centrifugal machine and spun. The sticks of sugar are easily removed from their position in the mould by pressing the flexible plates endways, thus causing them to assume a curved shape, so that the sticks of sugar, being on the convex side, fall out on to a travelling band provided to receive them.

—J. F. B.

## XVII.—BREWING, WINES, SPIRITS, Etc.

*Fermentation in Recent Years, The Progress in the Chemistry of.* M. Delbrück. Ber. 1898, **31**, [13], 1913—1925.

In spite of the important discoveries of Pasteur, which commenced in 1857, no considerable application of the scientific investigations in this branch of chemistry was made in the industries on a large scale between the years 1857 and 1870. In 1870, through the efforts of Maercker, Reess, and others, it was generally recognised that, in order to avoid loss, the fermentation must be purely alcoholic, conditions must be favourable to the yeast and adverse to foreign organisms, and that if sufficient yeast were sown, the growth of foreign organisms was hindered by its development, if the liquid were kept in motion. In the eighties the laboratory methods for obtaining pure cultures from single cells were developed by Pasteur, Koch, and Hansen, and then extended by Hansen and Jacobsen to the preparation of pure yeasts on the large scale. Differences were recognised between the properties of different varieties, and the types separated were applied to the purposes for which they were specially suited. Fischer's work on the synthetical sugars, and the effect on them of different yeasts, appeared towards the end of the decade, and the fact that the variations between different races depended on the presence of certain carbohydrate-enzymes, was recognised. Only glucose was found to be directly fermentable, and the other carbohydrates had first to be hydrolysed by the enzymes of the particular yeasts. In the fermentation industries, the system of the "struggle for existence" was applied to the separation and preservation of pure races from originally mixed yeasts.

In the present decade, Buchner's discovery of zymase, involving a modification of the vitalistic theory of fermentation, is the most important. The action of zymase, consisting of the simultaneous processes of oxidation and reduction in alcoholic fermentation, is far more complicated than that of the other enzymes, which merely hydrolyse, and it is an important step in the direction of those enzymes which bring about the syntheses and analyses which characterise the metabolism and growth of living plants.

If zymase be a proteid secretion of the living yeast-cell, it will be possible to increase its formation by favourable nutrition. Hayduck, in the eighties, showed that the fermentative power of living yeast varied with the nitrogenous nutrition, and, so far as can yet be judged, fermentative power is identical with percentage of zymase. If the multiplication of yeast be stimulated by aëration, the increased vegetative activity is only obtained at the expense of fermentative power and secretion of enzyme. "Lufthefe" is ordinary distillery yeast, Race II., whose multiplication has been so stimulated by aëration, that a given weight of malt yields more than double the weight of yeast obtained under ordinary conditions; its fermentative power and enzyme content are very low, and it is only used for baking purposes. Fermentation at low temperatures has the opposite effect; the multiplication is decreased and the percentage of albuminoids and zymase is increased, as, for instance, in the case of bottom-fermentation beer yeast with a high fermentative power. Thus, the formation of zymase from proteids is dependent on, and a measure of, the physiological activity of the yeast. The acclimatisation of yeast to adverse conditions, often with the evolution of new races, has recently been studied by Hansen and many others.

The latest method for the isolation of pure varieties is Lindner's droplet method, which is replacing the older ones.

Races cannot be conveniently classified by morphological characters and properties of nutrition alone, but only in conjunction with the content of carbohydrate-enzymes present. Thus top-fermentation beer yeast contains an enzyme which converts melitriose (raffinose) into melibiose, Apiculatus yeast only ferments the dextrose in boiled beer-worts, Saaz yeast also ferments maltese, Frohberg further attacks maltodextrin (isomaltose), Pombé and Lagos yeasts saccharify and ferment dextrin, and, finally, *Amylomyces Rouxii* of Calmette will saccharify and ferment soluble starch. The various properties of the enzymes of different races of yeasts have been applied in the chemical analysis of carbohydrates.

The different races and varieties are modified by acclimatisation to produce the greatest effects in their own particular applications, and institutes exist in Germany for their preparation and supply; the author quotes an instance where 14,000 kilos. have been supplied of a particular race from a single cell.

Naturally, since the properties of yeast are now so highly specialised, improvements have taken place in plant and machinery to ensure perfectly aseptic conditions throughout all the processes, so as to keep the pure cultures in use on the large scale as long as possible.

In the wine industry, progress has of necessity been much slower than in the brewing industry, since the must cannot be sterilised, and experiments can only be tried during a small portion of the year, and the properties of wines probably depend on the complex mixture of the wild yeasts employed. Nevertheless, pure cultures of yeasts, specialised for certain aromas and other qualities, are being tried with success, at Geisenheim am Rh. by sowing a quantity in active fermentation sufficient to suppress the growth of the others.

In the spirit industry the special application is in the suppression of acid-forming bacteria by the presence of lactic acid, developed in or added to the mash, and in the education of the yeast to withstand the presence of this lactic acid and to resist a high percentage of alcohol. On the large scale, a partial separation of varieties of yeast in a mixture can be effected by adjusting the temperature. At 24° C., distillery yeasts will predominate; at 15° C., beer yeasts; and at 5° C., wild yeasts predominate. This separation is aided by the "settling" process, in which the cells of the different varieties, after being kept in motion by the carbon dioxide evolved from them, sink to the bottom when the fermentation of each variety ceases at different times, and form layers, which can be separated by pumping off the liquid. The wild yeasts are the last to settle.

The motion caused by the evolution of gas assists in the success of the most abundant variety in the struggle for existence by distributing the special toxin which is fatal to the growth of the other organisms, provided these are not acclimatised to it.

Finally, the author summarises the many important questions which are suggested and nearing solution by the recent work in this branch of chemistry, especially the extension of the enzyme researches of Buchner, Beijerinck, and Wysman, and others, and the possibility of discovering enzymes or conditions of enzymes already known, which will build up as well as break down the carbohydrates, as is the case in living plants.

### *Fermentation Liquids, Deficiency of Phosphoric Acid and Nitrogen in.* Delbrück. Wochenschr. für Brau. 1898, **15**, [43], 586.

In factories where vinegar is manufactured from alcohol, it is necessary to add some artificial nutrient to preserve the vigour of the organisms. In the manufacture of wine, if the grapes contain an abnormally high percentage of sugar in relation to non-sugar, ammonium phosphate is often added to the fresh must, and the same procedure has been followed with advantage in molasses distilleries. The potatoes of this year's crop in Germany are unusually rich in starch and poor in albuminoids and ash, and distillers are recommended to try the effect of artificial nutrients in making up the mash. Also, this year's exhibition has shown that the present crop of German barley is of exceptionally good quality, very rich in starch and especially poor in albuminoids; phosphoric acid is also probably deficient. It will be interesting to hear whether the fermentation of beers made with the new malt has been of normal character, or whether the yeast quickly degenerates. Instead of adding nutrient salts, it is recommended that German malt be mixed with other malt to obtain sufficient food for the yeast.—J. F. B.

### *Spore-Formation in Alcohol-Yeasts, Regeneration of, when this Function has been in Abeyance.* M. W. Beijerinck. Centralbl. für Bakteriol., II. Abth. 1898, **4**, [17], [18], and [19].

The original, strong sporulation of the so-called "wild yeasts" is, to a great extent, suppressed by cultivation; this paper deals with the conditions under which spore-formation can be re-established. Very definite results were obtained with *S. octosporus*; as a rule, asporogenous cells gave only asporogenous descendants, and spores developed into both sporogenous and asporogenous cells. With the other species of yeasts and with *Schizosacch. Pombe* the relations are not so simple, since not only two, but several, forms of cells are met with, with varying powers of spore-formation, but generally colonies formed from spores are sporogenous and *vice versâ*. The fact that the wild yeasts in breweries so often exhibit sporulation is explained by the fact that they are generally descendants of spores occurring on the malt, which have not been killed at the temperature of kilning. With a plate culture of *S. Ludwigii*, containing only a few spores, the author found that by careful drying at a temperature of 50° C. for several hours, the vegetative cells could be killed, whilst all the spores survived, and each subsequently developed into colonies rich in spores. With different species, a different temperature and time of heating was required, and with a pressed yeast, described as *S. panis*, the vegetative cells could not be killed, even at 100° C., without killing the spores at the same time; attempts to effect the separation by moist heat and by various chemicals also failed. After the drying process, in a few cases it was found that some vegetative cells remained alive together with the spores, constituting a more resistant race, the properties of which were hereditary, and the descendants characterised by the very small size of their cells, which were also asporogenous and resistant to heat. The author states that this dwarf race was not evolved by the treatment, but was present in the original culture, and the treatment merely afforded the opportunity of development. Since the development of sporogenous colonies is much slower than that of the vegetative colonies, a partial separation can often be effected by selection of the smallest and least developed colonies.

For inducing sporulation in yeasts, the author used pure agar, freed from its soluble constituents by washing for a week and rotting in distilled water. The yeasts were sown on this on sloped plates, and incubated at a temperature of 21°—25° C., under which conditions, budding no longer took place. In the case of *octosporus*, a temperature of 28°—30° C. hindered spore-formation, but at 20° C. the ratio of the two forms was about equal.

In *octosporus* the sporulating modification is snow-white, liquefies gelatin rapidly, and gives a blue-black coloration with iodine; the asporogenous colonies, on the other hand, are first white, then brownish, do not liquefy gelatin, and give no reaction with iodine. With the other yeasts, the characteristics are not so marked, and are somewhat variable, according to the intensity of the sporulation. Spores and sporogenous colonies can generally be distinguished from vegetative colonies by means of iodine. With this reagent, either the spores are coloured blue by reason of their granulose, whilst the cells remain colourless; or the budding cells are coloured violet-brown in consequence of the presence of glycogen, whilst the sporulating cells remain colourless (*S. uvarum*); or, lastly, the sporulating modification may contain glycogen and give the iodine reaction, whilst the budding cells are free from the same (*S orientalis*). Sporogenous colonies liquefy gelatin far more quickly than vegetative colonies, and the former are often snow-white, whilst the latter have a watery-brown colour—a distinction attributable to the relative sizes of the cells.

A surface sub-culture of *Schizosacch. Pombe* on wort-gelatin was made from a sample which contained only a small percentage of sporulating cells. After 3—4 weeks the proportion of spore-bearing cells was much greater, and in a sub-culture from this, it increased still more. The sporogenous cells in the colonies of these cultures were found to have a regular, radial arrangement, but the streaks did not run from the centre, but from the middle of the radius.

*S. Pombe* is characterised by the fact that, by repeated sub-cultivation, the sporogenous form displaces the asporogenous, whilst in *octosporus* the reverse is the case.

A sample of *S. uvarum* was isolated in 1894 from currant-juice; at first nearly every cell contained spores, but after the lapse of time, sporulation had almost entirely disappeared. The sporogenous modification of *S. uvarum* can ferment malt-worts at 28° C., whilst the asporogenous modification only ferments it slightly at this temperature (from 10° Ilg. to 7°—8°), and then the fermentation stops; on cooling to 25°—20° C., however, it again proceeds and takes place normally. The reason for this effect of temperature may be looked for in the greater demand for oxygen of the budding cells.

*Mycoderma orientalis* has been isolated from Japanese "white koji" and later from Turkish currants and other Oriental fruits. It is best obtained by bringing Turkish currants into a malt-wort acidified with 3—5 c.c. of normal butyric acid per 100 c.c. at 28° C. It forms a very dry and white film containing spores; it is a glucose yeast, and maltose is not fermented by it, but if sufficient air be supplied, maltose is oxidised and utilised as a nutrient. This yeast rapidly liquefies gelatin, but loses this property after a time, and the film becomes moist and greyish. The spores store up much glycogen, whilst the vegetative cells contain none. The sporogenous and asporogenous modifications have identical fermentative properties. The sporulating colonies are very easily detected amongst the colonies of budding cells on wort-gelatin plates by their rapid liquefying action. After two or three sub-cultivations, a constant ratio of strongly sporogenous to less sporogenous colonies is established, the latter being comparatively few. The author has not tried to isolate the purely vegetative modification from colonies containing a few spores. The strongly sporogenous form is suppressed on repeated sub-cultivation, because the spores do not germinate immediately, but pass through a quiescent stage in the gelatin, whilst the vegetative form goes on budding.—J. F. B.

*Spirit from Sawdust: Results of Preliminary Experiments on a Manufacturing Scale.* E. Simonsen. Zeits. angew. Chem. 1898, [12], 962—966, and [44], 1007—1012.

In a parallel series of experiments on the small scale (this Journal, 1898, 365 and 481), the author found that the best yield of alcohol could be obtained from sawdust under the following conditions of inversion:—

Strength of acid, 0·5 per cent. ($H_2SO_4$); pressure, 9 atmospheres; ratio of wood to liquid 1:4 or 5; time, 15 minutes. The yield was 6·5 litres of absolute alcohol from 100 kilos. of air-dried sawdust (8 per cent. of moisture). The yield of sugar after inversion, as determined with Fehling's solution, was 22 to 23 per cent.

He has since made experiments on a large scale in Christiania, using for the inversion a large steam-pressure boiler and a cylindrical rotating autoclave of a capacity of 7·5 cubic metres, lined with lead. The inverted liquid was nearly neutralised in large wooden tanks, and fermented with yeast, to which was added, as food material, a solution composed of water, 2 litres; ammonium nitrate, 0·5 kilo.; hydrogen potassium sulphate, 0·25 kilo.; calcium phosphate, 0·25 kilo.; and magnesium sulphate, 0·25 kilo. The fermentation was carried out at a temperature of 25° C., and usually required from three to five days.

In the large boiler it was found that the time of heating was disproportionately long, on account of the slow rate at which the pressure rose, notwithstanding the fact that the boiler was well insulated with cement. The wood (500 to 1,000 kilos.) was thus maintained at a high temperature for too long a time, with the result that substances interfering with the fermentation were produced. Thus, in raising the temperature to 100°, the time taken was 15 minutes; from 100°—173° C. (corresponding to 9 atmospheres pressure), 2 hours; boiling, 15 minutes; blowing off steam, 1½ hour; total, 5 hours. By coupling with the boiler an ordinary locomobile, so as to introduce greater quantities of steam, the total time of heating was reduced to about 3¾ hours. But the fermentations of the inverted liquid were still unsatisfactory. The best yields by a single inversion in the large boiler were from 2·5 to 3 litres of alcohol from 100 kilos. of wood.

In the small autoclave the time of heating was:—

To 100° C., 15 minutes; 100° to 173°, 30 minutes; boiling, 15 minutes; blowing off, 1 hour; total, 2 hours. Experiments were made with double and treble inversion in the autoclave, in order to determine whether the liquid obtained after one boiling could invert a fresh charge of sawdust, but the results were unfavourable. The increase in the yield of sugar in the double inversions was usually only from 50 to 70 per cent., and in treble inversions the increase was usually in the ratio of 1:1·5:2 instead of 1:2:3. In the subsequent experiments only one inversion was used, and as large a yield of sugar was obtained as on the small scale, whilst the amount of alcohol produced was in the best experiments larger. The following examples, taken from a long table given in the original paper, illustrate the results obtained by single inversion.

| No. | Sawdust. | Moisture. | Total Liquid. | $H_2SO_4$ in Liquid. | Pressed Residue. | Water in Residue. | Sugar Solution Obtained. | Per Cent. of Sugar. | Amount of Sugar. | Yield of Sugar from Sawdust. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Kilos. | Per Cent. | Litres. | Per Cent. | Kilos. | Per Cent. | Litres. | | Kilos. | Per Cent. |
| 44 | 100 | 18·5 | 350 | 0·5 | 115 | 45 | 355 | 5·0 | 17·7 | 17·7 |
| 45 | 100 | 17·1 | 300 | 0·58 | 99 | 45 | 310 | 5·5 | 17·1 | 17·1 |
| 46 | 80 | 20 | 100 | 0·5 | .. | .. | 390 | 5·0 | 19·5 | 24·3 |
| 47 | 100 | 20 | 400 | 0·5 | .. | .. | 460 | 5·0 | 23·0 | 23·0 |
| 48 | 100 | 20 | 400 | 0·5 | .. | .. | 450 | 5·3 | 23·8 | 23·8 |

| No. | Acidity. 10 c.c. = c.c. N/10 NaOH. After Inversion. | Acidity. 10 c.c. = c.c. N/10 NaOH. After Neutralisation. | Unfermented Sugar, calculated on Total Sugar. | Fermented Sugar, calculated on Total Sugar. | Per Cent. of Alcohol in Fermented Liquid. | Alcohol, Quantity. | Calculated on 100 Kilos. of Wood. |
|---|---|---|---|---|---|---|---|
| | C.c. | C.c. | Per Cent. | Per Cent. | | Litres. | Litres. |
| 44 | 11·5 | .. | 25·7 | 74·3 | 1·70 | 3·0 | 6·0 |
| 45 | 21·0 | 3·0 | 43·0 | 57·0 | 0·4 | 0·6 | 1·2 |
| 46 | 11·0 | 2·0 | 22·2 | 77·8 | 1·33 | 5·18 | 6·5 |
| 47 | 17·0 | 2·5 | 22·0 | 78·0 | 1·56 | 7·17 | 7·2 |
| 48 | 11·0 | 1·5 | 21·1 | 78·9 | 1·61 | 7·24 | 7·2 |

With regard to the influence of the individual factors on the results, the author makes the following remarks:—

1. The sawdust may be coarse or fine, without affecting the yield. Pine sawdust is superior to fir sawdust, as the crude material, and yields a purer alcohol. In all the experiments, the sawdust was in a fresh condition, and the yield from decomposing sawdust has not yet been determined. The quantity of water it contains is immaterial, provided it be taken into account, and the correct ratio between the amount of liquid and wood be maintained. In an experiment on the small scale, with birch sawdust, a high yield of sugar was obtained (30·8 per cent.). Plane also appears to be a suitable raw material.

2. The ratio between the amounts of wood and liquid should be as 4 : 1. If the ratio is lower, the results are uncertain, and if it falls as low as 2·5 : 1, the results are bad. It was found experimentally that it was slightly advantageous to leave the wood in contact with the acid liquid for some time (*e.g*, overnight) with occasional stirring.

3. As in the experiments on the small scale, the best results were obtained by inversion with a liquid containing about 0·5 per cent. of acid. Greater or smaller degrees of acidity gave bad results.

4. The pressed residue from the inversion, containing about 45 per cent. of moisture, can be used as fuel for the autoclave. Any sulphuric acid left in it does not affect the bottom of the boiler.

5. The volume of the sugar solution varied somewhat, according to the quantity of steam condensed and the temperature of the liquid introduced into the boiler. The volume of the final solution was from 10 to 25 per cent. greater than the initial liquid.

6. The amount of sugar contained in the inverted liquid was usually at most 5 per cent., but was occasionally slightly higher, when the volume of the liquid was small.

7. Calculated on the quantity of wood used, the yield of sugar was about 22 per cent. (Birch, 30·8 per cent.). From sulphite cellulose a yield of 45 per cent. was obtained. A yield of 22 per cent. from the wood corresponds with 45 per cent. from the cellulose in the wood.

8. In all instances organic acids, including acetic acid, were formed during the inversion, and a considerably greater quantity in double and treble inversions.

9. The best yields of alcohol were obtained by not completely neutralising the liquid before fermentation.

10. With but few exceptions, better results were obtained with bottom yeast than with top yeast. Sometimes a top yeast gave as high a yield of alcohol, but never higher; and in most of the unsuccessful experiments (*e.g.*, No. 45 in the table) the yeast was top yeast. The temperature found to be most suitable for the fermentation was 25° C. At the end of the fermentation, the yeast was still capable of fermenting a further quantity of sugar. About 6 litres of yeast were used with each 450 litres of the inverted liquid. The fermentation continued until 75 per cent. of the sugar, as determined with Fehling's solution, had been fermented. The residue probably consisted of unfermentable sugars (xylose, &c.) (this Journal, 1898, 682).

11. The amount of alcohol in the fermented liquid varied in good results between 1·0 and 1·7 per cent. In one exceptional instance (bottom yeast), the yield was as high as 2·5 per cent.

12. The quantity of absolute alcohol obtained from 100 kilos. of air-dried sawdust (20 per cent. of water), was 7·2 litres in the best experiments. The yield in the exceptional case alluded to above, was 7·7 litres.

13. The quality of the alcohol distilled from the fermented liquid is said to have been excellent, and preliminary experiments indicated that the trifling impurities could be readily removed. An analysis was made by Schmelk, of Christiania, of a specimen of the spirit prepared by repeated distillation, but without the use of modern rectifying apparatus. His results were:—Specific gravity at 15° C., 0·8336; alcohol, 90·21 per cent. by volume; solid matter, none; methylic alcohol, trace; fusel oil, none; aldehyde, trace; furfural, trace; ester, trace; free acid, trace; ethereal oil, none.

—C. A. M.

*Beetroot Distilleries, Chemical Control of.* D. Sidersky. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 312—323.

*Exhaustion of the Pulp.* — From numerous analyses effected since 1894 in various beetroot distilleries it appears that the sugar left in the pulp only varies from 0·42 to 0·50 under normal conditions. The best exhaustion is obtained with small, narrow macerators, not holding more than 2,000 kilos. Under adverse conditions the pulp may contain over 1 per cent. of sugar.

The Boullenger diffusion process allows of an average exhaustion of 0·30 per cent. of sugar being obtained.

The pulp from continuous pressing contains on an average 1·60 per cent. of sugar, but the quantity of pulp left is much smaller than by other processes.

| — | Percentage of Sugar in the Pulp. | Percentage of Pulp. | Percentage Loss of Sugar. |
|---|---|---|---|
| By maceration ............ | 0·46 | 66 | 0·30 |
| By Boullenger diffusion .... | 0·30 | 66 | 0·20 |
| By continuous presses ...... | 1·60 | 26 | 0·41 |

With regularly-cut beetroot slices, a normal exhaustion is always obtained irrespective of their form. A distillery which had retained the old Champonnois root-slicer and producing ribbons gave 0·52 per cent. of sugar in the pulp.

The exhaustion is also influenced by the quantity of juice drawn off, the temperature of the wash, and the form of the macerators. If the macerators be too large, samples taken from different parts of the same vessel show abnormal variations. The amount of juice drawn off from 100 kilos. of beetroot should not be less than 150 litres, and usually ranges from 160 to 185 litres. The temperature of the wash exercises a great influence. Ordinarily it runs at 97° C., and leaves the perforated plate of the last macerator at 90° or 92° C.

When the juice reaches the last macerator at 84° or 85° C., the exhaustion is normal; if it be too hot, it is advisable to run a thin stream of cold water along with the boiling wash.

It is a striking fact that the exhaustion of the pulp varies from one factory to another, working under normal conditions, but is almost constant in each works. The small amount of sugar contained in diffusion pulp is only an apparent exhaustion, as the light liquors, which drain off on emptying the diffusers, are run to waste. This thin juice contains about 0·20 per cent. of sugar. Its volume is estimated at 80 to 90 litres per 100 kilos. of beetroots.

*Accidents of Fermentation.*—With beetroot juice containing a considerable proportion of sugar not converted into glucose (invert sugar), the yeast is charged with the double duty of converting the sugar and fermenting it. Examination showed that in maceration, 8 to 10 per cent., and in diffusion, 10 to 12 per cent., of the sugar is converted into glucose. Thus, maceration juice of specific gravity 1·030 contains 0·5 to 0·6 per cent. of glucose, and 5·4 to 5·5 per cent. of sugar not directly fermentable. Variations from these figures are due to the quantity of acid, the density of the juice, and slowness of running; the glucose formed being greater with greater acidity, weaker juice, and slower running.

Vicious fermentations, where the medium is invaded by lactic, butyric, and other foreign ferments, are caused by want of cleanliness, lack of air, insufficient acidity or too high a temperature. An excess of acid, or too low a temperature in the vats fatigues the alcoholic ferment and prevents suitable development.

On commencing the maceration with water, all goes well; it is only on working with wash that troubles begin, the wash containing all the elements of the preceding fermentation except the volatile products. Every fermentation, even the most normal in appearance, gives rise to the formation of secondary products capable of engendering *vicious* fermentations. These products are not destroyed by the ebullition in the column, but accumulate in the wash and return continually to the vats, rendering alcoholic fermentation more and more difficult. It is therefore, necessary to dilute the wash periodically and to reject the excess. For normal work the wash must have a density practically constant, and an acidity less than that of the fermented liquor from which it is derived.

After a bad fermentation it is advisable to wash with a solution of bisulphite of lime.

It is further advisable to thoroughly aërate the juice before it reaches the fermentation vat in order to dissolve as much oxygen as possible, of which the alcoholic ferment has need for its existence, while the lactic and butyric ferments develop in the absence of air. Aëration in the vat itself, although sometimes necessary, is inadvisable for fear of acetification or loss of alcohol. The accumulation of carbonic acid gas also acts injuriously and should be obviated by efficient ventilation. A want of aëration also causes the nitrous fermentation, recognised by a characteristic odour.

The most favourable temperature for the alcoholic fermentation is 27° to 28° C. Above 30° C. the lactic and butyric ferments develop rapidly, the acidity increases and the proper attenuation is not reached, entailing a loss of sugar.

The acidity of the juice run into the vats varies in different distilleries, but should range between 2 grms. and 3 grms. of $H_2SO_4$ per litre. It will depend on the nature of the beetroot, the method of extraction of the juice, its aëration, the density of the wash and the special nature of the yeast used. The acidity being due partly to the wash and partly to the sulphuric acid used, it is important to maintain a certain proportion between the two. The best conditions have been found to be when the acid added represents two-thirds of the total acidity.

The yeast should be frequently and regularly renewed. A good fermentation should be complete without sensible increase of acidity, a rise of 0·20 per litre being considered normal, and the specific gravity should be about 0·002 to 0·003 below that of the wash at the same temperature.

Although it is easy to correct an insufficient acidity, it is not so easy to find a remedy when the acidity is too high. Too high an acidity may arise from three causes. (1) from an excess of acid added at maceration; (2) from an insufficient amount of acid added, which has engendered secondary fermentations with production of lactic and butyric acid; (3) from too great a concentration of the wash.

Experience has shown that in a normal fermentation, that is, when there is no production of lactic and butyric acids, the mineral acidity represents about two-thirds of the total. This mineral acidity is recognised on titrating with potash by the blue colour which mineral acids give to paper coloured with Congo Red 4 R, 1 grm. per litre, or by the dark colour which the colouring matter of the beetroot itself shows as soon as all mineral acid is neutralised. The total acidity is found by litmus paper. The liquids must be filtered clear before testing.

If it be found that the mineral acidity is too high, too much has been added; if too low, too little, when the organic acidity will be excessive. If both acidities are too high, the wash has been over-concentrated and it should be thrown away and operations commenced afresh with water.—L. J. de W.

### *Wine Ferments, Contribution to the Biology of.* J. A. Cordier. Comptes Rend. 1898, **127**, [17], 628—630.

By exposing plates of must-jelose, at a height of 50 cm. from the ground, in the Pommery vineyards, the author obtained up to October 12th, colonies of moulds only, and no saccharomyces or dematium. The vintage in these districts begins from October 12th to October 18th; and after October 12th, mould fungi were still in the majority, but colonies of dematium and a few of true saccharomyces were also found. The deposition of micro-organisms on the grapes must be mainly effected by air transport, as insect life at that period of the year is scarce. Up to the time of the vintage, *Dematium pullulans* is present in large quantities on all parts of the vine in the filamentous form, but when the grapes are ripe the thallus breaks up and is scattered.

The true saccharomyces form a very small minority of the micro-organisms on fruits, but they increase as the fruits ripen; the same species of organisms are found on crops of other fruits, such as plums and cherries, as are afterwards found on the grapes of the same neighbourhood at the time of vintage.

*Dematium pullulans* is present in the flowers of the vine and imparts to its culture media the vanilla odour of flowering vines. In old cultures, the author has observed an intense black colour, accompanied by the formation of true perithecia through entangling of the filaments. Glucose appears to play the principal part in causing the breaking up of the thallus of dematium, and although these organisms are not true ferments of sugar they only form the collar-shaped bunches, described by Jörgensen, in sweet grape musts.

Saccharomyces pass unchanged through the intestinal canals of insects and animals and the author has isolated the characteristic ferments of the grapes, on which dormice have fed, from their intestinal contents.—J. F. B.

### *Hop-Cones, The Distribution of the Valuable Constituents in.* T. Remy. Wochensch. für Brauerei, 1898, **15**, [44] 593—594.

The mechanical analysis was effected by picking out the different parts by hand, and the percentage of stalks and foliage-leaves, flower-axes, seeds and petals, including perigona and lupulin-glands, was determined in samples of three representative German hops. The valuable hops constituted 67·4 per cent. of ordinary Altmark hops, 88·1 per cent. of fine Hallertauer and 86·0 per cent. of Neutomischel hops, the alcoholic-ethereal extracts being 11·80, 20·45, and 18·24 per cent respectively. Only the first-mentioned sample contained any notable quantity of seeds, of which 18·2 per cent. were found. Chemical determinations were then made of all the important constituents in each of the morphological parts separately *viz.*, in the lupulin-glands, in the flower-leaves (which could not be entirely freed from lupulin-glands), in the axes and stalks, and in the seeds, where present.

The matter extracted by ether, consisting mainly of resins mixed with a small proportion of fat and waxes, was by far the highest in the case of the lupulin-glands, constituting 63·70 per cent. of their dry weight. A large proportion (25 per cent.) was extracted from the seeds, but this was unimportant, and consisted entirely of oil; the petals yielded 11—12·6 per cent. of ethereal extract, part of which must be due to the admixed lupulin glands, which could not be perfectly separated mechanically.

The tannin was chiefly concentrated in the petals of the cones, which contained 2·1—3·8 per cent.; the lupulin contained about 1 per cent. of tannin, partly due to admixed particles of petals. The greatest proportion of nitrogen occurred in the axes and stalks (2·2—2·7 per cent.), less in the petals (1·5—2·3 per cent.), and least in the lupulin-glands, (1·1—1·5 per cent.); when seeds were present, however, they contained a much greater proportion than any other part (5·5 per cent.), doubtless in the form of albuminoid reserve material.

When the proportion of nitrogenous compounds, soluble in boiling water, was determined the relations changed. It was found that 60 per cent. of the total nitrogen in the lupulin was soluble, 40 per cent. of that in the axes and stalks, and 29 per cent. of that in the petals, whilst only 5·6 per cent. of the nitrogen in the seeds was soluble. As regards the mineral constituents, the author found that the phosphoric acid was distributed in the same relations as the total nitrogen, and the same was true of the potash, except in the seeds, in which it was low.

These investigations are of interest, since machines are now obtainable which can approximately separate the different morphological parts of hops, and it is possible that large, coarse hops, which are now of little value for fine beers, may, when freed from stalks, axes, and seeds, be quite fit for mixing with the finer, smaller varieties.

—J. F. B.

### *Wines, Disappearance of Nitric Acid in; to which Water containing Nitrates has been added.* W. Seifert. Oesterr. Chem. Zeit. **1**, 285—287; Chem. Centr. 1898, **2**, [15], 833.

Whilst it has been known for a long time that on the addition of water containing nitrates to wine, after some time, nitric acid can no longer be detected, views are conflicting as to the cause of this phenomenon. The author's experiments on the subject show that the nitrates present are not destroyed or altered by the fermenting action of pure yeast in the grape must nor by the action of pure cultures of two kinds of *Mycoderma vini* (mould fungus) on the wine. The main cause of the phenomenon appears to be the acetic bacteria. In order to determine the conditions under which denitrification takes place, sterile wine, infected with acetic bacteria was allowed to stand until a fine covering of bacteria had formed on the surface; at this stage, nitric acid could still be distinctly detected. The liquid was then agitated, so that the bacteria fell to the bottom, the vessel closed with a sterile cork, and allowed to stand at room temperature. After three weeks the liquid was quite clear, the amount of bacteria and free acid had increased, whilst nitric acid had completely disappeared. It appears therefore that complete denitrification is effected by acetic bacteria immersed in the liquid, with exclusion of air.—A. S.

*Taste, The Influence of Temperature on the Sense of.* M. Mendelsohn. Zeits. ges. Kohlensäure-Ind. 1898, [18], through Wochensch. fur Brauerei, 1898, **15**, 499—500.

The author advocates the tasting of beers between the limits of temperature of 10° and 20° C., any divergence, either above or below these temperatures, tending to a proportionate diminution of the faculty of appreciation of flavour.—J. F. B.

*Wort and Beer, Colour Determination of.* A. Reichard. Zeits. f. d. ges. Brau., **21**, [26], 335—339; [27], 351—355; [28], 368—372.

The author proposes to estimate the colour shade and depth of worts and beers by the aid of four standard solutions of iodine in potassium iodide, No. I., containing 18 grms.; II., 25; III., 40; and IV., 71 grms. of potassium iodide per litre, the last-named corresponding to the palest worts.

In carrying out the method, the shade and depth are determined by comparing 100 c.c. of the test liquid, in the usual test glasses, with an equal quantity of water coloured by the addition of the most nearly similar standard solution run in from a burette. The quantity of iodine solution consumed, per 100 c.c. of undiluted wort, &c., is the measure of the depth, and the number of the solution a gauge of the shade, of the colour.—C. S.

*Samshu.* F. Brown. Pharm. J. 1898, **69**, (1479), 460.

The term samshu is, strictly speaking, limited to the three spirits distilled from rice known as Leu Pun Chau, Sheung Ching Chau, and Sam Ching Chau. The term is often loosely applied to all spirits of Chinese production, in which one of the above may be only an ingredient, or even to a spirit obtained from molasses, Fa Chau. Of the three varieties of samshu, Leu Pun Chau (half materials liquor) is the most largely consumed. To produce it, equal quantities of rice and water are boiled together for half an hour to soften the grains, which are then spread on an inclined tray, so that the water drains off. The cooked rice is then put into pots, together with the drained liquor and a little water, and a ferment "Chau Pang." This is composed of rice, bean flour, red earth, and leaves, stated to be cassia leaves. It occurs in dry flat cakes about 7 ins. square and 1 in. thick. It was found by the author to contain 44 per cent. of ash. After the addition of this substance, fermentation is allowed to proceed for 23 to 28 days, when distillation is conducted in a primitive pot still, of which an illustration is given. To produce 200 catties of Leu Pun Chau, 180 catties of rice and 30 catties of Chau Pung are requisite. Sheung Ching Chau is a little stronger than Leu Pun Chau. It is prepared by distilling a certain quantity of the fermented liquid with Leu Pun Chau, drawing over three times as much distillate, as of Leu Pun Chau added. Sam Ching Chau is the strongest samshu; it is prepared like Sheung Ching Chau, but three times as much Leu Pun Chau is added, and only an equal volume distilled over. The composition of these liquors is given as follows:—

| Description. | Strength of Sample. | Total Solids Dried at 100° C. in 100 Fluid Parts. | In 100 Fluid Parts of Proof Strength. | | |
|---|---|---|---|---|---|
| | | | Free Acid as Acetic. | Compound Ethers as Acetic Ether. | Higher Alcohols. |
| Leu Pau Chau, 1 | 58·7 u.p. | 0·015 | 0·279 | 0·230 | 0·032 |
| „ „ 2 | 50·9 u.p. | 0·006 | 0·127 | 0·305 | 0·034 |
| Sheung Ching Chau, 1 | 53·5 u.p. | 0·035 | 0·217 | 0·231 | 0·062 |
| „ „ 2 | 42·4 u.p. | 0·008 | 0·123 | 0·321 | 0·035 |
| Sam Ching Chau, 1 | 15·0 u.p. | 0·012 | 0·078 | 0·242 | 0·034 |
| „ „ 2 | 22·6 u.p. | 0·018 | 0·073 | 0·205 | 0·038 |

All the samples contain a faint trace of furfuraldehyde.

Leu Pun Chau is the staple beverage, being preferred on account of its lesser alcoholic strength. Samshu is never diluted. When Sheung Ching Chau or Sam Ching Chau are drunk, very small sips are taken.

Samshu is a colourless liquid of characteristic odour. Leu Pun Chau and Sheung Ching Chau are usually slightly opalescent, but the turbidity disappears when alcohol is added.

By mixing samshu with other ingredients, a number of well-known Chinese beverages are prepared. Thus Ching Mui Chau is composed of Leu Pun Chau, sour plums, and sugar. Mau Mui Chau from *Triticum repens*, Leu Pun Chau, and sugar. Ka Pi Chau from the bark Ka Pi, Leu Pun Chau, and sugar. Ming Mun Chau from limes, Leu Pun Chau and sugar. Muh Kwa Chau from papaws, Leu Pun Chau, and sugar.

In addition to samshu, several distilleries make a liquor, Fa Chau, from molasses and the same wort. Two samples of this were found to be 41·5 and 37·9 u.p. respectively. Fa Chau is used for making several of the wines in which Leu Pun Chau is employed. Shut Li Chau is made from Fa Chau and Tientsin pears.

No Mei Chau is a liquid of varied composition. Fa Chau is used for it together with No Mei, or full-grown rice; or it is simply sweetened. Leu Pun Chau is prepared from full-grown rice. Shan Kut Chau is prepared from Shau Kut, a small hard, bitter orange, and Fa Chau. All the distillers make sauce by converting these alcoholic liquors into vinegar and adding condiments, and one distiller makes vinegar for the preparation of white lead, as well as for sauce making.—J. O. B.

*Yeast, The Nutrition of.* A. L. Stern. Proc. Chem. Soc. 1898, [198], 182.

This investigation was undertaken to determine the amount of inorganic and nitrogenous nutriment required to produce the largest crop of yeast, the greatest assimilation of nitrogen, and the most complete fermentation in a fixed time. During the course of the investigation, other points had to be considered, of which the most important was the part played by sulphur compounds on yeast nutrition. The earlier work of Hayduck, who employed a different variety of yeast and a different variety of sugar, is referred to (*Zeits. für Spiritusind.* 1881, 173).

The yeast employed was a pure form obtained from a Burton pitching yeast, the sugar was *d*-glucose, the nitrogenous nutriment asparagine, and the inorganic nutriment of two kinds; the first prepared from a yeast ash and free from sulphur, the second a mixture of potassium phosphate magnesium sulphate, and calcium sulphate. Each experiment consisted in fermenting 500 c.c. of a 10 per cent. solution of the sugar, to which varying amounts of the nitrogenous and inorganic nutriment had been added.

It is shown that sulphur is an essential constituent of yeast nutriment, and that, in the absence of other more suitable forms of sulphur nutriment, sulphates can supply the yeast with this element, a portion of the sulphate being invariably reduced to sulphuretted hydrogen. Attempts were made to find a substance of known constitution which could supply sulphur to yeast without the evolution of sulphuretted hydrogen, but without success.

It has been asserted that iron is an essential constituent of yeast nutriment, but no evidence could be obtained in support of this statement.

The author has examined the effect of variation in the amount of (1) nitrogen nutriment and (2) inorganic nutriment respectively on the amount of nitrogen assimilated, the percentage of nitrogen nutriment assimilated, the percentage of sugar remaining unfermented, and the weight of the yeast crop, and draws the following conclusion: any increase of nutriment beyond a definite limit will not materially increase the amount of nitrogen assimilated by the yeast, the percentage of nitrogen in the yeast, the weight of the yeast, or the amount of sugar fermented. This limit, called the normal supply, is the largest quantity that the yeast can assimilate under the conditions employed in the experiments, and is approximately 0·025 grm. of inorganic nutriment per 100 c.c., and the same weight of nitrogen supplied as asparagine.

*Wine, Determination of Total Acidity in.* E. Sellier and A. Vivien. Chem. Centr. 1898, **2**, [16], 873.

*See under* XXIII., *page* 1193.

*Proteids of Beer Worts and other Albuminous Liquids, Quantitative Estimation of the Various Types of.* H. Schjerning. Zeits. f. anal. Chem. 1898, **31**, [7], 413.

*See under* XXIII., *page* 1191.

*Malt, The Analysis of.* O. Reinke. Wochenschr. für Brau. 1898, **15**, [42], 555.

*See under* XXIII., *page* 1193.

*Hops, Chemical Analysis in the Commercial Valuation of.* T. Remy. Wochenschr. fur Brau. 1898, **15**, [41, 42, and 43].

*See under* XXIII., *page* 1192.

*Malt, The Analysis of, and Proposals for Uniformity in.* L. Aubry. Wochenschr für Brau. 1898, **15**, 497.

*See under* XXIII., *page* 1192.

*"Saccharin" in Beer, Detection of.* Wochenschr. für Brau. 1898, **15**, [45], 608.

*See under* XXIII., *page* 1188.

## PATENTS.

*Micro-Organisms, A Process for Obtaining the Liquid Cell Contents of, in an Unaltered State.* E. Buchner, Tubingen. Ger. Pat. 99,508, Jan. 12, 1898.

The patentee claims the process for obtaining the liquid cell contents of micro-organisms, in such a form that their concentration and properties are practically the same as in the living cell, by triturating the organisms with some insoluble, indifferent substance, such as sand, powdered glass, or kieselguhr, and by extracting the liquor by very high pressures.

To obtain the extract from beer yeast, 1 kilo. of washed yeast, freed from adherent moisture by pressure, is mixed with an equal weight of quartz sand and 250 grms. of kieselguhr, and carefully triturated until the whole mass is moist and plastic. 100 grms. of water are then added, and the mixture is wrapped in a cloth and the liquor squeezed out under a pressure of 400—500 atmospheres. The cake is again triturated with the addition of 50 grms. of water, and again squeezed under the same pressure. In this manner about 500 c.c. of pressed extract are obtained, which, after shaking with kieselguhr and filtering through a kieselguhr candle, has a faintly opalescent, clear yellow colour.

The process may also be applied to the preparation of toxic extracts from various bacilli, such as the tubercle bacillus and *B. pyocyaneus*.—J. F. B.

*Beer, Impts. in and relating to Preserving.* J. F. H. Grönwald, Berlin, Germany. Eng. Pat. 26,532, Nov. 13, 1897.

The patentee claims to have established, by experiments, that the deterioration in flavour, which is frequently observed when beer is pasteurised, is due to some change—probably an oxidative one—in certain of its volatile constituents; and that such deterioration may be avoided, provided these volatile substances be removed prior to sterilisation. For this purpose the beer may be warmed *in vacuo*, or scrubbed with an indifferent gas ($CO_2$). The latter plan is preferred; and a combined purifier and steriliser is figured and described, for carrying it out in practice. The apparatus consists of a closed cylindrical vessel, mounted horizontally, provided with a jacket for heating and cooling purposes, and fitted with a rotating stirrer. In this the beer is warmed to 20°—30° C., and thoroughly agitated in an atmosphere of carbonic acid under slight pressure, the gas being blown off at intervals, and replaced by fresh, until the beer is sufficiently purified. The final pasteurisation of the beer is effected in the usual way.

—H. T. P.

*Fires in Breweries, Distilleries, and other like Premises; An Improved Method and Apparatus for the Prevention and Extinction of.* H. M. Kettelwell, London. Eng. Pat. 29,157, Dec. 9, 1897.

This invention relates to a process and apparatus for utilising the carbonic acid produced during fermentation in breweries and distilleries as a preventive against fires, and, especially, malt-dust explosions. To this end the carbonic acid is collected, in the case of "rounds" and "squares," by a fan-shaped hopper, placed just below the sill of the manhole, and into which the gas overflows; or, when a parachute is employed, by a pipe passing through its centre. The gas is then allowed to flow, by its own gravity, through pipes to the rooms or spaces to be protected; or, if required on a higher level, is forced up by a fan or pump.—H. T. P.

*Beer, Ale, and Porter, A Process for Recovering from Vat Bottoms, Vat Sweepings, or Vat Deposits generally.* A. F. Watson, Dublin. Eng. Pat. 4943, March 1, 1898.

According to this process, the bottoms from tuns, &c. are heated to 105°—212° F. in a closed vessel for about 10 minutes, then rapidly cooled. By this means, it is claimed, the suspended matter—originally bulky and light—is caused to coagulate and settle down in a compact mass, from which the supernatant beer may be drawn off.

—H. T. P.

*Condensation of Vapours for Distilling and like Purposes, Impts. in Apparatus for the.* C. H. Cribb, London. Eng. Pat. 1496, Jan. 19, 1898.

The improved condenser is of the vertical type, and consists of a wide inner tube sealed at its lower end, and surrounded by a close-fitting jacket, to which it is joined at the top, either by fusion, or preferably by a rubber or screw joint. The jacket is drawn out below to a tube, from which the condensed liquid escapes. The narrow annular space between the two tubes is utilised as condensing chamber, the vapour being passed into it by a suitably fixed side-piece. The cooling water enters the hollow cavity of the inner tube, flows over the top of the condenser, and runs down its exterior surface, being finally caught in a shallow cup surrounding the outlet of the condenser, and fitted with a waste-pipe. To ensure even distribution of the cooling water over the outside of the condenser, the latter may be wrapped round with string or muslin, and provided with a perforated, cover. Moreover, the condensing chamber may be converted into a spiral channel, and the efficiency of the apparatus much increased, by filling up the annular space between the two tubes with a few turns of wire, thick enough to bridge over the gap.

The condenser is claimed to be very efficient and compact, readily adaptable for distillation under reduced pressure, and easily got at for cleaning purposes.—H. T. P.

## XVIII.—FOODS; SANITATION, WATER PURIFICATION, & DISINFECTANTS.

### (*A.*)—FOODS.

*Margarine, Denaturing.* — Mainsbrecq. Bull. Assoc. Belge des Chim. **12**, [5 and 6], 185—187.

The author finds that, contrary to existing opinion, the potato starch employed for denaturing margarine cannot be entirely removed therefrom by subsequent washing in a butter-worker, provided the starch has been used in a dry state, mixed with oil, and added to the fat directly after churning; but can still be detected by examination under the polariscope or by the iodine reaction, the fat, for this latter purpose, being either previously dissolved out by ether, or treated with an ethereal solution of iodine. He therefore concludes that this starch affords a suitable means for attaining the object in view.—C. S.

*Mangels, Changes in, during Storage.* T. B. Wood. J. Roy. Ag. Soc. Eng. 1898, **9**, [3], 555—557.

The nitrogen present in mangel juice in different forms—ammonia, nitrates, amides, proteids, and peptones—was determined at intervals, commencing Nov. 1, 1897, and ending May 1, 1898. It was found that during storage, the nitrates, which are not only useless as food, but injurious, diminish in quantity, whilst there is an increase in the proteids, and a still greater increase in amides. The results thus lend support to the generally accepted opinion that mangels improve as food for stock when kept until after Christmas.—N. H. J. M.

*Dried Fruit, German, Amount of Zinc in.* P. Kulisch. Zeits. angew. Chem. 1898 [44], 1015—1016.

According to the author, the zinc so frequently met with in American dried fruit is due not to the addition of zinc salts, but to the action of the acid juice of the fruit on the wire network of the trays in the drying apparatus. On continued use the trays become coated with a hard crust of dried juice which prevents the fruit from coming into contact with the metal, and in such dried fruit the amount of zinc is naturally less. Since all the more modern fruit-drying apparatus in Germany is constructed on the American plan with zinc wire trays, the author has examined different kinds of German dried fruit, in order to determine to what extent they were contaminated with the metal. The quantities of zinc found in 100 grms. of fruit (with the usual commercial amount of water) were :—Apple rings, *a* 0·031, *b* 0·021 grm.; apple chips, *a* 0·023, *b* 0·027 grm.; pear slices, *a* 0·020, *b* 0·026 grm. All the samples thus contained a percentage of zinc not much less than the mean found in American apple rings.—C. A. M.

*Glycogen, The Supposed Transformation of Fat into, in the Animal Body.* Berthelot. Comptes Rend. **127**, [15], 491—496.

M. Bouchard has shown that animals may, under certain conditions, increase in weight temporarily without taking food; a fact which he has attributed, no doubt rightly, to an absorption of oxygen not compensated by the excretion of gaseous matters during the period in question. It has been asserted that this absorption of oxygen occurs during the conversion of the fats of the body into glycogen. The author points out that there is no direct evidence that this change goes on; that to account by it for the observed amount of increase in weight would require the formation of a wholly abnormal amount of glycogen; and that the production of sugars by the oxidation of fats is improbable, inasmuch as in most of the reactions known to chemists, fats are produced at the expense of sugars. He suggests that the fixation of oxygen more probably arises as a stage in the breaking up of albuminoids, with the formation of Maly's oxyproteine or Schutzenberger's glucoproteine, or from the oxidation of fats or fatty acids, in such ways as, for example, the change of fatty acids into acids of the oleic or oxalic series. He also notes that the increase of weight in the human subject during one hour, observed by M. Bouchard, is equal to the total amount of oxygen absorbed in that period under normal conditions; so that in the case under observation there must have been an abnormally high absorption of oxygen and an abnormally low excretion of carbon dioxide.—J. T. D.

*Butter and other Fats, Relation between Specific Gravity and Insoluble Fatty Acids of.* N. Leonard. Analyst, 1898, **23**, [272], 282.

*See under* XII., *page* 1156.

### PATENTS.

*Preserving Meat, Process and Apparatus for.* L. Wacker, Munich, Germany. Eng. Pat. 22,686, Oct. 4, 1897.

The meat to be preserved is placed in a vessel provided with a movable perforated partition and a lid, the edges of which reach down to below the centre of the vessel. The partition rests on a rim caused by the narrowing of the lower portion of the vessel. A saturated solution of salt in vinegar is placed in the lower part, and the meat is allowed to soak in this solution for some time. The partition and lid are then put into position and the vinegar is boiled. On reversing the vessel the meat remains on the partition and the liquid passes through into the upper (but now the lower) compartment. On cooling, a partial vacuum is created, the liquid making a hermetical joint between the lid and the sides of the vessel.—W. P. S.

*Milk for Food, Impts. in the Preparation of.* D. H. Arslanian and B. H. Arslanian, London. Eng. Pat. 24,279, Oct. 20, 1897.

Milk which has been boiled twice is coagulated by the addition of rennet or lemon-juice. It is then strained through flannel and allowed to stand at a warm temperature in a closed vessel for two hours, when the coagulated portion acquires a creamy consistency and settles to the bottom. After drawing off the supernatant liquid, this coagulum may be used for the treatment of more milk in the place of rennet.

For the production of a thick cream, the milk is well boiled, let cool for six hours, and then placed in a vessel which is heated at the sides only. After being heated for one hour it is allowed to cool for 24 hours, when a thick cream may be removed.—W. P. S.

*Tea Essences, Impts. in and relating to.* R. R. Bell, Glasgow, and A. C. Butler, Glasgow. Eng. Pat. 26,292, Nov. 11, 1897.

According to this invention, tea is infused in a boiling solution of sodium bicarbonate, glycerin, salicylic acid, and sugar in water. The extract is then strained off and filled into bottles.—W. P. S.

*Cooling Liquids* [*Milk, Cream, &c.*], *Impts. in Apparatus for.* J. T. Bentley, Ohio, U.S.A. Eng. Pat. 26,525, Nov. 13, 1897.

The apparatus consists of a receptacle containing a coiled pipe, which is connected at its lower end to suitable outlet pipes, and at its upper end to a pump and suction tube through which the liquid to be cooled is supplied. The inner face of the coil is lined with a perforated screen, within which the ice or cooling mixture is placed and stirred by a suitable agitator. Means are also provided for admitting steam and water for cleansing and sterilising the pipes.—R. A.

*Preserving Meat and other Alimentary Substances, New or Improved Process for.* W. Lauwer, Berne, Switzerland. Eng. Pat. 27,617, Nov. 24, 1897.

The meat is plunged into a boiling 5 per cent. solution of sodium chloride, and is then immersed in a solution consisting of gelatin (20 parts), starch (5 parts), sodium chloride (5 parts), and water (70 parts). It is removed from this liquid, allowed to drain and dried; afterwards the meat is again immersed, and whilst wet is placed in a chamber where it is subjected to the vapours of formic aldehyde, the object being to form an impervious coating or skin over the meat. When so treated, the meat may be stored in stout cotton bags, the necks of which contain a small bag full of cotton soaked with formic aldehyde, the neck of the outer sack being tied round the smaller antiseptic bag.—W. P. S.

*Sterilising Meat in the Raw State, Impts. in Apparatus for.* H. H. Lake, London. From A. Moyet, Lyons. France. Eng. Pat. 28,379, Dec. 1, 1897.

The meat is cut into strips and steeped for two hours in a liquid consisting of white willow bark (10 grms.), poplar bark (15 grms.), flowers of arnica (10 grms.), sea salt (100 grms.), tannin (½ grm.), alcohol, 95° (50 grms.), mustard oil (2 drops), beech creosote (1 drop), oil of cinnamon (10 drops), oil of citron (6 drops), oil of cloves (6 drops), and spring or river water (1 litre). The strips of meat are then chopped into small pieces and dried for three weeks. The meat is now pasteurised in a special steam-heated apparatus and finally grated quite fine, sifted, and stored in closed vessels. 1 grm. of vanillin to 1 kilo. of meat may be added to improve the flavour.—W. P. S.

*Bread and Biscuit Baking, and in the Manufacture of an Improved Flour and Meal for Use therein, or for Pastry, Infants and Invalids' Food Purposes; Impts. in.* J. T. O'Callaghan, London. Eng. Pat. 17,726, Aug. 17, 1898.

The improvements claimed are for processes for making "digestive whitey-brown meal," "digestive brown meal," "malt milk flour," "malt milk meal," "malt white flour," and "malt meal," by malting rice, white maize, and yellow maize. The latter are ground and dressed, and mixed with a boiled extract of malt, sugar of malt, lime water, vegetable fat, and salt. In some cases phosphoric acid and potassium bicarbonate may be added to this extract. The product obtained is blended with flour or whole wheat meal.
—W. P. S.

*Margarine or Artificial Butter, Process for the Production of.* M. Poppe, Bielefeld, Germany. Eng. Pat. 18,500, Oct. 15, 1898.

The patentee employs a cultivation liquid obtained from pure cultures of bacilli (bacillus aromaticus, or other suitable bacilli). This liquid is used in place of the liquids (buttermilk, &c.) usually employed in the manufacture of margarine, and gives to the article the peculiar "butter" aroma and taste.—W. P. S.

## (*B.*)—SANITATION; WATER PURIFICATION.

*Sewerage, The Liernur System of, in Amsterdam.* Glaser's Annalen fur Gew. und Bauw. March, 1898, 93.

The town is divided into 37 districts for treatment by the Liernur system, each district being provided with a reservoir, usually situated at the intersection of two main streets, and connected with eight mains draining the houses from either side of the streets. The sewage is brought to the central station by opening valves, the reservoirs being coupled with vacuum cylinders, and is then lifted pneumatically to high-level water tanks, and run into settling basins. The solid matter is converted into manure, and the liquid is treated with lime, the effluent from which, after settling, is distilled for ammonia, to be fixed by sulphuric acid. The volume treated weekly is 417,000 gallons, one-eighth consisting of solids, one-eighth of lime sludge, and three-fourths of effluent. The annual cost of treatment per head, and the cost and details of construction, are fully given.—A. W.

*Filters, Impt. in.* W. Dalton, Blackpool, Lancashire. Eng. Pat. 22,779, Oct. 5, 1897.

The object of the invention is to secure perfect filtration, and the destruction of microbes and animalculæ. The filter, which may be made of metal, consists of one or more shells containing carbon, or other filtering material, through which the liquid is forced after it has passed through a gauze passage. Provision is made for the introduction of various substances, such as limestone (antidote to lead poisoning), iron, or potassium permanganate, and for the automatic removal of deposits.—N. H. J. M.

### PATENTS.

*Sewage or other Liquids, A New or Improved System of Purifying by Aërated Filtration, and Apparatus therefor.* R. B. Duncan, Newcastle-upon-Tyne. Eng. Pat. 24,180, Oct. 19, 1897.

An aërating filter, which may be used continuously. It consists of vertical columns, on a concrete foundation, connected by horizontal bars, which support four filtering boxes, one over the other. The filtering material is crushed clinker, coke breeze, burnt ballast, sand or gravel, &c. Sewage, which has preferably first passed through a covered tank, is conveyed by means of a perforated channel pipe to the upper filtering box, from which it passes successively, in fine streams, to the lower boxes, and finally to the concrete floor.

The arrangement ensures abundant aëration to maintain the vitality of the nitrifying organisms in each layer.
—N. H. J. M.

*Noxious and other Vapours, New and Improved Process for the Effective Removal of, and in Appliances for Use in connection therewith.* J. Rudman and H. J. G. Rudman, Bristol. Eng. Pat. 23,913, Oct. 18, 1897.

The vessel is provided with a cover connected with a wide tube, which, after ascending to a suitable height, bends downwards. At some distance from the bend, steam is injected. The resulting partial vacuum causes the fumes produced in the vessel to be drawn through the tube and to pass eventually to the underground drain-pipe, with which it is connected. If desired, a stream of cold water may be inserted in the tube to condense the steam.

Instead of being passed into the drains, the fumes, mixed with steam, may be made to pass through a coil, placed in a cistern of cold water, into a covered tank; any vapour still remaining is conveyed by an outlet pipe into a chimney-shaft.—N. H. J. M.

*High Temperature Furnace or Destructor, for the Purpose of Burning House and Town Refuse, and other Combustible Substances.* W. Saint, Manchester. Eng. Pat. 26,466, Nov. 13, 1897.

*See under* I., *page* 1129.

*Heating, Softening, and Purifying Water; Impts. in and Apparatus therefor.* H. McPhail, Wakefield. Eng. Pat. 27,026, Nov. 18, 1897.

Improvements are devised in feed-water boilers of the kind described in Eng. Pats. 10,153, 1895 and 5391, 1896. A vertical cylindrical casing is arranged, into the top of which, water is sprayed, and down which it flows, passing over a number of superposed dishes or trays, provided with central openings and covers, arranged within the upper or steam space of the cylinder. From the bottom tray the hot water is collected by a central pipe, which carries the water nearly to the base of the cylinder. The water then rises up the cylinder to the hot water outlet, which is situated some little way below the lowest tray. The precipitated mud and salts collect in a drain pipe, from which they are blown out. The lower, or water, part of the cylinder is heated by a number of upwardly-curved steam-pipes, communicating with a steam-box on each side, one in connection with the steam-generator, the other with a pipe leading the steam to the top of the cylinder.—R. S.

## (*C.*)—DISINFECTANTS.

*Creasote, Notes on Some Commercial Specimens of.* R. Thal. Chem. Zeit. 1898, **22**, [90], 961—962.

The Russian military pharmacopœia (1896) requires that all creasote supplied to the military hospitals shall distil over completely between 205° and 220° C. The results of the examination of four samples submitted by the manufacturers to the Russian military authorities and analysed by the author showed that none of them completely complied with this test. The specific gravity at 15° C. of the four samples, were Lambiotte Frères (Brussels), 1·106; Merck, 1·077; de Haën, 1·081; and Köhler and Co. (Moscow), 1·077. Fractional distillation gave the following results:—

| Creasote from | Commenced to Boil. | Distilled over | | Boiling above 220° C. (by difference). |
|---|---|---|---|---|
| | | To 205° C. | From 205° to 220° C. | |
| | ° C. | Per Cent. by Weight. | | |
| Lambiotte Frères | 198 | 31·07 | 66·58 | 2·34 |
| Merck .......... | 198 | 10·14 | 81·50 | 8·36 |
| de Haën ........ | 198 | 23·84 | 51·36 | 24·80 |
| Köhler and Co. . | 198 | 48·70 | 46·29 | 5·01 |

On testing the fractions distilling below 205° with an alcoholic solution of potash (1 : 5 of 90 per cent. alcohol), the difference in the rapidity with which these solidified showed that they contained different amounts of guaiacol, the distillate from Lambiotte's creasote being considerably the richest in this respect. On mixing it with an equal volume of 33 per cent. aqueous soda, it immediately yielded a white crystalline mass, which did not happen in the case of the distillates of the other three samples. After a considerable time the distillate of de Haën's creasote became partially solid, but the other two creasotes remained liquid. Moreover, on mixing 1 c.c. of the original creasote with 2·5 c.c. of a 33 per cent. aqueous solution of soda, Lambiotte's sample immediately solidified to a white crystalline mass, whilst the others only became solid after some time, and the mass had a yellow colour. None of the creasotes became solid when treated with soda solution ranging in strength from 15 to 32 per cent. In the light of these results the author considers that the requirements of the German Imperial Pharmacopœia (*viz.*, that creasote shall distil for the most part between 205° and 220° C.) are insufficient. Basing his conclusions on the properties of the sample submitted by Lambiotte frères, which was regarded as the best, he advocates the following tests for creasote:—1. On shaking 1 c.c. of the creasote with 2·5 c.c. of 33 per cent. aqueous soda, the mixture should immediately solidify to a white crystalline mass, which should dissolve without turbidity in 50 c.c. of water. 2. It should boil at 198° C., contain only traces of substances boiling below 200° C., and should distil almost completely between 200° and 220°, leaving at most 3 per cent. of substances boiling above 220° C. The mixture of the constituents distilling below 205° should immediately solidify to a white crystalline mass when mixed with an equal volume of 33 per cent. aqueous soda.—C. A. M.

*Formaldehyde and the Work of Public Disinfection.* F. Abba and A. Rondelli. Zeits. für Hygiene, **27**, 1898, 49.

The authors have experimented with the spores of anthrax, diphtheria, typhoid, &c., in closed apartments, both small (½ cub. metre) and large, and also with an invalid carriage. Their conclusions are as follows:—That the disinfecting power of formaldehyde is increased by a rise in temperature and in the presence of dry air; that the gaseous aldehyde has very little penetrating power; and that it does not injure clothes, paper, photographs, &c., nor affect colours, except certain tar derivatives which change tints in a similar way to fresh flowers. Certain precautions are stipulated in the general use of formaldehyde as a disinfectant. It has advantages, but it has also certain drawbacks, and the authors do not consider it preferable to corrosive sublimate or steam when they can be obtained and employed, although where these means are inapplicable formaldehyde may be used in public disinfecting stations with advantage.—A. W.

*Formaldehyde as a Disinfectant.* K. Walter. Zeits. für Hygiene, **26**, 1897, 454.

The results of a number of experiments with Trillat's apparatus with formaldehyde, under various conditions, on certain spores, &c., are given, together with arguments for and against the methods put forward for the use of this material as a disinfectant. The author has arrived at the following conclusions:—That the complete disinfection of a room with all its contents cannot be carried out in one operation, however the formaldehyde may be generated; that, in face of this, the use of aldehyde is nevertheless of importance in a complete system of disinfection; and, that the use of gaseous formaldehyde cannot be considered efficacious in the disinfection of wall surfaces, although it is possible to thoroughly disinfect garments, &c., by its vapour, without injury to the material.—A. W.

### PATENT.

*Disinfecting Process by Means of Methylaldehyde [Formaldehyde] or Polymeric Methylaldehydes, an Improved; and Method of Employing Same.* G. Krell, Husten, and M. Elb, Dresden, Germany. Eng. Pat. 28,164, Nov. 30, 1897.

The process claimed consists in subjecting formaldehyde or its polymers to the heat of a "glimmering mass" (carbon impregnated with nitrates) at a low temperature, whereby the aldehyde is volatilised without ignition.—A. C. W.

## XIX.—PAPER, PASTEBOARD, Etc.

*Sulphite Cellulose Waste Liquors, Disposal of.* Papier Zeit. 1898, **23**, [93], 3527.

The offer of a sum of 500*l.* for a process of rendering harmless the waste liquors of the paper and cellulose factories of Unterkochen and Wolfach resulted in 83 applications, of which 75 were German, the rest being from America, England, Sweden and Norway, and Austria-Hungary. Besides the 500*l.* given for the process selected, a sum of 25*l.* was given to the inventors of the next best process; and three other processes were considered worthy of attention. In very many cases suggestions were made which had already been tried and found to be impracticable. Many of the proposed processes failed to take into account the local conditions of the factories; others were obviously useless, or else introduced other inconveniences.

L. J. Dorenfeld, of Rheindürkheim a. Rh., received the first prize; Drs. Appel and Max Buchner, of the University of Würzburg, received the second prize: and Dr. Oesterlen and Assistant-Inspector Honold, of Stuttgart, with Drs. Kühn and Gilg, of Berlin, received Honorable Mention.
—N. H. J. M.

*Sulphite Wood Pulp Lye, Utilisation of.* A. Harpf. Zeits f. angew. Chem. 1898, 925—931.

The various projects for the utilisation of sulphite lye by obtaining from it adhesive, sizing, tanning, or mordanting materials, all labour under the disadvantage that in their preparation, waste liquors are still produced, and, according to Müllner, the only thoroughly efficient method is to evaporate the lye and burn the residue, which, however, adds about 10 per cent. to the cost of the cellulose. Müllner has worked out a process in which the original lye is continually regenerated, and pure aluminium hydrate, free from iron, is obtained as a byproduct.

The lye from the soda and sulphate wood pulp industry contains caustic soda, sodium carbonate, sulphate and sulphide, and organic acid sodium compounds of unknown composition. On evaporating and calcining, the final products are sodium carbonate and sulphide, since the sodium sulphate is reduced by the organic matter. If now bauxite be added during the evaporation, and an amount of lime (CaO or $CaCO_3$) corresponding to the sulphur in the lye, the following reactions take place on calcination:—

$$3Na_2CO_3 + Al_2(OH)_6 = Al_2(ONa)_6 + 3CO_2 + 3H_2O$$
$$3Na_2S + 3CaO + Al_2(OH)_6 = Al_2(ONa)_6 + 3CaS + 3H_2O$$

The melt is then extracted with water, when the sodium aluminate dissolves, leaving calcium and iron sulphide. On passing carbonic acid into the solution, pure aluminium hydrate is precipitated in a crystalline form. The sodium carbonate solution is causticised with lime, and again utilised in the original operation. The 8—10 per cent. of sodium salts lost in the various recovery operations is added in the form of carbonate, sulphate, or bisulphate. In the manufacture of wood pulp, when sodium bisulphite (said to be preferable to calcium bisulphite) is used, the recovery process is modified in so far that the aqueous extract after calcination is treated with sulphur dioxide until neutral, when the aluminium hydrate is precipitated and filtered off. The solution of neutral sodium sulphite is treated with a further quantity of sulphurous acid and converted into bisulphite. The aluminium hydrate is dissolved in sulphuric acid (50° B.), forming aluminium sulphate, which is used for sizing paper. The whole of the sulphur is recovered as calcium sulphide, and can be utilised as in the Leblanc soda process. With regard to the cost of the process, the following figures are given (the prices in Austrian currency) for 10,000 kilos. of air-dried sulphite cellulose. The prices which are only approximate, are per 100 kilos. :—1,000 kilos. of bauxite at 2 fl.; 100 kilos of lime ($CaO$ or $CaCO_3$ calculated as $CaO$) at 1 fl.; 300 kilos. of sodium bisulphate at 1 fl. The yield is approximately 700 kilos. of aluminium hydrate at 8 fl., or 3,200 kilos. of aluminium sulphate (14—15 per cent. soluble $Al_2O_3$) at 4 fl.

In a discussion which follows Müllner's paper referring also to a previous one by Seidel, the author (Harpf) contradicts Seidel's and also Cross and Bevan's views that wood is to be considered as an ethereal compound of cellulose and lignin, since cellulose is present in the plant some time before the lignin appears. With regard to the utilisation of the waste sulphite lye, the author states that the evaporation may be carried out not only in vacuum pans but also in open-hearth furnaces, although in this case, the organic substances cannot be utilised for heating purposes, whereas the mass from a vacuum pan may be mixed with sawdust and employed as fuel. The waste sulphite lye only possesses tanning properties when acid, since hide immersed in a neutral or alkaline solution is very little affected, and even the effect of the acid liquor is not to produce a true leather, and it appears probable that the hide pores become choked with calcium sulpholignate. The schemes for the recovery of sulphur are not to be regarded as hopeful, since only a small portion is present as sulphur dioxide. Attention must therefore be paid to recovering as much sulphurous acid as possible from the boiler, by which means a saving of 37 per cent. of the whole sulphur used may be effected. A method for the utilisation of the lye, which was not alluded to, consists in converting it into briquettes by mixing the thickened lye with charcoal powder, but the briquettes are not suitable for blast-furnace work. With regard to Muller's process the author considers it would only be suitable in such a case that the manufacturer was compelled to get rid of his waste liquor. Seidel's method, which consists in utilising waste liquor as a wool mordant, is not capable of dealing with the enormous masses which are produced yearly. In a North German factory, using the Mitscherlich method (dry, and not wet steam, as in Kellner's process), two boilers, each holding 120 cbm., contain each for a charge 65 Rm. of wood and 85 cbm. of lye. 1 Rm. of wood gave, on working up, in round numbers, 130 kilos. of air-dried product, and thus 1 kilo. of the finished product produces 10 litres of lye. The factory has a monthly output of about 1,000 kilos. of cellulose, and hence, in round numbers, a daily production of 30 cm. of lye. This contains 10 per cent. of dry residue, 8 per cent. of combustible matter, and 2 per cent. of ash; or, in other words, 3,000 kilos. of dry substance, 2,100 kilos. of organic matter, and 600 kilos. of ash daily. The author is of opinion that the river pollution by sulphite wood pulp lye has been exaggerated, and suggests that the fulfilment of the following requirements ought to be considered sufficient. 1. The manufacturer in his own interest should recover as much free sulphurous acid from the boiler as possible. 2. The lye should not be allowed to run directly into the stream but should be stored in settling tanks until cold, to allow any fibres to deposit. 3. The cooled lye should be diluted as much as possible with other waste water from the factory, and eventually, before running into the river, be further mixed with fresh water. By allowing the hot lye to pass over calcium carbonate, the free sulphurous acid may be removed; but the addition of caustic lime must be avoided, since this renders the liquor alkaline, which promotes the growth of algæ in the drain pipes. A manufacturer from Voitsberg stated that he had made use of a filtering trench into which the lye is run. It gradually passes away through a lime formation and flows into a stream, dark coloured, but not poisonous to fish, which congregate at the influx. In paper factories the wash-waters from the bleaching vats might be added to the lye, thereby oxidising it, rendering it lighter coloured and destroying its smell. Finally, the sub-section IX. D of the Third International Congress for Applied Chemistry passed a resolution that sufficient evidence was not yet forthcoming as to the poisonous nature of these waste liquors and as to their utilisation.—T. A. L.

### PATENTS.

*Paper, An Improved Process for the Metallic Coating and Ornamenting of.* C. Endruweit, Berlin, Germany. Eng. Pat. 22,859, Oct. 5, 1897.

In this process an endless metal band is led over guiding rollers, and conveniently insulated on its surface, is guided through a number of galvanic baths in a quicker or slower manner according to the thickness of the deposit to be formed. A paper band, previously coated with cement and running from a roller, is then applied, the two bands being guided through a drying apparatus, and afterwards detached, so that the paper band carries away the metallic coating from the metal band.—G. H. R.

*Waste Cocoa-nut Husk Dust, Fibre, or Refuse; Impts. in Treating.* H. R. Romney, J. Thane, and the Fibrous Materials Syndicate. Eng. Pat. 3100, Feb. 7, 1898.

This process consists in utilising the cellulose dust, and short fibre, from the manufacture of mats, which has, until now been burnt or thrown away.

The dust is mixed, in the proportion of from 70—90 per cent. with any rough vegetable fibre capable of being reduced to pulp, and then moulded into sheets and slabs, and used for a number of purposes, such as, packing for use in battleships, covering for steam pipes and boilers, elastic flooring, &c., &c. The product can be rendered waterproof by adding during the manufacture, from 2 to 15 per cent. of rosin, turpentine, or other suitable size. To make it more incombustible it may also be mixed with asbestos dust or fibre.—C. M.

*Rendering Materials Fireproof, Impts. in; and in Extinguishing Fire.* L. Litynski, A. Rodakiewicz, and F. Kuroioski, Lemberg, Austria. Eng. Pat. 14,532, July 1, 1898.

*See under* V., *page* 1141.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

*Ozone.* A. Ladenburg. Ber. 1898, **31**, [14], 2508—2513.

Ozonised oxygen containing 8—9 per cent. of ozone was produced by means of the new Siemens' apparatus with 5 tubes, using a large Ruhmkorff and an electromotor with 350 interruptions per second. Such a mixture readily condenses in liquid air to a deep blue liquid. The condensation vessel, contained in liquid air in a Dewar's tube, was connected to the ozone apparatus without the use of cork or rubber. When full (22 c.c.), the oxygen current was stopped, the condensation vessel raised into the upper part of the Dewar's tube, and the liquid allowed to slowly evaporate. At first almost pure oxygen was evolved, then the gas contained ozone, and finally when the volume of liquid was reduced to a tenth, evaporation completely ceased. The vessel was again allowed to fill and the oxygen to evaporate; 2—3 c.c. of a bluish-black opaque liquid remained, which did not evaporate until the vessel was placed considerably higher in the Dewar's tube. The density and ozone content of the gas then evolved, were determined, the former by means of Schilling's apparatus, which measures the time of escape of a given volume, the latter by titration of the iodine liberated from potassium iodide. The gas contained 86·14 per cent. of ozone and had the density 1·3698, compared with oxygen. By allowing for the oxygen contained in the mixture, the density of pure ozone is calculated as 1·456, a satisfactory agreement with theory. In an experiment in which the boiling point of ozone was to be determined by means of a thermo-couple in the condensation vessel, the temperature rose rapidly to − 125° C. after evaporation of the oxygen, then ebullition appeared to commence and immediately a violent explosion occurred, apparently due to the endothermic nature of the change of ozone into oxygen.—A. C. W.

*Ozone, Production of, and Cost, as compared with other Ordinary Agents.* J. B. C. Kershaw. Elect. Rev. 1898, **43**, 151—153.

*Production and Types of Ozonisers.*—Andreoli's is of the "open" type, *i.e.*, the ozonised air is in direct contact with the electrodes, which are made of aluminium, and are separated by a sheet of glass acting as dielectric. Alternating currents are used. No cooling is required, but the air must be quite dry and free from dust. It is manufactured by the Electric Ozone Syndicate. In Otto's ozoniser each element consists of a sheet of metal laid upon a sheet of glass, or fixed between two glass plates. Otto's chief discovery has been that of the fact that the "output of ozone is proportional to the periodicity of the alternating current used to work the ozoniser." The elements of the Yarnold ozoniser consist of plates of glass coated on one side with gold leaf. These are connected alternately parallel to the two poles of a transformer. The air or oxygen passes into the space between two neighbouring plates and does not come into contact with the metallic films. This ozoniser is employed in the purification of oils and fats by the Ozone Oil Refineries and Industries Company (Rosenblum's patents). Each element of the Siemens and Halske ozoniser consists of an inner metal tube and an outer tube covered on its external surface with tinfoil; cooling is effected by passing water down the inner tube. It has been used at Greiffenberg since 1894 in connection with bleaching operations. The following table gives the output of ozone for the above types, with the cost of the electrical energy necessary to produce 1 kilo. of ozone and 1 kilo. of nascent or active oxygen :—

| Form of Ozoniser. | Yield of Ozone in Grms. per E.H.P. Hour. | Cost of Electrical Energy in Pence. | |
|---|---|---|---|
| | | Per Kilo. of Ozone. | Per Kilo. of Active Oxygen. |
| Yarnold ............ | 175 | 8·56 | 25·68 |
| Otto ................ | 150 | 9·99 | 29·99 |
| Andreoli............ | 94 | 15·90 | 47·70 |
| Siemens and Halske. | 20 | 75·00 | 225·00 |
| Theoretical figures.. | 1,000 | 1·50 | 4·50 |

*Uses and Comparative Cost.*—Ozone has been used with more or less success for the following purposes :—1. Ageing wood for musical instruments. 2. Maturing wines and spirits. 3. Sweetening foul beer casks. 4. Drying and thickening oils. 5. Bleaching waxes and fats. 5. Seasoning linoleum. 7. Bleaching fabrics and yarns. 8. Sterilising drinking water. 9. Producing vanillin and organic bodies of similar type. Some of these results can only be obtained by the use of ozone. But in many cases other oxidising agents are applicable, those most commonly employed being bleaching powder, manganate of soda, and bichromate of soda. In the following table the author gives estimates of the comparative costs of 1 kilo. of active oxygen from various sources. (In both tables the cost of electrical energy has been taken as $1\frac{1}{2}$*d.* per E.H.P. hour, since it would be impossible to obtain it at a lower cost in the small quantities demanded by ozonisers) :—

| Source. | | Cost in Pence. |
|---|---|---|
| Bleaching powder | ............................ | 18·3 |
| Sodium bichromate | ............................ | 48·0 |
| Ozone | Yarnold's ozoniser ...................... | 61·5 |
| | Otto's ozoniser ......................... | 66·0 |
| | Andreoli's ozoniser...................... | 83·7 |
| Sodium manganate | acid solution ............. | 86·0 |
| | alkaline solution ........ | 153·0 |
| Ozone—Siemens and Halske ozoniser | .......... | 261·0 |

From these figures he concludes: (1) That bleaching powder is the most economical oxidising agent yet produced, and that when chlorine gives satisfactory results it will not be displaced by ozone. (2.) That the industrial applications of ozone will be restricted to those processes in which at present the two sodium salts or atmospheric air are the customary oxidising agents; owing to its ease of application, &c., the use in the purification of oils, fats, and organic chemicals may be expected to extend rapidly. (3.) The extended application of ozone to the purification of water and the maturing of wines and spirits is less certain. Here its activity as an oxidising agent may cause unforeseen difficulties. (See also pages 1103—1106.)—D. E. J.

*Aluminium Chloride ($AlCl_3.6H_2O$), Preparation of.* S. P. L. Sörensen. Tidsskrift Physik og Chemi, 1898, **3**, 268. From Chem. Zeit. Rep. **22**, [91], 269.

The salt was prepared from metallic aluminium, which contains little or no magnesium and alkali-metals, by dissolving it in dilute hydrochloric acid (1 : 1). The solution was poured through a filter containing no magnesia or lime, and the filtrate mixed with an equal volume of concentrated hydrochloric acid. The mixture was cooled with ice-water, saturated with hydrochloric acid gas and allowed to stand for an hour, when the aluminium chloride was deposited. The liquid was decanted off and the residue shaken with an equal volume of ether, then filtered through a disc of hardened filter-paper and washed, first, with ether saturated with hydrochloric acid, then with pure ether. The solid was dried in the exsiccator over concentrated sulphuric acid and solid caustic potash, until it ceased to smell of ether or of hydrochloric acid. In the exsiccator the salt did not lose water.—J. McC.

*Mercury, Direct Introduction of, into Aromatic Compounds.* O. Dimroth. Ber. 1898, **31**, [13], 2154—2156.

Benzene, heated to 110° with mercuric acetate for some hours, yields phenyl mercuric acetate $C_6H_5.HgO.CO.CH_3$. Toluene behaves similarly. Phenol undergoes the same change very readily. A mixture of concentrated aqueous solutions of phenol and mercuric acetate, when warmed, rapidly deposits a crystalline mass of hydroxyphenyl dimercuric diacetate $OH.C_6H_3(HgO.CO.CH_3)_2$, melting at 216°—217°. The addition of sodium chloride to the hot mother-liquor precipitates *p*-hydroxyphenyl mercuric chloride, melting point 219°—220° C., at once, the corresponding ortho-compound, melting point 152·5°, crystallising out on cooling. These compounds dissolve without decomposition in caustic soda, the sodium salts being precipitated by alcohol. By heating these with methyl iodide the corresponding anisyl mercuric iodides are formed. The *o*- and *p*-phenol mercuric chlorides are split up by boiling hydrochloric acid into their constituents, whilst an ethereal solution of iodine gives *o*- and *p*-iodophenols. The mercury salts of substituted phenols, such as thymol and salicylic acid employed in pharmacy, probably possess constitutions similar to the above phenol derivatives.

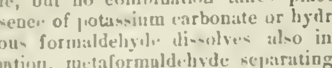
—T. E.

*Periodethylene or Diodoform.* Erdmann. Chem. and Druggist, 1898, **53**, [968], 749.

Periodethylene, $C_2I_4$, which was discovered by McKean, is prepared by the interaction of iodine and calcium carbide. It forms a very light, innocuous, bright yellow, inodorous powder, gives up its iodine when heated, and may be used as a substitute for iodoform.—A. S.

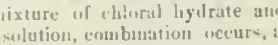
### *Chloral and Formaldehyde, Compounds of.* A. Pinner. Ber. 1898, **31**, [13], 1926—1928.

Chloral hydrate dissolves readily in a 40 per cent. solution of formaldehyde, but no combination takes place, either alone or in presence of potassium carbonate or hydrochloric acid. Anhydrous formaldehyde dissolves also in chloral without combination, metaformaldehyde separating out on standing. When excess of concentrated sulphuric acid is added to a mixture of chloral hydrate and 40 per cent. formaldehyde solution, combination occurs, a thick colourless oil separating out. This cannot be purified, decomposing into its constituents when distilled, but if the mixture is allowed to stand for one or two days, the oil solidifies to a mass of colourless crystals, containing two substances, which the author terms hexachlordimethyltetroxan—



$$CCl_3.CH\left\langle\begin{matrix}O.CH_2.O\\O.CH_2.O\end{matrix}\right\rangle CH.CCl_3,$$

and hexachlordimethyltrioxin—



$$CCl_3.CH\left\langle\begin{matrix}O.CH_2\\O-CH\end{matrix}\right.\begin{matrix}\searrow O\\ \nearrow\ \ \end{matrix}\!\!-CCl_3$$

—T. E.

### *Essences of Petit-Grain and Néroli.* E. Charabot and L. Pillet. Bull. Soc. Chim. 1898, **19**, [20 and 21], 853—857. See also this Journal, 1896, 919; 1897, 159, 632.

Tiemann and Semmler (Ber. **26**, 271) state that essence of néroli contains 20 per cent. of limonene, 30 per cent. of *l*-linalool, 40 per cent. of *l*-linalyl acetate, and small quantities of geraniol. Two pure samples examined in Schimmel's laboratory, from the south-east of France, had a saponification value of 38, corresponding to 13·3 per cent. of esters. The authors have themselves prepared and examined nine samples of néroli and eight samples of petit-grain. The following table shows the most important results obtained.—

| —— | Specific Gravity at 15° C. | Rotation in 100 mm. Tube. | Percentage of Esters. |
|---|---|---|---|
| Essence of néroli.. | 0·872—0·876 | + 2·15° to + 4·06° | 10·1—18·0 |
| " petit-grain | 0·891—0·894 | − 4·45° to − 6·15° | 51·5—53·7 |

The index of refraction of essence of néroli is about 1·474 at 180 C.; its solubility in alcohol appears to decrease with age. This essential oil is found to be dextrorotatory; the contrary is generally stated. An observation of the rotatory power would indicate admixtures with citron, orange, and bergamot oils, which increase it, whilst petit-grain and linalool reduce it. Oil of néroli contains a considerable fraction boiling about 175° C., which is not found in the essence of petit-grain. The ester content of the oils was found by boiling for half an hour with semi normal potash, and calculating to $C_{10}H_{17}.C_2H_3O_2$. The esters in essence of neroli appear to greatly influence its perfume, for, after saponification, the essences of néroli and petit-grain lose their characteristic odours, and acquire that of linalol. The alcohols of these essences are the same; their difference in odour must be due to the different acids contained in their esters.

In extracting the alcohols from oil of petit-grain, which contains linalool in quantity, by the phthalic anhydride process, a mixture of alcohols was obtained only slightly levorotatory, and possessing the odour of geraniol. The latter alcohol appears to be more easily extracted by this process.—A. C. W.

### *Strophanthin.* L. Kohn and V. Kulisch. Monatsh. für Chem. 1898, **19**, [7 and 8], 385—402. See also this Journal, 1898, 268, 369, 370, 601, 689, 791.

The Austrian and German Pharmacopœias direct that *Strophanthus* tinctures should be prepared from seeds of the *Kombé* or *hispidus* varieties, which are chiefly met with in commerce. Generally the green seeds are referred to *Kombé* and the smaller brown to *hispidus*, this classification being supported by the colour reactions with sulphuric acid. It is very doubtful whether these two distinctions are really reliable; a careful microscopical study is required. Fraser (Pharm. J. **16**, 109; **18**, 6, 69; **20**, 328) extracted from *Kombé* seeds the glucoside $C_{20}H_{34}O_{10}$. Arnaud extracted from seeds of the same variety, by a somewhat different process, the substance $C_{31}H_{48}O_{12}$, which is dextrorotatory and crystallises with 1 mol. of water. The same author has extracted from the seeds of *Strophanthus glaber* a glucoside identical with ouabain. The authors used good commercial *Kombé* seeds, they obtained a substance similar to Arnaud's and identical with a preparation from Merck. This forms strophanthidin by the action of acids, but no sugar. Feist examined a commercial preparation of *Kombé* strophanthin, the elementary analysis of which gave figures nearly identical with those of Fraser, and differing from the results of Arnaud and the authors; his strophanthidin differed in a similar manner. A comparison of specimens from Feist with those of the author's preparation left no doubt of the difference.

The *Kombé* seeds were carefully freed from the fibrous tufts, broken up as finely as possible, thoroughly extracted with petroleum spirit, dried, and exhausted with 70 per cent. alcohol. The filtered alcoholic extract was precipitated with basic lead acetate and lead hydroxide, filtered, freed from lead by means of sulphuretted hydrogen, and, after filtering, evaporated *in vacuo*. Repeated crystallisation from water produces a white microcrystalline preparation, of neutral reaction and free from nitrogen. It does not reduce Fehling's solution and is inactive, whilst Arnaud's strophanthin was dextrorotatory and Feist's inactive. Strophanthin is hygroscopic. The formula $C_{38}H_{58}O_{15}$ is preferred to Arnaud's formula $C_{31}H_{48}O_{12}$, on the ground of the results of a methoxyl estimation. The glucoside ouabain, $C_{30}H_{46}O_{12}$, extracted by Arnaud from *Strophanthus glaber*, differs only by $CH_2$ from his formula for strophanthin. It is possible that strophanthin is the methyl ester of ouabain, and that the substance isolated from *Strophanthus Kombé* is a mixture of ouabain and its methyl ester.

Acetylstrophanthin is obtained by boiling strophanthin with anhydrous sodium acetate and acetic anhydride; it is insoluble in water, soluble in alcohol, and melts at 236°—238° C. The analyses point to the formula—

$$C_{31}H_{44}O_8(C_2H_3O_2)_4,$$

or—

$$C_{38}H_{53}O_{10}(C_2H_3O_2)_5.$$

*Strophanthidin* is formed when strophanthin is boiled with dilute hydrochloric acid. The yield is 52·5 per cent. Recrystallised from hot alcohol, strophanthidin forms fine white needles, which unite to very light silky leaflets of melting point 195° C. Though insoluble in water, strophanthidin is very hygroscopic. Ultimate analysis points to the formula $C_{19}H_{28}O_4$, or $C_{28}H_{40}O_6$; the result of a methoxyl estimation makes the latter more probable. Feist's strophanthidin was $C_{26}H_{38}O_7.1\frac{1}{2}H_2O$ and $C_{26}H_{38}O_7.\frac{1}{2}H_2O$, and contained no methoxyl group.

The specimen of strophanthin from Merck, which was found to be identical with the authors' own preparation, was prepared from *hispidus* seeds.—A. C. W.

### *Digitoxin and Digitaline.* H. Kiliani. Ber. 1898, **31**, [14], 2154—2164.

Digitoxin is readily decomposed into digitoxigenin and digitoxose, to the former of which the composition $C_{22}H_{32}O_4$ is assigned. Digitoxose forms an oxime, $C_6H_{13}O_4N$; this does not give the blue coloration with acetic acid containing iron and strong sulphuric acid, which is given by digitoxose. Digitoxose unites with hydrocyanic acid to form a lactone, $C_7H_{12}O_5$, melting at 153°—154° C. In the hydrolysis of digitoxin, digitoxose is the only sugar formed—

$$C_{34}H_{54}O_{11} + H_2O = C_{22}H_{32}O_4 + 2C_6H_{12}O_4.$$

The formula here ascribed to the glucoside agrees well with the analytical results, and with the quantity of digitoxigenin isolated from the products of the hydrolysis.

Strong hydrochloric acid acting on digitoxigenin at the ordinary temperature leaves anhydrodigitoxigenin, $C_{22}H_{30}O_3$, which softens at 205°—210° C., and is oxidised in hot glacial acetic acid by chromic acid mixture, with the production of the ketone toxigenone, $C_{20}H_{26}O_3$ or $C_{19}H_{24}O_3$.

Molecular weight determinations and new analyses of digitaligenin point to one of the formulæ, $C_{23}H_{32}O_3$ or $C_{22}H_{30}O_3$, in preference to the older $C_{16}H_{22}O_2$.

Digitalin on hydrolysis yields *d*-glucose and a sugar, $C_7H_{14}O_5$, digitalose. Its decomposition may be represented by the equation—

$$C_{35}H_{56}O_{14} = C_{22}H_{30}O_3 + C_6H_{12}O_6 + C_7H_{14}O_5.$$

Digitaligenin, when oxidised by chromic acid, yields a substance probably identical with toxigenon.

The author has examined the French preparation—"digitaline cristallisée," which he finds to be very similar to digitoxine, but to possess certain differences.—A. C. W.

### *Essential Oils, "The Solubility" Value of.* E. Dowzard.

Chem. and Druggist, 1898, **53**, [968], 749.

The method proposed by the author is stated to possess the following advantages: (1) only one strength of alcohol is necessary; (2) the results represent the actual solubility; and (3) they are directly comparable.

Exactly 5 c.c. of the oil are mixed with 10 c.c. of alcohol (sp. gr. at 15° C., 0·799), and then water run in from a burette, until the solution becomes turbid. The end reaction is stated to be very distinct, although some of the more soluble oils give an opalescent solution before becoming turbid. The number of cubic centimetres of water required to produce turbidity is multiplied by 100, the result being termed the "solubility value." On testing different oils by this method, the following results were obtained:—

| Oil of | Solubility Value. | Number of Samples. | Specific Gravity (15·5° C.) | Optical Rotation. 100-mm. Tube. |
|---|---|---|---|---|
| Anise | 120—130 | 3 | 0·981—0·987 | To − 1° 25′ |
| Bergamot | 225—255 | 4 | 0·883—0·8845 | + 12° to + 15° |
| Cajaput | 450 | 1 | 0·923 | − 1° 30′ |
| Cananga | 265 | 1 | 0·916 | − 21° |
| Caraway | 250—290 | 3 | 0·911—0·917 | + 74° to + 79° |
| Citronella | 335—350 | 3 | 0·902—0·906 | − 10° to − 12° |
| Clove | 740—780 | 4 | 1·048—1·054 | To − 1° |
| Coriander | 520—530 | 2 | 0·870—0·875 | + 9° to + 12° |
| Dill | 320—330 | 2 | 0·907—0·915 | + 72° to + 75° |
| Geranium | 575—595 | 4 | 0·895—0·898 | − 7° to − 10° |
| Lavender (English) | 520—560 | 4 | 0·885—0·891 | − 8° to − 9° |
| „ (French) | 490—500 | 3 | 0·888—0·895 | − 5° to − 8° |
| „ (Spike) | 470—495 | 2 | 0·908—0·913 | + 2° to + 3° 30′ |
| Lemongrass | 460—470 | 2 | 0·900—0·902 | − 1° to − 3° |
| Nutmeg | 120—125 | 2 | 0·885—0·905 | + 14° to + 16° |
| Orange | 60 | 2 | 0·849—0·851 | + 91° to + 98° |
| Pennyroyal | 460—475 | 2 | 0·925—0·936 | + 21° to + 23° |
| Peppermint (American) | 200 | 1 | 0·909 | − 22° |
| „ (English) | 360—460 | 4 | 0·905—0·906 | − 23° to − 25° |
| Pimento | 730—780 | 4 | 1·045—1·049 | − 1° to − 4° |
| Rosemary (English and French) | 285—325 | 3 | 0·913—0·916 | + 2° to + 9° |
| Rue | 450—465 | 2 | 0·835—0·837 | + 1° to + 2° |
| Sandalwood (E. I.) | 390—400 | 6 | 0·975—0·978 | − 17° to − 18° |
| Sassafras | 130—145 | 3 | 1·065—1·069 | + 2° to + 3° 30′ |
| Savin | 190 | 1 | 0·922 | + 53 |
| Tansy | 475 | 1 | .. | .. |
| Thyme | 290 | 1 | 0·910—0·924 | − 3° to − 5° |
| Turpentine (American) | 50 | 1 | 0·860 | + 7° |

The specific gravity and optical rotation figures given are those obtained with the pure oils, and only in a few cases refer to the samples whose solubility values are given.—A. S.

### *Sandalwood Oil, Western Australian.* E. J. Parry.

Chem. and Druggist, 1898, **53**, [967], 708.

The Western Australian sandalwood oil, which is now seldom imported into this country, is usually distilled from the wood of *Santalum cygnorum*, probably admixed in some cases, however, with that of *S. lanceolatum*, *S. acuminatum*, and *S. persicarum*. The author has examined four samples of oil distilled from wood which had been cut for several years. The samples were too dark to conveniently take their optical activity (the oil is, however, always slightly dextro-rotatory). The following results were obtained:—

| — | Specific Gravity at 15° C. | Iodine absorbed. | KOH for Saponifying the Esters. |
|---|---|---|---|
| | | Per Cent. | Per Cent. |
| 1 | 0·9650 | 200·0 | 1·46 |
| 2 | 0·9644 | 204·5 | 1·15 |
| 3 | 0·9632 | 198·2 | 1·66 |
| 4 | 0·9613 | 197·6 | 1·35 |

The four samples were then mixed, the mixture having a specific gravity of 0·9643 at 15° C. After treatment with acetic anhydride and anhydrous sodium acetate, the dried acetylated oil (sp. gr. 0·9723) had a very pleasant and fruity odour, resembling that of the acetylated oil of *S. album*. On saponifying, a result corresponding to 75·7 per cent. of santalol was obtained (East Indian oil, 90 per cent.) The author considers that the acetylisation process causes other reactions with sandalwood oil than simple esterification, and that the figure obtained does not represent the true percentage of santalol (or whatever mixture of sesquiterpene alcohols may be present). The low specific gravity of the oil corresponding to so low a content of santalol is in close agreement with results previously obtained in the case of the oil of *S. album* (Chem. and Druggist, Aug. 3, 1895, 197).—A. S.

### *Eucalyptus Oil from Portugal.* Schimmel's Semi-annual Report, Leipsic, Oct. 1898, 26.

Three samples of Eucalyptus oil from trees cultivated in Portugal, have been examined. The first, derived from *Eucalyptus rostrata*, had a pleasant odour, contained much cineol and no phellandrene; it had a sp. gr. 0·921, and the optical rotation was − 1° 8′ at 30° C. It may be considered equal in value to the higher grades of oil at present met with in commerce. It was soluble in 2 parts of 70 per cent. alcohol. The second specimen, of doubtful botanical origin, probably from *E. resinifera*, contained cineol, phellandrene, and a terpene not found in the previous sample; the specific gravity was 0·893, and the rotation − 17° 5′ at 30° C. It was not soluble in 70 or 80 per cent. alcohol. The third specimen, derived from *E. obliqua*, contained cineol and phellandrene, and was soluble in equal volumes of 80 per cent. alcohol. The specific gravity was 0·914, and the rotation − 7° 28′. In the present condition of the supply of Eucalyptus oil, there being a surplus of good oil available, the introduction of low-grade oil from new sources is not likely to be profitable.—J. O. B.

### *Rose Oil (Adulteration of with Guaiacum Wood Oil).* Schimmel's Semi-annual Report, Leipsic, Oct. 1898, 38.

The possible application of the concrete oil of guaiacum wood to the adulteration of rose oils has not failed to be observed by Bulgarian producers. A specimen of otto of rose was recently met with, which contained this body, with an admixture of geranium oil. The congelation of otto is still regarded as a reliable test in Bulgaria; the addition of the easily crystallising guaiacum oil to specimens adulterated with non-congealing geranium oil enables them to pass this test, while the mild tea-like odour of the adulterant blends well with that of rose oil, so that it is not noticeable, even when the admixture is in considerable proportions.

—J. O. B.

### *Spike Lavender Oil (Characters of).* Schimmel's Semi-annual Report, Leipsic, Oct. 1898, 41.

In order to detect the addition of small quantities of French turpentine to spike lavender oil, it is essential to fractionate off the first 10 per cent. and observe the optical rotation of this fraction; in all the reliable spike oils hitherto examined, this first fraction has a distinct dextro-rotation, while any appreciable addition of turpentine will render it levogyre. Attention is called to the importance in carrying out this test of using Ladenburg's flasks of uniform size, and of slowly distilling the fraction, so that not more than one drop of distillate passes over in a second. Under these conditions four genuine spike oils gave fractions having a rotation from + 7° 11′ to + 5° 10′.—J. O. B.

### *Guaiacum Wood Oil (Botanical Source of).* Schimmel's Semi-annual Report, Leipsic, Oct. 1898.

Paetzold, of the Pharmaceutical Institute of the University of Strassburg, has ascertained that the origin of this wood, which has hitherto been uncertain, is *Bulnesia sarmienti*, a tree 40 ft. to 60 ft. high, which is indigenous to the Argentine provinces of Oran and Grand Chaco, particularly along the course of the Rio Bermijo, a branch of the Rio Paraguay. The wood is shipped from Buenos Ayres, and has recently been thrown upon the markets in quantities far in excess of the demand for the oil.—J. O. B.

### *Camphor Oil (Light, White).* Schimmel's Semi-annual Report, Leipsic, Oct. 1898.

The rectified white oil of camphor, consisting of the lighter fractions, continues to find use in European industries as a substitute for oil of turpentine, for dissolving caoutchouc and resins for cleaning machinery, and for perfuming the so-called turpentine soft soap. It has the advantage over other solvents of a relatively low inflammability. Thus, while the flash-point of oil of turpentine is 33·7° C., that of light camphor oil is 41·5° C.—J. O. B.

### *Menthol, Characters of, in the British Pharmacopœia.* Schimmel's Semi-annual Report, Oct. 1898, 63.

The menthol recognised in the British Pharmacopœia may not be pure, since it is stated that the crystals are "usually more or less moist from adhering oil." The requirements of the German Pharmacopœia, however, are more stringent, and can only be met by the pure article, having brittle crystals and a melting point of 43° C.—J. O. B.

### *Thymol, Melting Point of.* Schimmel's Semi-annual Report, Leipsic, Oct. 1898, 64.

The range of melting points from 43·3° to 51·7° C. adopted by the British Pharmacopœia is considered too wide, since pure thymol should not melt at a lower temperature than 51° C.—J. O. B.

### *Santonin Industry in Turkestan.* Chem. Ind. 1898, 21, [20], 444.

Santonin is obtained from the capitula of various species of Artemisia. The plants grow in great profusion on the steppes of Bezirke Tschimkent and Aulije-ata in the province of Syr Darja in Turkestan. The area under cultivation amounts to about 400,000 desjatinen (1 d. = 109·25 ar.). This is the only locality where the plant continues to flourish, other habitats, in the governments of Saratow and Pensa in Barbary and in the Levant, where the drug was originally collected, are no longer profitably worked. The harvest takes place in the month of August, when the capitula are richest in santonin. It is conducted in a very primitive manner, the plants are pulled up, and the small unexpanded flower heads stripped off, the rest of the plant being used for fuel. The gathered capitula are called darmin, the maximum yield of one desjatin is 15,000 pud (1 pud = 16.38 kilo.) of darmin, which fetches from 6 to 8 kopeks a pud on the steppes, or 25—30 kopeks in Tschimkent. Previous to the year 1870 the whole of the crop was carried in caravans to Nischnij Novgorod and Moscow. But then Russian enterprise started a factory on the spot for the production of pure santonin; at the present time these works take some 130,000 pud of santonica, of which 100,000 pud is worked up into santonin and the rest exported as "Semen Cinæ." In addition to this factory, there are two others, which do not produce santonin, but limit their output to the calcium compound, which is exported to Hamburg for further treatment.

During the period 1889—1894 the demand for santonin underwent marked diminution in consequence of the alleged superiority of the tannates of cinchonidine and quinidine as vermifuges. As a consequence, the collection of the darmin was neglected, and the plains were covered with the unharvested plants in immense numbers. In 1895, however, experience having shown that the so-called santonin substitutes were not equivalent in efficacy to santonin, the demand again increased and the factories resumed full work. In consequence of the danger of the extinction of the plant, chiefly from fires started by the nomadic tribes to improve the quality of the pasturage, and from over collection, the Russian officials have instituted measures for its protection, and have also made attempts to acclimatise it in Transcaspian and Transcaucasian districts.—J. O. B.

### *Hydurinphosphoric Acid.* E. Fischer. Ber. 1898, 31, [15], 2546—2549.

Attempts to prepare free purine, $C_5H_4N_4$, by the reduction of trichloropurine with hydriodic acid and phosphonium iodide, result in the formation of the hydriodide of hydurinphosphoric acid, $C_4H_9N_4PO_3.HI + H_2O$. This substance does not show the reactions of phosphoric acid, but, on warming with dilute hydrochloric acid, phosphoric acid is split off, and the hydurine residue is at the same time decomposed.—A. C. W.

### *Purine and its Methyl Derivatives.* E. Fischer. Ber. 1898, 31, [15], 2550—2574. See also this Journal, 1898, 763, 947.

Methylpurine is obtained by converting 7-methyldichloropurine into 7-methyl-2-iodopurine by treatment with hydriodic acid at a low temperature, and reducing the iodo-compound by boiling with zinc-dust and water. In a similar manner, trichloropurine is reduced by hydriodic acid and phosphonium iodide at 0° C. to 2.6-di-iodopurine, which is reduced by zinc-dust to free purine. The same di-iodopurine, by heating in a sealed tube with strong hydrochloric acid, is converted into xanthine. The latter may be converted into its methyl derivative, caffeine, in the dry or wet way; but as a test for xanthine, it is better to make bromoxanthine, and methylate this to bromocaffeine, which is readily recognised. Purine melts at 211°—212° (216°—217° corr.), is readily soluble in cold water and warm alcohol. The aqueous solution is without action on litmus or turmeric. Purine nitrate and picrate are characteristic salts, the former, $C_5H_4N_4.HNO_3$, melts at 205° C. with decomposition. The picrate melts at 208° C., and is soluble with difficulty. Like its halogen derivatives, purine forms salts with metals.—A. C. W.

### *Allocaffeine.* H. A. Torrey. Ber. 1898, 31, [13], 2159—2162.

Allocaffeine ($C_8H_9N_3O_5$) (Ber. 30, 3009) is converted into allocaffuric acid when boiled with water. Crystallised from ethyl acetate, allocaffuric acid ($C_7H_{11}N_3O_4$) forms delicate colourless needles, melting at 158° C., which change after standing in contact with the mother-liquor for a few days into more compact crystals, melting at 164°—165° and having the same composition as the lower melting modification, into which they are reconverted by recrystallisation from ethyl acetate. The acid is soluble in water, alcohol, and acetone, has a faint, sweet taste, does not reduce ammoniacal silver solution, nor does it give the murexide reaction. It is decomposed by bases. When boiled for three or four minutes with baryta solution, it is split up into mesoxalic acid, dimethylurea, and methylamine.—T. E.

*Iodoform, The Crystalline Form of.* W. J. Pope. Proc. Chem. Soc. 1898, [199], 219.

Considerable difficulty is experienced in obtaining iodoform in well developed crystals from solutions in the ordinary organic solvents, but it separates from acetone in magnificent six-sided tablets belonging to the hexagonal system.

*Hyponitrites, Their Preparation by Sodium or Potassium and Properties.* E. Divers. Proc. Chem. Soc. 1898, [199], 223.

Sodium nitrite reduced in concentrated solution by sodium amalgam, yields a third of its weight of sodium hyponitrite, and nearly the equivalent amount of several other hyponitrites, without the need of this salt as an intermediary. In this respect, the process has an advantage over the oxyamidosulphonate method. The hydroxylamine, which always accompanies the sodium hyponitrite, is easily got rid of by the continued action of the sodium amalgam, which is without action on the hyponitrite itself. The resulting ammonia is removed by exposure of the solution over sulphuric acid for a night under reduced pressure. Mercuric oxide, employed by all other chemists to remove hydroxylamine, has the fault of regenerating nitrite. The concentrated solution of sodium hyponitrite and hydroxide, thus obtained, is remarkably stable. In the preparation from it of other salts than the sodium salt itself and the silver salt, the sodium hydroxide has first to be neutralised, after diluting the solution with ice and water, mercurous nitrate being employed as an indicator with portions of the solution.

Sodium hyponitrite is prepared from the most concentrated alkaline solution, obtained as above, by precipitation with absolute alcohol, or (Jackson) by evaporation in a vacuum, and washing the salt with alcohol. It forms minute crystalline granules $(NaON)_2 + 5H_2O$, which effloresce and evolve nitrous oxide in the air, but in a vacuum desiccator are converted into an anhydrous non-coherent powder, stable in dry air. The anhydrous salt bears a somewhat high temperature in dry air without decomposition, then fuses and effervesces, forming sodium oxide, sodium nitrate, and nitrogen.

Silver hyponitrite is prepared by adding a highly dilute solution of silver sulphate or nitrate to the alkaline solution already described. It decomposes exceedingly slowly in the moist state, forming products similar to those obtained on heating. It is also decomposed by bright light, becoming brown, but is not blackened, by the light alone. When heated, it is decomposed without explosion, its yellow colour changing directly to that of metallic silver, without intermediate darkening; the products are silver, silver nitrate, nitrogen, nitric oxide, and nitric peroxide.

Hyponitrous acid, in solution, is more acid to litmus paper than carbonic acid, but its effect disappears as the paper dries. It decomposes slowly into nitrous oxide and water. When pure, it has no action on iodine-water or with the starch-iodide test. It decomposes silver carbonate, sulphate, nitrate, and chloride, when present in excess, but not the iodide. It is easily oxidised, but is unaffected by reducing agents. Apparently it has not yet been obtained free from every trace of nitrous acid; when carefully prepared, it will remain, however, for more than an hour without producing a blue colour with the starch-iodide test. Hyponitrous acid can be estimated gravimetrically as the silver salt, and volumetrically by potassium permanganate. Thum's permanganate process, in which the hyponitrous acid becomes ultimately nitric acid, is an excellent one; the failure of Hantzsch and Sauer and of Kirschner in its use is due to the modifications they made in it.

*Hyoscyamine, The Occurrence of, in the Hyosyamus muticus of India.* Wyndham R. Dunstan and Harold Brown. Proc. Chem. Soc. 1898, [200], 240.

The authors find that the stem and leaves of the Indian *Hyoscyamus muticus* contain about 0·1 per cent. of hyoscyamine, and that the alkaloid can be extracted in a pure state from this plant more readily than from ordinary henbane. The plant is fairly abundant in the Punjab and Beluchistan, and is likely to prove of value both as a drug and as a source of hyoscyamine.

*The Cinchona Alkaloids, Some Derivatives of.* W. Koenigs and M. Höppner. Ber. 1898, **31**, [14], 2355.

*See under* XXIV., *page* 1196.

*Ethyl Aldehyde, Volumetric Estimation of.* X. Rocques. J. Pharm. Chim. 1898, **8**, [9], 390.

*See under* XXIII., *page* 1194.

*Mustard Oil from Seeds of the Cruciferæ, Conditions of Development and Determination of Volatile.* G. Jörgensen. Nyt. Tidskrift fur Fysik ok Kemi, **3**, 91; Chem. Centr. 1898, **2**, [18], 927.

*See under* XXIII., *page* 1193.

*Solidifying Point of Essential Oils, Method of Determining.* Schimmel's Semi-Annual Rept., Leipsic, Oct. 1898, 43.

*See under* XXIII., *page* 1189.

*Iodoform, Some Reactions of and Detection of; in Aqueous Liquids.* L. v. Stubenrauch. Zeits. Untersuch. f. Nahrungs- und Genussmittel. 1898, **1**, [11], 737.

*See under* XXIII., *page* 1188.

## PATENTS.

*Aromatic Aldehydes, Impts. in the Production of.* G. B. Ellis, London. From La Société Chimique des Usines du Rhône, Lyons, France. Eng. Pat. 22,121, Sept, 27, 1897.

The patentee claims the process for manufacturing aromatic aldehydes by the oxidation of the methyl group in aromatic hydrocarbons or their derivatives, which consists in treating the hydrocarbons with an oxidising agent (sulphuric acid and manganese dioxide) in quantity insufficient for complete oxidation, and in subsequently separating the aldehyde produced from the unaltered hydrocarbon.—A. C. W.

*Oxypurines and their Alkyl Derivatives, Impts. in the Production of, from Trichloropurine.* J. Y. Johnson, London. From C. F. Boehringer and Söhne, Waldhof, Germany, Eng. Pat. 23,227, Oct. 9, 1897.

Trichloropurine, treated with alcoholic or aqueous alkali at the ordinary temperature, is converted into 6-ethoxy-2:8-dichloropurine, which hydriodic acid and phosphonium iodide reduce to 6-oxypurine (hypoxanthine). By previous methylation of the trichloropurine, dimethylhypoxanthine may be similarly obtained. Trichloropurine is converted into 2:6-ethoxy-8-chloropurine by heating with excess of sodium ethylate; this compound on reduction yields xanthine. Methylated xanthines are similarly obtained. The processes described are claimed.—A. C. W.

*Soluble Formaldehyde Compounds of the Proteïn Bodies, Manufacture of.* A. Classen, Aix-la-Chapelle, Germany. Eng. Pat. 25,942, Nov. 8, 1897.

The manufacture of compounds of formaldehyde and the proteïns soluble in water by subjecting these bodies to the action of formaldehyde under pressure at 120°—150° C., evaporating and treating the dried residue with alcohol or other solvent, is claimed, as also the compounds themselves, as new articles of manufacture.—A. C. W.

*Emetine, Impts. in the Preparation of a Crystalline Compound of.* W. G. Whiffen, London. Eng. Pat. 26,412, Nov. 12, 1897.

The process for preparing crystalline emetine hydrobromide is claimed, which consists in treating the crude alkaloidal extract with hydrobromic acid and concentrating the solution.—A. C. W.

*Artificial Mask, Impts. in the Manufacture of.* O. Imray, London. From La Société Fabriques de Produits Chimiques de Thann et de Mulhouse, Thann, Alsace. Eng. Pat. 28,467, Dec. 2, 1897.

The manufacture of dinitrobutylxylidine by the direct nitration of butylxylidine in sulphuric acid solution at 0° C., is claimed, and also the conversion of dinitrobutyl-xylidine and toluidine into dinitrobutylxylyl- and dinitrobutyltolylazoimide by diazotising the amides, converting into the perbromides, and treating with ammonia, or by diazotising and treating with hydrazoic acid.—A. C. W.

*Guanidine Salts, A Process for the Preparation of.* A. Goldberg, Chemnitz; W. Siepermann, and H. Flemming, Kalk, Germany. Eng. Pat. 29,308, Dec. 10, 1897.

In the production of guanidine by fusing ammonium sulphocyanide, the decomposition takes place slowly, and the yield is unsatisfactory. In the process claimed, ammonium sulphocyanide is heated with a metallic oxide or salt in an atmosphere of ammonia, or the double sulphocyanide of ammonium and a heavy metal is heated under ordinary or increased pressure. A second claim is for the admixture of a neutral porous substance with the reacting mass.

—A. C. W.

*Ethyl Alcohol, Impts. in the Manufacture of.* F. C. Coudert, jun., New York, U.S.A. Eng. Pat. 17,159, Aug. 9, 1898.

Acetylene, obtained from calcium carbide, is brought into contact with a solution of a reducing salt, such as the double ammonium chromous sulphate. Ethylene is produced, which is absorbed by sulphuric acid. After the addition of water to the solution of ethylsulphuric acid, alcohol is obtained by distillation. The reducing agent is regenerated by the action of nascent hydrogen electrolytically produced. The process described is claimed. A suitable apparatus is also described.—A. C. W.

## XXI.—PHOTOGRAPHY.

*Photographic Negatives into Positives, Conversion of.* R. E. Liesegang. Phot. Corr. 1898, **35**, 561; through Chem. Zeit. Rep. 1898, 260.

When a gelatino-bromide negative or positive is immersed in a cold saturated solution of ammonium persulphate, it is bleached; and on treatment with warm water (even the natural warmth of the hand suffices), the deposit dissolves, leaving the rest of the film unchanged. From a negative, therefore, a plate is produced which contains the shadows in relief, whilst the lights are more or less eaten away. Ammonium persulphate only behaves in this manner when its solution has been exposed to the air for some time; the fresh liquid seems rather to harden the gelatin.

In order to employ this reaction for the preparation of a coloured positive, for which purpose it is well adapted, a negative should be soaked in a saturated solution of ammonium persulphate that has stood for 14 days in an open flask; when completely bleached, it is floated with water and gently rubbed with the finger. The plate is then dipped into an aqueous solution of a suitable dye; or, if special permanency be required, colouring matter is precipitated within the gelatin by double decomposition—F. H. L.

*Turpentine, Photo-Chemical Action of.* J. Sperber. Chem. Zeit. 1898, **22**, [90], 961.

In the course of experiments on the action of various substances on photographic plates, Russell (Proc. Royal Soc. 1897, **61**, 424) found that turpentine had a marked influence, which he attributed to the liberated vapour. In order to determine whether this was the correct explanation of the phenomenon, or whether the change in the plate was not rather brought about by the emission of photo-chemically active rays, the author repeated Russell's experiments in the following way. Three photographic plates were supported horizontally at different levels in a skeleton stand which was placed upon a glass plate, and covered with a glass bell-jar, forming a tight chamber. On the middle photographic plate was placed a small dish of turpentine, the vapour from which soon filled the extemporised chamber. The apparatus was then left in a dark room for four or five days. In the first experiment the photographic plates at the bottom and middle were placed with the film uppermost, whilst the top plate had its film underneath. After the exposure to the action of the turpentine, all three plates were completely blackened, with the exception of the spot on the middle plate, where the porcelain dish containing the turpentine had stood. In the second experiment the position of the plates was reversed, the top one having its film upwards, and the other two downwards. In this case, after the given exposure, the plates were only darkened at the edges, and in other parts were transparent. In both experiments the air in the bell jar was completely saturated with turpentine vapour, so that had the vapour been the active agent, the plates should have been completely blackened in each case. But the author considers that the theory of the emission of active rays by the turpentine would account for the results in each case. In the first experiment these rays would strike immediately on the film of the top photographic plate, and on the inner walls of the bell-jar, whence they would be reflected on to the films of the middle and bottom plates, whereas in the second experiment only the edges of the films would receive the reflected rays. In the author's opinion there is a continual exchange going on between the cosmic ether which lies between the molecules of a substance and the cosmic ether outside, and in the case of turpentine, the intermolecular ether is endowed by the influence of the turpentine molecules with vibrations similar to those of the ultra-violet rays of light, and which, like them, are photo-chemically active.

—C. A. M.

*Platinum, Gold, and Silver Chlorides; Action of Light on.* E. Sonstadt. Proc. Chem. Soc. 1898, [198], 179.

The action of direct sunlight on very dilute solutions of potassium chloroplatinate is similar that of prolonged heating, platinum monochloride being separated as described in an earlier paper (Proc. 1898, **14**, 25), The action of bright sunshine is, however, slower and feebler than that of heat. Solutions of gold chloride, if sufficiently dilute, are more readily affected by light than corresponding platinum solutions. An aqueous solution containing about 0·04 per cent. of gold chloride required exposure to bright sunshine for a day or two before any action was perceptible, and the reduction was incomplete even after exposure during several weeks. A more dilute solution, containing 0·007 per cent. of gold chloride, became distinctly bluish after a few hours' exposure, and the blue coloration deepened day by day until a deposit formed, which was blue by transmitted light, and afterwards assumed the usual brown colour of precipitated gold, the liquid being left colourless. A trace of hydrogen peroxide was detected in the liquid. The reaction may therefore be considered to be the same in this case as when a very dilute solution of auric chloride is decomposed by heat (Chem. News, 1898, **77**, 74).

It seemed probable that the action of light on silver chloride would, like the decomposition of platinum and gold chlorides in dilute solution by the action of heat or of light, depend on the presence of water and the formation of hydrochloric acid and hydrogen peroxide. Some silver chloride, precipitated from solution of silver nitrate by hydrochloric acid, was thoroughly washed by agitation with repeated portions of water, and decantation, the operation being conducted by feeble gaslight, and then exposed under water to direct sunlight, with frequent agitation. After some days, the supernatant water was examined, and was found to contain a very appreciable quantity of hydrochloric acid, and to give a faint reaction for hydrogen peroxide. One of the two formulæ which will satisfy these conditions is $6AgCl + 4H_2O = 2Ag_2Cl + 2Ag + 2H_2O_2 + 4HCl$. But as silver, set free molecularly in presence of hydrochloric acid, would, on a reasonable presumption, form silver chloride with liberation of hydrogen, and as the nascent hydrogen, would react on part of the hydrogen peroxide to form water, the equation, with these corrections, becomes—

$$6AgCl + 4H_2O = 2Ag_2Cl + 2AgCl + 2HCl + 2H_2O + H_2O_2.$$

The same series of reactions would occur with the reformed silver chloride and water, and it is obvious that, as, at each stage of the reaction, one-third of the silver chloride and one-half of the water are reproduced, and similarly with the hyrochloric acid and hydrogen peroxide, it follows that when the silver chloride taking part in the reaction is completely converted into argentous chloride, water will still be left as a permanent residue in addition to hydrochloric acid and hydrogen peroxide. This assumes that the silver chloride in a condition of accessibility to the influence of light is in the exact proportion to the water present indicated in the equation. But if the attackable silver chloride is present in a certain excess over that required by the equation, the end products of the series of reactions may be represented by the simple equation

$$4AgCl + 2H_2O = 2Ag_2Cl + 2HCl + H_2O_2,$$

in which no residue of water appears. This is the second of the two possible formulæ referred to.

Some silver chloride, precipitated and washed as already described, was dried for several hours on a sand bath, the temperature being raised progressively until the chloride became adhesive. Although the process was conducted under very feeble gas light, the chloride was sensibly discoloured. This discoloration is probably due to the same reaction taking place between moisture and the chloride under the influence of a high temperature as is effected by light in the cold. It appears that a higher temperature is needed for the reaction than that of water boiling at ordinary pressure, as the chloride is not discoloured by heating under water. A tube containing imperfectly dried silver chloride was exposed to direct sunshine, when a bluish tint instantly spread over the chloride, deepening to a rusty brown, but leaving patches unaffected, so that, after long exposure, the fragments presented a mottled appearance with white spots on a dark ground. Evidently, portions of the chloride were really dry, though the bulk was not so, the dry portions resisting the action of light. Another tube, rather long and narrow, was similarly filled with the dry chloride, sealed, and exposed to sunshine with about half its length covered. The exposed portion of the chloride darkened as in the former case, with patches remaining unaffected. The covering was then shifted over the previously exposed part of the tube, with a like result; but the darkened chloride, now sheltered from light, became slowly bleached, until it was quite white. On again reversing the conditions, the bleached portion darkened, and the coloured portion was bleached, the same happening on each subsequent reversal. Even in the portion of the tube exposed to bright sunshine, the chloride on the under side became partially bleached, and on turning the the tube round, the effect, after a few hours was reversed. Dry silver chloride was then placed in the central part of a phial tube, and was protected by a little recently ignited asbestos from contact with fragments (placed in its lower and upper parts) of calcium chloride, and of caustic soda taken from sodium that had been long kept in a loosely closed bottle. The well-closed phial was kept in the dark during two or three days, to give time for absorption of moisture, and was then exposed to sunshine. The silver chloride became coloured, with white spots remaining unattacked, as in the former cases; but, on transference to a dark place, or shading a portion, there was no bleaching. The coloration remained practically constant in light and in shade. The bleaching effect, or the reformation of argentic from argentous chloride under the conditions described in the former case, must therefore be attributed to a reversal in the dark of the primary reaction which takes place in the light; a reversal made impossible in the last-mentioned case by the absorptive action of the calcium chloride and the soda. Hydrochloric acid has no bleaching effect on light-altered silver chloride suspended in water, either in darkness or in light.

### PATENT.

*Silver, Impts. in the Production of Halogen Compounds of.* J. C. Mewburn, London. From Chemische Fabrik von Heyden Gesellschaft mit beschränkter Haftung, Dresden, Germany. Eng. Pat. 26,176, Nov. 10, 1897.

Solutions of colloidal silver are treated with chlorine, bromine, or iodine, preferably in aqueous solution, until decolorisation takes place, to obtain the corresponding halogen compound in solution. Light is excluded during the process. An example is given, according to which 10 litres of a dark aqueous solution of 0·5 per cent. of colloidal silver is treated with chlorine solution of 0·6 per cent., gradually added until the solution is decolorised. Mineral acids and salts precipitate insoluble silver chloride from such a solution, but sodium acetate gives a precipitate in which the silver salt is partly in the colloid state. But when it is desired to obtain the soluble silver haloid as a precipitate, gelatin and ammonium citrate are added. It is stated that these silver haloid salts are suitable for photographic and medical purposes.—E. S

## XXII.—EXPLOSIVES, MATCHES, Etc.

*Guncotton, Moist; The Slow Decomposition of.* J. C. A. Simon Thomas. Zeits. angew. Chem. 1898, [44], 1003—1006.

On extracting moist pressed guncotton, such as is used for torpedo charges, with water, calcium nitrate and nitrite can be detected in the extract. Such guncotton contains at most 10 to 15 per cent. of water, and in order to increase its stability, the manufacturers add from $1\frac{1}{2}$ to $2\frac{1}{2}$ per cent. of chalk, so as to neutralise any acid produced by the spontaneous decomposition of the explosive. In the author's opinion the chalk causes a saponification of the nitric acid ester, partially by means of the calcium carbonate itself, which is not altogether insoluble in water, and partially by its formation into calcium bicarbonate through the carbon dioxide in the water. He does not, however, agree with O. Guttmann (this Journal, 1897, 283) (at least in the case of damp guncotton), that it would be better not to add chalk. For, he contends, there is always a slight spontaneous decomposition in such guncotton, with the formation of free acid, and if this is not neutralised by chalk, there is a danger of its setting up a further decomposition. In his opinion the advantage gained by the neutralisation of the free acid more than compensates for the disadvantage of the slight saponifying effect of the neutralising agent.

The experiments described in the present paper were undertaken with the objects of obtaining further proofs of the saponifying influence of chalk, and of determining at what rate the saponification proceeded. A specimen of guncotton from Stowmarket, which was more than 12 years old, was mixed with water and chalk so as to increase the saponifying factors. The amount of water, calcium carbonate, and calcium nitrate in the original sample were determined before the addition, and the substance used for the experiments finally contained 25 per cent. of water and 9 per cent. of chalk. Portions of this mixture were kept (1) for six months at the ordinary temperature; (2) for six months at 30° C.; and (3) for 14 days at 50° C. In experiments (2) and (3) the flasks, filled as completely as possible with the guncotton, were connected with a gas-measuring apparatus. In the second experiment only 2·5 c.c. of gas was collected, consisting entirely of air expelled from the flask. In the third experiment the volume of gas (at 0° and 760 mm.) was 6·9 c.c., and contained only a trace of carbon dioxide. From these results the author concluded that any considerable decomposition of the guncotton was out of the question; for otherwise, in the last experiment, the free nitric acid would have liberated carbon dioxide from the calcium carbonate. But as this was not the case decomposition could only have proceeded as far as the formation of calcium bicarbonate.

$$2CaCO_3 + 2HNO_3 = CaH_2(CO_3)_2 + Ca(NO_3)_2.$$

Two specimens of guncotton made at the same time were examined in a similar manner. The first had been kept in Holland and the other for four years in Java. The results were:—

| —— | CaO. | $N_2O_5$ found. | Equivalent $N_2O_5$ for ½ CaO. |
|---|---|---|---|
| | Mgrms. | Mgrms. | |
| I. Kept in Holland ........ | 66·0 | 64·0 | 63·7 |
| II. Kept in Java ........... | 126·6 | 121·0 | 122·1 |

From this it appears that the influence of a tropical temperature had almost doubled the amount of soluble calcium in this guncotton, and also that, if any soda was used in its manufacture, the sodium salts were subsequently completely washed out.

The general conclusion which the author arrives at is that it would take many years for the acid formed by the spontaneous decomposition of moist guncotton to neutralise the chalk usually added. Such guncotton should be removed from the stores before all the chalk has been neutralised; or it may be cut up small or otherwise subdivided, mixed with a fresh portion of chalk, and again pressed, unless the percentage of nitrogen has fallen too low.

—C. A. M.

*Guncotton and Smokeless Powder, Stability Tests for.* J. C. A. Simon Thomas. Zeits. für angew. Chem. 1898, [45], 1027—1031.

Guttmann (this Journal, 1897, 283) has shown that the presence of certain constituents in modern smokeless powders, renders the application of the Abel test untrustworthy. The author finds that the test is equally unsatisfactory with many samples of guncotton, a fact which he ascribes to the presence of traces of mercury, left in the cotton after it has been treated with very dilute corrosive sublimate solution. A mere trace of metallic mercury (such as would be reduced in the cotton) would, by vaporising and combining with any iodine that might be set free, suffice to prevent, or at least to hinder, the bluing of the iodide-starch paper. An old sample of Stowmarket guncotton, which gave the reaction after four minutes at 80° C., was washed with a 0·1 per cent. solution of mercuric chloride, and then with water, after which it resisted the test for an hour; and a strip of paper already blued in a different experiment, was actually decolorised after a minute's suspension in the apparatus with the mercury-treated sample. The presence of the mercury can be demonstrated by suspending a gold leaf in the upper part of a corked test-tube containing a little of the cotton heated to 70°—80° C. The author has not succeeded in obtaining with Guttmann's test (*loc. cit.*) the sharpness of reaction by which it was said to be characterised. The author believes, however, that tests, based on the detection of mere traces of decomposition products, are both too sensitive and too liable to be vitiated by the presence of foreign substances, such as are often found in smokeless powders. The only reliable test is, to heat the material for a long period, and to determine the time that elapses before red-brown fumes are evolved. Zigall, at the Vienna Congress on Applied Chemistry, described a heat test, by which the sample is heated to 130°—135° C., in strong glass eprouvettes, closed with corks surrounded by paraffined paper, until a piece of litmus paper, moistened with glycerin and water, is bleached by the brown nitrous vapours evolved. The author confirms Zigall in his statement that any good samples of nitrocellulose powder should stand this test for five hours.

The author recommends a test, which he has used with satisfaction for a long time. A very carefully cleaned reagent cylinder, 16 cm. high and 1·5 cm. in diameter, is fitted with a well ground glass stopper, and is supported, with its lower end in an oil-bath, and with the upper part freely exposed. The sample to be tested is placed in the bottom of the tube, and is thus heated by the oil-bath to a given temperature (of about 100° C.), which is carefully maintained for eight hours a day, until the production of red-brown fumes is observed; for which purpose a strip of white glazed paper is held behind the upper portion of the tube. The tube is always left in the oil-bath, during the whole time of heating and cooling. Two tests are made with each sample, and the exact time after which the first distinct appearance of brown fumes is observed, is taken as the end of the test. When the samples withstand the test for a long period, the variation in the results may be considerable, but greater concordance is found when decomposition sets in early. If, however, the sample has borne the heating for a sufficient time to indicate its safety in use, discrepancies (probably due to physical variations) are of small account. Powders containing nitroglycerin are less stable than those without; and it would appear that this substance is more sensitive than guncotton to an increase in temperature. The results of this test cannot be compared with those obtained by Abel's method.

From experiments made, it is concluded that a powder containing nitroglycerin should show no brown fume until it has been heated to 94°—96° C., as in this test, for four days; and that guncotton and powders free from nitroglycerin should show no fume until they have been heated to 99°—101° C., for three days. The higher temperature is used in the latter case because the time required under these conditions is shorter, and because, for that reason, the results are more concordant.

Samples of smokeless powder which have been maintained at a constant temperature of 30° or 35° have been tested by the above stability-test at 94°—96° C., at half-yearly intervals; and others kept at 45°—50° C. have been similarly tested at intervals of three months. In all cases there was a distinct decrease of stability, induced by the prolonged exposure to temperatures varying from 30° to 50° C., very slight in the former case, but very marked in the latter. The inference to be drawn is that magazines in which such powders are stored should be kept as cool as possible, even by artificial means, if necessary, in the case of ships employed on service in the tropics.

Some of the results obtained are given in the following table:—

| Powder. | Composition. | Quantity used. | Abel's Test at 80°. | Red-brown Fumes evolved. 94°—96°. | 99°—101°. | 104°—106°. |
|---|---|---|---|---|---|---|
| | | Grms. | Minutes. | Days. | Days. | Days. |
| Guncotton, Stowmarket ........ | Guncotton .................................. | 2·5 | 4 | 9—11 | 6½—8 | 4—4½ |
| " Muiden................ | " .................................. | 2·5 | 11 | 8—8½ | 4—4 | 2 |
| " Powder Muiden ...... | " granulated and faced with graphite. | 2·5 | 20—22 | 7—9 | 5—5 | 2—2 |
| " " " ...... | " gelatinised and faced with graphite. | 5 | 35 | 6—8 | 4—5½ | 3—4 |
| " " Cöln Rottweil | " gelatinised and faced with graphite; with camphor and paraffin. | 5 | 7—9 | 8—8½ | .. | .. |
| Ballistite, Muiden................ | 60 per cent. guncotton + 40 per cent. nitroglycerin, faced with graphite. | 4 | 23—27 | 6½—8 | 3—3½ | 1—1 |
| " Hembrug................ | 50 per cent. guncotton, + 50 per cent. nitroglycerin, faced with graphite. | 4 | 11 | 2—3 | .. | .. |

—W. G. M.

*Matches, A New Compound for.* Eng. and Mining J., Nov. 26, 1898, 634.

In response to the reward offered by the French Government for a substitute for white phosphorus in making matches, MM. Sevene and Cohen have submitted their matches. These matches contain phosphorus sesquisulphide and chlorate of potash. The sesquisulphide is a grey-yellowish substance, which is prepared by heating amorphous (non poisonous) phosphorus and sulphur. The substance is very stable; Lemeine, who studied it in 1864, kept it for 15 years exposed to the air without noticing any change. Its latent heat is low; it ignites at 95° C. (203° F.), and can be, therefore, lighted by rubbing, like ordinary phosphorus. The mixture, with chlorate of potash, burns quietly, whilst the mixture of amorphous phosphorus, which takes fire at 200° C. only, and chlorate of potash, is really explosive. For this reason inert substances are added to the chlorate in safety matches; but safety matches are still to be found which spit unpleasantly. The new matches are not likely to contain other impurities than amorphous phosphorus and water. They have become popular during the few months they have been obtainable, and are known as the S. C. matches, after the initials of their inventors. The public may hardly have noticed the change, for in their appearance the new matches resemble the old; they may have a faint smell, more a sulphide than a phosphorus smell, however. The sesquisulphide, at any rate, has such a faint smell, but the employes in the works are said not to complain about it. The new matches do not phosphoresce even when rubbed energetically, but they are poisonous to some extent.

### PATENTS.

*Explosives, Impts. in.* H. Boyd, Tamworth. Eng. Pat. 30,442, Dec. 23, 1897.

The following ingredients in or about the proportions stated are mixed together with cotton-seed oil and compressed into cartridges.

Nitrate of soda, 7 parts by weight; sulphur, 4 parts; picrate of ammonia, 2 parts; bichromate of potash, 1 part; peat dust, 1 part; commercial lime, 1 to 3 parts. Instead of peat may be used sawdust, fossil meal, powdered mica, and instead of lime, Irish bog ore, or oxide of iron which has been used for gas purification.—W. M.

*Matches, Impts. relating to the Manufacture of.* C. Kastner and A. Swoboda, Vienna. Eng. Pat. 30,546, Dec. 24, 1897.

It is proposed to use vegetable stalks such as those known as alicante-straw cut into suitable lengths as a substitute for wood in the manufacture of matches. The opening at the head or foot end of the hollow match may be closed before dipping by a paste made of cellulose.—W. M.

*Explosives and their Use, Impts. relating to.* G. H. Hosie, Innellan. Eng. Pat. 3863, Feb. 16, 1898.

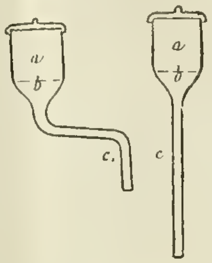
This patent describes safety cartridges to be placed in the borehole above the explosive charge, with the object of preventing ignition of any explosive gas which may be present. They are composed of one or more of the following ingredients: Glauber's salt, soda crystals, the alum compounds, borax; the hydrates, chlorides, sulphates, tungstates, and binoxalates of ammonium and magnesium; minerals, such as clays, infusorial earths, asbestos, charred organic matter; and nitrogen, ammonia, air, and carbonic anhydride in a compressed state. The compressed gases are contained in metal cases suited to the size of the boreholes. —W. M.

## XXIII.—ANALYTICAL CHEMISTRY.

### APPARATUS, ETC.

*A New Crucible, the "Tube-Crucible."* E. Murmann. Monatsh. für Chem. 1898, **19**, [7—8], 403.

The "tube-crucible" has a perforated bottom ending in a straight or bent tube. Over the asbestos layer covering the bottom is placed another perforated plate. The use of such a crucible enables the operator to heat precipitates in a current of any gas with the exclusion of air.—A. C. W.



*Boiling Points, Reduction of, to Normal Pressure; and on a Thermometer with Temperature and Vapour Pressure Scales.* P. Fuchs. Zeits. angew. Chem. 1898, [38], 869—871.

The author tabulates the variations of the boiling points of a limited number (eight) of substances, with changes of pressure recalculated from the experimental work of Regnault, Ramsay and Young, and others, and then describes a thermometer which may be used to ascertain whether any one particular substance is pure, or to assist in reducing the observed boiling point to the boiling point under the normal pressure of 760 mm.

A thermometer intended for the determination of the reduced boiling point of benzene, for example, is provided with a scale, which is graduated on one side of the mercurial thread into degrees of temperature, the whole range covered being from about 77·5° C. to 81° C.; whilst on the other side of the thread of mercury, the scale is graduated into the corresponding values of the vapour pressures of benzene vapour.

The boiling point of pure benzene under a pressure of 760 mm. is taken to be 80° C., and in order to test the purity of a sample of benzene, or to reduce its boiling point, taken at any pressure, not too far removed from 760 mm., to the normal pressure, the thermometer is inserted in the vapour of the boiling benzene, and the temperature and the corresponding vapour pressure noted. The barometric height, reduced to 0° C., is then obtained, and if this is identical with the vapour pressure read off on the instrument, the sample of benzene is pure, and has a reduced boiling point of 80° C. If, however, there is a difference, then the sample is not pure, and the reduced boiling point may be obtained by adding the difference to 760 mm., if the barometric reading is greater than the observed vapour pressure (or subtracting, if it is less), and observing the corresponding temperature on the instrument.—J. S.

*[Specific Gravity Determinations] Hydrometer Readings, Necessity for the Better Definition of.* H. Göckel. Zeits. angew. Chem. 1898, [38], 867—868.

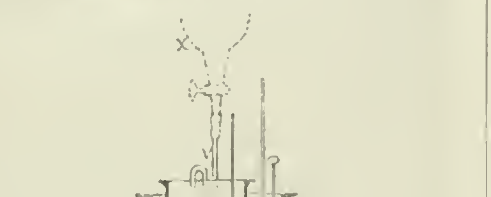
As a rule there is always affixed to each hydrometer, &c., a number which represents the temperature at which the determination ought to be made in order that the reading so obtained shall correctly represent the specific gravity of the liquid. The specific gravity so read off, however, may refer to water at 0°, 4°, 15°, 17·5° C., &c., as unity, and hence, owing to the absence of any uniform system, the author points out that the confusion that has arisen, and the necessity for affixing to all future hydrometers, &c., the temperature at which water is taken as unity.

Instead of the single number representing the temperature at which the instrument is correct, each instrument should, therefore, be provided with a second number, thus, $\frac{15^\circ}{15^\circ}$, $\frac{15^\circ}{4^\circ}$, $\frac{15^\circ}{0^\circ}$, &c., the lower of the two being the temperature at which the specific gravity of water is taken as unity.

In Germany, the legal alcoholmeter gives the percentage of alcohol by volume or by weight, deduced from determinations of specific gravity made at 15° or 15·5° C., and referred to water of the same temperature as unity, whereas water at 4° C. is taken as unity in the standardisation of lactometers, and of similar instruments for the examination of mineral oils.—J. S.

*Oils, Lubricating, and more especially Oleo-Naphthas; Contributions to the Study of.* A. Chenevier. Monit. Scient. **12**, [4], 1898, 785—789.

Recognising the practical necessity of determining th fluidity of lubricating oils at any temperature that is likel to occur through the heating of the bearings to which the oil is to be applied, *i.e.*, up to 200 C. (or higher) in the case of high-pressure cylinders, the author has devised the apparatus shown in the figure. The apparatus being clean, the reservoir X, provided with a stopcock below, is filled with the oil to be tested, which is then allowed to flow through the long copper U-tube, V, into the lower reservoir, R, in which the level may be kept constant by means of the overflow pipe, S. From R the oil passes down the copper tube, AA, to the horizontal pipe, B, and so to the vertical copper tube, CC, which is the chief essential of the arrangement. This tube, CC, is of copper, 200 mm. long, with an internal diameter of 5 mm.; through the centre, and maintained accurately in position by the guides at either end, is a copper core of such thickness that the width of the annular space between the tube and the core is just 0·3 mm. at all points. From the top of this annular capillary tube the oil is delivered by a movable spout, D, into any suitable receptacle. The whole arrangement excepting the reservoir, X, is immersed in an oil-bath, holding 7 litres, heated by a gas-jet, and provided with a stirrer, TT, to equalise the temperature, so that the oil, even when delivered into the lower receptacle, R, is at the temperature to be employed for the test. A thermometer is provided at the entrance to the tube, A, and two others are placed in the oil bath, one near the top, and one near the bottom. In use, the oil should flow from X at such a rate that there is a slight flow, drop by drop, from the overflow, S. The temperature having been accurately adjusted, a tared vessel is placed under the spout, D, and is allowed to remain there for an exactly measured period of from 1 to 12 minutes, the time being so chosen that from 1 to 10 grms. of oil will have been collected. The oil that has thus passed through the capillary is weighed, and the weight in grms. that would flow per hour is calculated. Thus a number may be obtained (expressed in units of weight) indicating the rapidity of flow, or fluidity, of any oil at constant pressure and at any given temperature. The indication may be expressed in units of volume by dividing the other number by the specific gravity of the oil at the temperature employed. The apparatus should be standardised by means of an oil of known and uniform fluidity, such as colza, as an irregularity in the adjustment of the core in the tube would introduce serious errors.



Petroff has determined the coefficient of friction, $\mu$, of an oil from the formula, $\mu = \frac{\pi . d^4 . p . \theta}{128 . L . q}$, where $d$ is the internal diameter and L the length of the tube, $p$ is the pressure under which the flow takes place, $q$ is the capacity of the bowl of the receiver in his special apparatus (Monit. Scient. 1891, 1072—1081), and $\theta$ is the time necessary to fill this volume. This formula is applicable to the author's apparatus; but, as $q$ is the only variable quantity, $\frac{\pi . d^4 . p . \theta}{128 . L}$ may be determined once for all as the constant (C) of the apparatus. Then, $\mu = \frac{C}{q}$, and C may be found by multiplying the known value for colza oil (according to Petroff's tables) by the volume of oil (in c.c.) which has passed through the capillary of the apparatus in one hour. When C has once been found, the value $\mu$ may be calculated for any oil at any required temperature. Tables and curves are given showing the lubricating values of various oils at different temperatures, as determined by the apparatus.

—W. G. M.

### PATENT.

*Thermometer, A New or Improved.* J. G. Lorrain, Norfolk House, Victoria Embankment, London, W.C. From C. S. Ruckstuhl, St. Louis, U.S.A. Eng. Pat. 29,178, Dec. 9, 1897.

Fever thermometers are made prismatic in cross-section, with one edge formed into a cylindrical lens adapted to magnify the image of the thread of mercury, and with narrow coloured streaks on its surface alongside of the edge lens, for indicating the place to look for the magnified image of the mercury thread and for rendering such image more easily visible.—R. A.

## *INORGANIC CHEMISTRY.—QUALITATIVE.*

*Sodium Bicarbonate, Detection of Monocarbonate in.* M. Kubli. Archiv. der Pharm. **236**, 321. Pharm. J. **61**, [1180], 481.

The author proposes as a test for sodium monocarbonate in the bicarbonate, a 0·4 per cent. solution of quinine hydrochloride. If equal volumes of this and a 6 per cent. solution of the bicarbonate made in the cold are mixed, no immediate and permanent turbidity should be produced. In the presence of 2 per cent. of normal carbonate, a permanent turbidity is obtained at once. (See also this Journal, 1898, 490).—A. C. W.

*Phosphates, Mineral and Animal, Method of Distinguishing.* F. Martinotti. Staz. Sper. Agr. Ital. 1897, 663.

The method depends on the different amounts of insoluble residue obtained when the phosphates are treated with hydrochloric acid. The phosphate is first freed from organic matter, if present, then boiled with hydrochloric acid and evaporated to dryness. It is then treated with hydrochloric acid and water, the insoluble matter separated by filtration, ignited, and weighed.

The results of the examination of a large number of phosphates indicated that whilst mineral phosphates yield at least 5·25 per cent. of insoluble matter, bone-meal yields at most 0·99 per cent. Bone-ash often contains more than 3 per cent. of silicates; mineral superphosphate contains at least 1·20 per cent., bone superphosphate not more than 0·80 per cent. of silicates. Double superphosphates contain about 3 per cent., and mixtures of bone- and mineral-superphosphates, at least 2·26 per cent. of silicates.

From these not altogether consistent data it is concluded that all phosphates and superphosphates, of animal origin, which contain more than 1·0 and 0·8 per cent. respectively of insoluble matter must be assumed to be adulterated with mineral phosphates.—N. H. J. M.

## *INORGANIC CHEMISTRY.— QUANTITATIVE.*

*Portland Cement, Detection and Estimation of free Lime* [*$Ca(OH)_2$*] *in Set.* N. N. Ljamin. Zap. Imp. Russ. Tech. Obschtsch., 1898, **32**, 75. Analyst, 1898, **23**, [271], 277.

The author found that the glittering crystals which are often apparent when thin sections of set Portland cement are examined by transmitted light, and which appear dark under the polariscope with crossed Nicols, consist of slaked lime, and he gives the following methods for its determination:—

(1.) The set cement is ground until its particles are smaller than these crystals, and is then passed through a sieve into a Hauenschild's apparatus. The powder is treated with a mixture of benzene and methylene iodide of sp. gr. 3·34, the vessel being placed under the receiver of an air-pump to remove air-bubbles, which would prevent the heavier portions from sinking. The calcium hydrate which rises to the surface is collected on a filter and the treatment repeated till all the floating matter is recovered. The lime is washed with benzene and ether, dried at 120° C. and weighed. For greater accuracy the lime may be further treated with a mixture of benzene and methylene iodide of sp. gr. 1·0—2·0 in order to remove fragments of coke, &c., but the amount of carbon rarely exceeds 1 per cent. of the total mass. The lime obtained is not quite pure, but the impurities only cause an error of about 0·5 per cent.

(2.) The second method depends on the fact that calcium hydroxide only loses its water of constitution at 150°—480° C., whilst the other constituents of set Portland cement lose their water of hydration at 160° C. A sample of the cement is heated for two or three hours at 180°—200° C.; in another sample, the carbon dioxide is determined, and in a third, the total volatile matter. On deducting the amount of carbon dioxide and the loss on heating to 200° C., from the total volatile matter, the amount of water in the calcium hydroxide is obtained, and from this, the amount of free lime may easily be calculated. The two methods are stated to give closely concordant results.—A. S.

*Mercury, A New Assay for.* R. E. Chism. Eng. and Min. J. 1898, **66**, 486.

Silver foil is used to collect the vaporised mercury, and to retain it whilst being weighed. From one-half to 1 grm. of the ore is mixed with 5 grms. of iron filings, which have been previously washed with alcohol to remove grease, and ignited, and the mixture is placed with a top dressing of filings in a Battersea annealing cup or crucible of unglazed white clay, 4·5 cm. high, 2 cm. diameter at the base, and 3·5 cm. diameter at the mouth. Over the mouth is pressed a piece of thin silver foil, which has been gently ignited and weighed, and on this is placed a flat copper, or preferably silver, evaporating basin, containing cold water. Heat is applied to the base of the crucible for 10 to 15 minutes, the upper part being shielded and kept cool by a piece of sheet tin with a hole, through which the greater part of the crucible passes. The mercury vapour rises, and is condensed and weighed on the cold silver foil. Ten minutes heating is not long enough for most ores, whilst with 15 minutes, mercury is apt to be lost. After weighing, the mixture should be heated again for 10 minutes to ascertain if any more mercury can be collected, in which case a fresh assay should be made, and the time limit extended, so as to confine the operation to one heating. The silver foil shows a distinct stain with only a trace of mercury, and its depth is a rough indication of the amount present. On gentle ignition the stain disappears, and the foil may be used again for two or three more assays. The underneath surface of the silver dish should be kept bright, and examined after each operation for mercurial stain to guard against loss of mercury through the foil by leakage or soaking.—A. W.

*Lead and Tin, Determination of Small Quantities of.* F. Hundeshagen. Zeits. für öffentl. Chem. **4**, 673—676. Chem. Centr. 1898, **2**, [15], 829.

Filtrates from sulphate of lead precipitated in the usual way, often show, on adding excess of ammonia, more or less appreciable quantities of lead. In consequence of this, the effect was tried of precipitating the lead at first in presence of excess of ammonia, with the result that the ammoniacal filtrate, in absence of tartaric acid, &c., was found to be always absolutely free from lead, whilst the precipitate contained all the tin present, even such small traces as might easily escape detection; also, the stannic oxide precipitated by ammonia, filters easily, and is more completely separated from copper, zinc, nickel, &c., than by the usual nitric acid method.

For the determination of lead and tin, with which may be combined that of iron, nickel, aluminium, and antimony, the sample (1 to 5 or 10 grms., according to the nature of the substance) is treated as usual in a covered porcelain dish with nitric acid. After the substance has dissolved, the liquid which has collected at the junction of the covering funnel and the dish is carefully displaced by dropping in just as many c.c. of concentrated sulphuric acid as grms. of substance have been taken. The funnel is rinsed and the contents of the dish evaporated until fumes of sulphuric anhydride are evolved. The liquid is then allowed to cool, and water added, with stirring and warming, until all the soluble salts are dissolved. Strong aqueous ammonia is then added, with agitation, until the hydroxides of copper, zinc, &c., precipitated at first, are redissolved. The ammoniacal liquid is allowed to stand at least 24 hours in a cool place, shaking vigorously from time to time. The precipitate may contain, according to the nature of the original substance, either lead sulphate with some ferric hydrate, or besides, tin, in the form of dense flocks of stannic oxide (sometimes also as basic sulphate), together with bismuth, aluminium, antimony, phosphorus, silicon, traces of arsenic, and residual carbon. The precipitate is washed, first by decantation and then on the filter, with dilute ammoniacal ammonium sulphate solution (containing 2·5 per cent. of $NH_3$ and 2·5 per cent. of $(NH_4)_2SO_4$) till all soluble metallic compounds are dissolved. If the precipitate contain only lead sulphate with a little iron, and the quantity be not too small, it is filtered directly on to a Gooch crucible, washed as above, dried, the ammonium sulphate volatilised at below red heat, and the residue of sulphates of lead and iron weighed, from which the iron is washed out by dilute acid and determined as ferric sulphate. If the amount be very small (only a few mgrms.), the precipitate is dissolved in hot dilute hydrochloric acid (1—1), the filter and dish being washed several times with hot acid, and finally with water, the solution evaporated, with addition of one drop of concentrated sulphuric acid, the ammonium salts volatilised, and the lead and iron determined as above.

With very small quantities of iron, the colorimetric determination is sufficient.

When besides lead and iron, tin, bismuth, aluminium, &c., are present, the precipitate is dissolved in hot hydrochloric acid, the bismuth precipitated by adding a large excess of water, and the filtrate saturated with sulphuretted hydrogen. Excess of pure caustic soda is added, together with a few drops of yellow ammonium sulphide, the mixture warmed, the insoluble sulphides filtered into a Gooch crucible, washed with water containing ammonium sulphide, dried, and after the addition of a little sulphur, gently ignited in a current of hydrogen. In the residue, which consists of the sulphides of iron and lead, the iron is determined. If the precipitate be very small, it is collected on a paper filter, oxidised with bromine vapour, dissolved in hot hydrochloric acid, and the iron determined as before. The filtrate from the insoluble sulphides is acidified with hydrochloric acid to separate the dissolved sulphides, and in the filtrate, after suitably concentrating, aluminium is precipitated by cautious addition of ammonia and some ammonium sulphide, the precipitate added to that obtained from the first ammoniacal filtrate, and then purified from silicon and phosphorus.

The reprecipitated sulphides are collected in a Gooch crucible and ignited, or dissolved in ammonium sulphide solution, the solution evaporated, the residue oxidised with nitric acid, and ignited in a current of air containing ammonia. The residue is dissolved in strong, hot hydrochloric acid, the antimony precipitated by metallic iron, and the tin again precipitated and determined separately. In the final ammoniacal filtrate, after separation of lead, tin, &c., copper, zinc, silver, cadmium, nickel, cobalt, &c., are determined.—A. S.

### *Copper, Iron, Lead, Alkali Ferrocyanides, Dextrose and Sulphuric Acid (in Sulphates), Volumetric Processes for the Estimation of.* Rnoss. Zeits. f. anal. Chem. 1898, 37, [7], 126—136.

The author makes use, in a number of cases, of the general fact that normal salts (especially the insoluble sulphates, ferrocyanides, &c.) of certain metals are decomposed quantitatively, when boiled with barium hydrate, into oxides or basic salts. The amount of alkali thus neutralised may be accurately titrated.

In application the following details are given:—

1. *Estimation of Lead.*—The lead solution is boiled with an excess of sodium sulphate, cooled, and exactly neutralised by means of pure caustic alkali, and dilute sulphuric acid, using phenolphthaleïn as indicator. The actual titration which now begins is effected by adding $\frac{N}{5}$ barium hydrate to the liquid, boiling after each addition until the solution is permanently alkaline, after which the excess of base is titrated back by $\frac{N}{5}$ $H_2SO_4$. In this reaction, $PbSO_4$ is converted into $PbOPbSO_4$, therefore, 1 c.c. $\frac{N}{5}$ alkali = 0·0412 Pb.

2. The lead is precipitated by an excess of potassium ferrocyanide, and the liquid and precipitate afterwards treated exactly as described under 1. Here also the insoluble lead compound is converted into the basic salt.

3. *Lead Oxalate* is not attacked, even at a boiling temperature by barium hydrate, but may be readily estimated, after conversion into sulphate by treatment with sodium sulphate and calcium chloride.

*Estimation of Sulphuric Acid in Sulphates.*—The process is the converse of that for lead, *i.e.*, the sulphuric acid is precipitated by boiling with an excess of lead nitrate and the precipitate, after washing, titrated with barium hydrate. Similarly, the negative radicle in alkali ferrocyanides and chromates may be estimated by precipitation with lead nitrate, &c. With chromates, however, it is advisable to filter off the precipitate and titrate the excess of lead in the liquid by the sulphate process (1), instead of titrating the precipitate itself, because the red colour of the basic salt, into which the lead chromate is converted, somewhat obscures the indications of the phenolphthalein.

1. *Estimation of Copper.*—An excess of sodium oxalate (sufficient to redissolve the copper oxalate at first thrown down) is added, the liquid exactly neutralised as described under lead, an excess of calcium chloride added, and finally titrated at a boiling temperature with $\frac{N}{5}$ barium hydrate. Alkalinity is indicated (phenolphthalein) when the whole of the copper has been precipitated as oxide. With solutions of copper free from organic acids, the process is simpler. The solution is neutralised by the aid of methyl orange—in moderately dilute solutions, the transition from red to yellowish-green is sharply marked—and then titrated hot with baryta water and phenolphthalein as before.

2. Applicable to small quantities of copper. The solution is boiled with potassium ferrocyanide, potassium nitrate being added to assist the precipitation, filtered, and the precipitate titrated with baryta-water and phenolphthalein. Alkalinity obtains when one-half of the copper ferrocyanide has been converted into oxide.

*Estimation of Reducing Sugars (Dextrose).*—The precipitate of cuprous oxide obtained in the usual way by the application of "Fehling's" solution, is dissolved in a small quantity of nitric acid, and the solution boiled to expel oxides of nitrogen. A slight excess of pure caustic alkali is then added to the hot solution, and the excess exactly neutralised by dilute acid and phenolphthalein. Finally, the precipitated cupric oxide is dissolved (hot) in a slight excess of standard sulphuric acid, and the liquid, when cool, titrated back with standard alkali and methyl orange as indicator.

*Estimation of Iron (in Ferric Salts).*—1. The *modus operandi* is the same as that given for the determination of copper under "dextrose," with this exception, that the precipitate of ferric hydrate is dissolved in standard oxalic acid, and the excess titrated back by baryta water. Under these conditions methyl orange is neutral to normal ferric oxalate; *i.e.* 1 c.c. of N acid = 0·0186 grm. Fe.

2. The ferrocyanide process is also applicable to iron. The solution is boiled with potassium ferrocyanide and nitrate, the precipitate washed with a weak solution of the latter salt, and titrated in the usual way. The whole of the iron is converted into hydroxide; therefore, 1 c.c. N alkali = 0·0186 grm. Fe.—H. T. P.

### *Molybdenum: Determination of the Technically Available, in Molybdenum-Glance.* H. Borntråger. Zeits. f. anal. Chem. 1898, 37, 438.

As a rule, the methods in use aim at the estimation of the total, rather than of the technically available molybdenum. The latter desideratum the author claims to be satisfactorily met by the following process:—1 grm. of the ore is digested for two hours with 25 c.c. of strong nitric acid, in an Erlenmeyer flask. The molybdic acid formed is then dissolved in ammonia, the solution filtered, and the residue again treated with nitric acid and ammonia. The combined filtrates are acidified with nitric acid, and evaporated to dryness. From the residue, consisting of ammonium nitrate and molybdic acid, the former is dissolved by digestion with 50 per cent. alcohol; the latter collected on a tared paper and weighed; or, better still, dissolved in normal ammonia and estimated by titration.—H. T. P.

### *Uranium, Patera's Method for the Estimation of.* H. Borntråger. Zeits. anal. Chem. 1898, 37, 136—137.

Patera's method for the estimation of uranium in its ores is as follows (Fresenius, Quantitative Analysis):—The ore is dissolved in nitric acid, the solution diluted, saturated with sodium carbonate, boiled, and filtered. From the filtrate the uranium is thrown down by the addition of sodium hydrate, and the precipitate of sodium uranium oxide collected and weighed as such. The author points out that this method, although fairly accurate in the case of pure high-grade ores, yields results much too high with poor silicious samples. With such the precipitate always contains a considerable proportion of silica. It is advisable, therefore, to separate the silica from the crude precipitate, in the usual way, by evaporation with hydrochloric acid, and afterwards to reprecipitate the uranium by ammonia or soda.—H. T. P.

### *Iron and Steel; Rapid Methods of Determining the Principal Elements in.* M. L. Campredon. Monit. Scient. 12, [4], 1898, 778—781.

The following are proposed as standard methods:—

*Iron.*—Dissolve the metal in dilute sulphuric acid, boil off the hydrogen, add 1 to 2 grms. of sodium bicarbonate to produce an atmosphere of $CO_2$ in the flask, and titrate with permanganate. Time required: 10 to 15 minutes. Accuracy: to within 0·1 c.c. of permanganate or 0·15 per cent. When the metal is incompletely soluble in sulphuric acid, other solvents are used; the iron is then usually present in solution as peroxide, and is titrated with stannous chloride.

*Total Carbon.*—Treat the metal with the double chloride of copper and potassium by Carnot's method, transfer to Wiborgh's apparatus, and determine the carbon gasometrically as $CO_2$. Time: about 2 hours. Accuracy: to within 0·1 per cent.

*Graphitic Carbon.*—Separate the graphite by treatment with nitric acid, and determine gasometrically by Wiborgh's process. Time: about 2 hours. Accuracy: to 0·1 per cent.

*Combined Carbon.*—Determine by difference or by the Eggertz process. Time: for mild steels, 1 hour; for hard steels, 2 hours; but by the Eggertz test, a series of 10 or 20 samples can be tested in an hour: Accuracy, to within 0·01 per cent.

In all carbon tests, certain materials, such as ferrochrome and ferro-tungsten, necessitate the use of slower methods.

*Manganese.*—Samples containing less than 1 per cent. are treated by converting the manganese into permanganate by means of bismuth tetroxide, and titrating with standard hydrogen peroxide solution (Schneider's process). Time: 10—15 minutes. Accuracy: to 0·01—0·02 per cent.

For samples containing more than 1 per cent:—Treat the metal so as to obtain a solution containing the iron as ferric chloride and the manganese as manganous chloride; precipitate the iron by adding excess of zinc oxide; and titrate the residual liquid with potassium permanganate at 80° C., adding the permanganate until a rose colour is produced (Guyard-Volhard's process). The titre of the permanganate must be determined on samples containing known manganese percentages, under the conditions of the test, as standardisation by iron leads to fallacious results. Time: 10—15 minutes. Accuracy: to within 0·025—0·25 per cent. according to the percentage of manganese present. It is sometimes necessary (*e.g.*, with ferro-chromes) to precipitate the Mn as oxide and redissolve in HCl.

*Sulphur.*—Dissolve in HCl containing a little $H_2SO_4$, pass the gas mixed with $CO_2$ and purified hydrogen, through a red-hot porcelain tube to convert all volatile sulphur compounds into hydrogen sulphide; pass the gases through a slightly acid solution of zinc acetate. Add an excess of iodine solution to the flask containing the zinc sulphide ($ZnS + I_2 = ZnI_2 + S$), and titrate back the excess of iodine with standard thiosulphate. This (Rollet-Campredon's) method is used in most of the French metallurgical works. Time: for iron and steel, 15 minutes; for cast-iron, 25—30 minutes. Accuracy: for samples containing up to 0·1 per cent. S., to within 0·005 per cent.; for others, to within 0·01 per cent.

*Phosphorus.*—Treat 2 grms. of steel with 75 c.c. of a (1:1) nitric acid, add 5 c.c. of permanganate solution (10 grms. per litre); when manganese oxide begins to deposit, add 50 mgrms. of sugar; then, when the liquid is clear again, add 25 c.c. of (1:1) ammonia solution, and heat to 85° C. Now add 50 c.c. of molybdate solution and shake for 5 minutes; filter by suction and wash with a 1 per cent. potassium nitrate solution; place the filter and precipitate in a flask containing a known quantity of standard soda, and titrate back the excess of soda with standard nitric acid (Pittsburg method). Time: 15—20 minutes. Accuracy: for samples containing less than 0·1 per cent. P, to within 0·005 per cent.; for others to within 0·01—0·02 per cent. The determination is less simple when Ti or As is present.

*Arsenic.*—Dissolve in nitric acid as in the determination of phosphorus. Evaporate to dryness. Redissolve in strong HCl and transfer to a flask with a side tube, add 1 grm. of ferrous chloride (free from As) and distil, collecting the distillate; when the liquid in the flask is syrupy, add more HCl and re-distil. Neutralise the distillate with ammonium carbonate, and titrate the $AsCl_3$ with iodine solution. Time: 2 hours. Accuracy: to within 0·005 for samples containing little As.

*Silicon.*—For samples of grey pig-iron: Take 1—2 grms. of the sample, mix with 1 grm. of potassium chlorate, and a few c.c. of boiling water; add 30 c.c. of strong HCl, and evaporate to dryness. Redissolve in 10 c.c. of strong HCl, dilute with a little boiling water. Filter (filtration is very rapid), ignite, and weigh. Time: about 1 hour. Results are likely to be a little too high. For white pig-iron, steel, or iron samples: dissolve in nitric and sulphuric acids. Time: about 2 hours. Results: accurate. For ferro-silicon or silicon-spiegel: treat in a flask with 30 c.c. of HCl containing bromine. When the metal appears to be dissolved, add 20 c.c. of sulphuric acid (half-strength) and evaporate until white fumes are evolved. Redissolve, filter, ignite, and weigh the $SiO_2$. Rapid determinations in samples of pig-iron may be made by Ford's method in 15 mins.

*Copper.*—Dissolve 1—5 grms. in a pear-shaped flask in 50—250 c.c. of (1 : 5) sulphuric acid. Heat until dissolved, dilute with boiling water to 250—300 c.c., and add, gradually, 3 grms. of sodium thiosulphate in crystals, the liquid boiling throughout, and being kept boiling until it has cleared. Filter, wash, and ignite the precipitate, dissolve in nitric acid, add ammonia in excess, and filter off any iron that may be present. Acidify the filtrate with $HNO_3$, evaporate to dryness, and volatilise ammoniacal salts. Redissolve in a few drops of nitric acid, add a slight excess of sodium carbonate, and acidify with acetic acid. Add 1 grm. of potassium iodide (the liquid measuring 10—20 c.c.), and titrate with thiosulphate. Time: 1 hour. Accuracy: to within 0·005 per cent.

*Chromium.*—Stead's method—by oxidation with potassium permanganate, followed by titration of the resulting chromic acid—should be followed accurately. Time: about 1 hour for steels. Accuracy: to within 0·05 per cent. For ferrochromes with difficulty dissolved in dilute sulphuric acid, the metal is attacked by sodium peroxide in a nickel capsule.

For *nickel, tungsten, molybdenum, titanium, and aluminium*, the author has not yet found a rapid method that he considers reliable.—W. G. M.

### *Ferro-Tungsten, Analysis of.* A. G. McKenna. Eng. and Mining J. 1898, 66, [21], 607.

0·5 grm. of the finely-ground sample is mixed thoroughly with 3 grms. of sodium peroxide in a copper crucible. It is necessary to use a weighed quantity of peroxide, and correct for the small amounts of silica and iron which are always present. The mixture is fused for about one minute at a dull red heat with slight agitation, cooled, and the melt washed out with hot water. A few c.c. of alcohol are added, and the mixture heated to render all the manganese insoluble. If the decomposition be complete, the precipitate, which is filtered off with aid of suction, contains the iron, manganese, and copper from the crucible, whilst the whole of the tungsten is present in the filtrate. The silicon occurs partly in the precipitate and partly in the filtrate. The filtrate is acidified with hydrochloric acid, evaporated to dryness, the residue taken up with 15 c.c. of hydrochloric acid, and the solution again evaporated to dryness. The residue is then again taken up with 15 c.c. of hydrochloric acid, 100 c.c. of water added, the mixture boiled for a few minutes, filtered with aid of suction, and washed with dilute nitric acid (1:10). The filtrate should be tested for tungsten by evaporating to dryness. The precipitate containing the iron and manganese is dissolved in hot dilute nitric acid, the solution evaporated to dryness, the residue taken up with hydrochloric acid and water, and the insoluble silica filtered off through the filter already containing the remainder of the silica, together with the tungsten trioxide. The precipitate is washed on the filter with dilute nitric acid, ignited, weighed as $WoO_3 + SiO_2$, then treated with 0·5 c.c. of sulphuric acid, and about 5 c.c. of hydrofluoric acid, evaporated slowly to dryness, ignited strongly, and again weighed. The residue consists of tungsten trioxide, whilst the loss in weight gives the amount of silica.

The solution containing the iron and manganese is rendered alkaline with ammonia, boiled, filtered with aid of suction, the precipitate washed with hot water, redissolved in dilute nitric acid, ammonia added, and the precipitated ferric hydrate filtered off, washed, ignited, and weighed. Manganese is precipitated from the filtrate by means of bromine, filtered off, dissolved in hydrochloric acid, reprecipitated with ammonia and ammonium phosphate, boiled, filtered, ignited, and weighed as manganese pyrophosphate.

Ferro - tungstens sometimes contain chromium and aluminium. When this is the case, after the tungsten has been separated by evaporating to dryness, the chromium and most of the aluminium are precipitated by ammonia, filtered off, ignited, weighed, and then separated by dissolving in concentrated nitric acid, with the addition of 4 or 5 grms. of potassium chlorate. The chromium is converted into chromate, and the aluminium may be re-precipitated by ammonia, filtered off, ignited, and weighed. The chromium may be determined in the filtrate, either by adding nitric acid and titrating with ferrous sulphate and permanganate, or by reducing in acid solution with sodium peroxide, and determining as chromic oxide. If aluminium be present, part of it occurs in the iron precipitate, and must be allowed for.

Carbon is determined separately by fusing in a tall, narrow, covered crucible with sodium peroxide, and determining the carbonate formed; but a correction is necessary, as the peroxide always contains a certain amount of carbonate.—A. S.

*Zinc, Electrolytic Estimation of.* H. Paweck. Zeits. f. Elektrochem. 1898, **5**, [18], 221—224.

The author has adopted the following method for the electrolytic determination of zinc. The cathode consists of one or two brass wire gauze discs 6 cm. in diameter supported on a brass wire, about 10 cm. long and 1 mm. thick, which is pushed through the central mesh. When two discs are employed they are kept about 12 mm. apart. The wires composing the disc are about 0·5 mm. in thickness, and the size of each mesh is about 1 sq. mm. Before fitting the brass disc or discs to the supporting wire, they are thoroughly cleaned with damp chalk, washed with water, and placed for a short time in dilute sulphuric acid, until they show a pure yellow colour. The prepared clean cathode is next electrolytically amalgamated once for all by suspending it in a bath containing in 200 c.c. about 0·6 grm. of mercuric chloride and 3—5 c.c. of concentrated nitric acid. In an hour a current of 0·1—0·2 ampère deposits sufficient mercury on the cathode. The amalgamated electrode is then washed with dilute hydrochloric acid, water, alcohol, and ether, and finally dried and preserved on a watch glass in a desiccator. As anode a perforated platinum electrode is employed.

The electrolytic estimation may either take place in an alkaline or an acid solution. Test analyses are given to show the value of the method. The volume of the electrolyte was, in general, 200 c.c. and it contained—

| Alkaline Electrolyte. | Acid Electrolyte. |
|---|---|
| 0·18–0·31 zinc as $ZnSO_4$. | 0·23–0·31 zinc as $ZnSO_4$. |
| 7–12 grms. Rochelle salt. | 6–14 grms. $K_2SO_4$ or $Na_2SO_4$. |
| 5–8 „ KOH or NaOH. | 3–4 drops concentrated $H_2SO_4$. |

The E.M.F. recommended is 3·6 volts. When the electrolysis is complete the cathode is washed in distilled water and then with alcohol, and finally with ether and dried at a low temperature.

After removing the deposited zinc by treating with nearly concentrated hydrochloric acid, the electrode may be used over again.

The results of the test analyses are reported as the percentage amount of zinc in $ZnSO_4.7H_2O$, and show the presence of from 22·49 per cent. to 22·76 per cent. Zn, instead of the theoretical quantity, 22·68 per cent.—J. S.

*Zinc and Manganese as Sulphide, Estimation of.* E. Murmann. Monat h. fur Chem. 1898, **19**, [7 and 8], 404—405.

The estimations of zinc and manganese by precipitation as sulphide and ignition in a current of hydrogen give accurate results, but are unsatisfactory because of the difficulty of obtaining a clear filtrate and the long duration of the washing. If the precipitation be made in the presence of mercuric chloride, the finely-divided zinc or manganese sulphide is carried down along with the mercuric sulphide and quickly deposited. A clear filtrate is obtained, and with careful washing, the precipitate does not go through the filter. With small quantities of precipitate it is expedient to close the pores of the filter with precipitated mercuric sulphide washed by decantation. The mercuric sulphide is readily and completely removed in the subsequent ignition. The operation is best conducted in the author's "tube-crucible." (See page 1181.) The test analyses quoted are satisfactory. The test manganese estimations were made under similar conditions to those which obtain in iron analysis.—A. C. W.

*Chili Saltpetre, Allowable Quantity of Perchlorate in.* Maercker. Landw. Vers.-Stat. **51**, 39; Chem. Centr. 1898, **2**, [18], 935.

*See under* XV., *page* 1160.

*Basic Slag, Estimation of Citrate Soluble Phosphoric Acid in.* J. Freundlich. Chem. Zeit. 1898, **22**, [92], 974.

P. Wagner has found, from experiments comparing the new citric acid solvent (containing 2 per cent. of $C_6H_8O_7 + H_2O$) with the old citrate solution, that the results obtained with the former correspond better with the values found by means of field experiments. The acid extract is precipitated by magnesia mixture containing citrate, and it is stated that this procedure hinders the precipitation of silica, which occurs when the phosphoric acid is precipitated by successive additions of alkaline citrate solution and magnesia mixture.

Wagner, in his new process, has failed to provide for the error due to the contamination of the ignited precipitate by oxide of iron, which occurs when the original slag contains much sulphide of iron. In such cases the author recommends a double precipitation, the sulphide of iron, which separates at first, being oxidised with nitric acid or aqua regia.—A. S.

*Phosphates, Reynoso's Method for the Analysis of.* U. Antony and G. H. Mondolfo. Gaz. chim. ital. **28**, [2], 142—147; Chem. Centr. 1898, **2**, [18], 941.

According to this method, the phosphoric acid combined with alkaline earths is removed by treating the precipitate obtained by ammonia with tin and nitric acid. The disadvantages are that some tin goes into solution, and that much iron and some chromium, manganese, and aluminium are precipitated along with the metastannic acid, and so escape observation. The authors find that by using nitric acid of 77 per cent. strength (sp. gr. 1·427), or even stronger acid, the first difficulty is removed, and in order to avoid that arising from losses of Fe, Cr, and Mn, these metals are tested for before removal of the phosphoric acid: iron by ferrocyanide, manganese by lead peroxide and nitric acid, and chromium by boiling with permanganate and nitric acid, and testing for any chromic acid formed. The main quantity of the precipitate is treated with 77 per cent. nitric acid and tinfoil, cooled, filtered, and excess of ammonia added. The precipitate contains any aluminium presnet, and the filtrate, the alkaline earths. From the precipitate which, in presence of phosphoric acid, is formed by tin and nitric acid, concentrated hydrochloric acid dissolves a compound which contains the phosphoric acid in the ratio $PO_4 : 7Sn$, is soluble in alkalis, precipitated from alkaline solution by carbon dioxide, and is called by the authors phosphostannic acid. Tin and nitric acid react in presence of iron to form a compound slightly soluble in hydrochloric acid, and soluble in cold alkalis, but precipitated as ferric hydrate on boiling the alkaline solution. If the reaction takes place in presence of phosphoric acid, ferric phosphostannate appears to be formed.—A. S.

*Sulphuretted Hydrogen, Sulphurous and Thiosulphuric Acids, Quantitative Determination of.* W. Feld. Die. Chem. Ind. 1898, **21**, 372—380.

The methods described are applicable to the alkali or alkaline earth salts of the above acids, even when present in small quantities:—

(1.) *Sulphides.*—Alkali or alkaline earth sulphides evolve the whole of their sulphur as sulphuretted hydrogen when boiled with a concentrated solution of magnesium chloride in an atmosphere of carbon dioxide. The powdered and moistened sample is placed in a 300 c.c. Erlenmeyer flask provided with a doubly-bored rubber stopper. Through one hole a small tap-funnel passes to the bottom of the flask, through the other a glass tube leads to four sets of potash-bulbs in series. The last of these is connected to a 10-litre bottle acting as aspirator. The neck of the tap-funnel is connected to a supply of carbon dioxide, which must have no action on a solution of iodine. The first set of potash bulbs is empty, the second and third contain rather more iodine solution than will suffice to absorb all the $H_2S$ evolved, the fourth contains $\frac{1}{10}$N thiosulphate solution, to take up any iodine carried over by the carbon dioxide. About 1 litre of carbon dioxide is first passed, in order to displace the air in the apparatus, the tap of the funnel is then closed, and some 20 c.c. of 25 per cent. magnesium chloride solution introduced. Connection is now made to the supply of $CO_2$, and the magnesium chloride solution run into the flask, the contents of which are slowly heated to boiling in a current of carbon dioxide passing at the rate of 10 litres in three-quarters of an hour. The operation is usually ended when 5 litres have passed. The contents of the potash bulbs are finally washed out and titrated; the reactions are—

$$BaS + MgCl_2 + CO_2 + H_2O = BaCl_2 + MgCO_3 + H_2S, \text{ and}$$
$$H_2S + 2I = 2HI + S.$$

Test analyses with $BaSH.OH + 5H_2O$ gave good results.

(2.) *Sulphites* are determined in the same apparatus and in the same way, hydrochloric acid taking the place of magnesium chloride.

(3.) *Thiosulphates* evolve some sulphuretted hydrogen when treated with hydrochloric acid. The following method is found, however, to give accurate results:—The thiosulphate is first converted (by titration with iodine solution) into tetrathionate. The solution of the tetrathionate, diluted with 50 c.c. of water, is placed in the flask with excess of aluminium foil, and treated, in an atmosphere of carbon dioxide, with *dilute* hydrochloric acid in the cold. The reduction to sulphuretted hydrogen, which is collected as before, takes place quantitatively according to the equation—

$$Na_2S_4O_6 + 20HCl + 6Al =$$
$$2NaCl + 3Al_2Cl_6 + 6H_2O + 4H_2S.$$

(4.) *Thiosulphate in presence of Sulphite.*—This determination is made by method (3). The titration with iodine oxidises the sulphite into sulphate, which is not affected by nascent hydrogen.

(5.) *Sulphite in presence of Thiosulphate.*—Excess of mercuric chloride is added to the substance; the thiosulphate is thus converted into mercuric sulphide—

$$Na_2S_2O_3 + HgCl_2 + H_2O = Na_2SO_4 + HgS + 2HCl,$$

whilst the sulphite is not affected and is determined by (2).

(6.) *Sulphide, Sulphite, and Thiosulphate.*—The sample is first distilled with magnesium chloride, as described in (1). This gives the sulphide. The potash bulbs are then refilled, excess of mercuric chloride added to the cold contents of the flask, which are then distilled with hydrochloric acid, as described under (2). This gives the sulphite. The thiosulphate is determined in a fresh sample by titrating with iodine, by which the sulphide is oxidised to sulphur and the sulphite to sulphate, and then reducing by nascent hydrogen, as described under (3).

When, in addition to the alkali or alkaline earth salts of the acids considered, the substance contains polysulphides, free sulphur, and sulphides of the heavy metals, the difficulties are much greater, and the author promises further information. In the meantime he has obtained satisfactory results, as follows:—Free sulphur is extracted by carbon bisulphide, and weighed after evaporation of the solvent. The sulphur present as sulphide is then determined by method (1). In this operation the sulphur of the polysulphides is evolved partly as sulphuretted hydrogen, the remainder separating in the free state. The latter part is extracted by carbon bisulphide. If a sulphite is present, however, some thiosulphate is formed. The solution is now titrated with iodine, during which operation the sulphur present as ferrous sulphide separates in the free state and is extracted with carbon bisulphide. The solution is now treated by method (3) to determine the thiosulphate. The presence of other polythionic acids introduces an error here. In solid substances sulphites may occur in presence of polysulphides; in this case they are determined by treatment with mercuric chloride and distillation with hydrochloric acid, according to method (2).—T. E.

*Cyanide Solutions, Estimation of Oxygen in.—II.* A. F. Crosse. J. Chem. and Metall. Soc. of S. Africa, **1**, 1898, 125—127.

This is a continuation of a former paper (this Journal, 1898, 1074), and describes a modification of the author's previous process that has been rendered necessary by the unexpected discovery of small quantities of nitrites in the cyanide solutions. The following reactions occur in the pipette, and after the addition of thiosulphate and starch solutions:—

$$H_2SO_4 + KNO_2 = KHSO_4 + HNO_2.$$
$$2HNO_2 + 2HI = 2H_2O + I_2 + N_2O_2.$$
$$N_2O_2 + H_2O + O = 2HNO_2.$$
$$HCy + I_2 = ICy + HI.$$
$$2Na_2S_2O_3 + I_2 = 2NaI + Na_2S_4O_6.$$
$$HI + ICy + 2Na_2S_2O_3 = HCy + 2NaI + Na_2S_4O_6.$$

The process, therefore, is now as follows:—Add potassium hydroxide, and then zinc sulphate; determine the thiosulphate required by Thresh's method with clear solution decanted from precipitates formed in the closed bottle; make a qualitative test for nitrites by acidifying a little of the clear solution with dilute sulphuric acid, and adding potassium iodide and starch; and finally apply a correction for the nitrites and reagents used. To make this correction, pour into a very strong 350-c.c. flask, a quantity of solution equal to that used in the experiment (say, 293 c.c.), add a few drops of potassium hydroxide, and close the flask with a rubber stopper having one perforation, through which is passed a glass tube with a glass stopcock. Boil the solution for a few minutes and close the stopcock. Cool the flask, and, when cold, pour the liquid into the pipette, and add the 1 c.c. of iodide-and-nitrite solution and 1 c.c. of sulphuric acid (1:1). Then let it stand for 10 minutes, and, in the presence of coal-gas, run it into the bottle described in the previous paper, add starch, and titrate with thiosulphate. The quantity required gives the correction for nitrites and for the reagents, as the same amount of acid and of iodide and nitrite solution is used in each case.

—W. G. M.

*Hydroxylamine and Hydrazine, Separation of Metals in Acid or Ammoniacal Solutions by.* P. Jannasch. Ber. **31**, [14], 2377—2494.

From 0·2 to 0·4 grm. each of copper sulphate and mercuric chloride are dissolved in 50 c.c. of water, and 1 grm. of tartaric acid is added, followed by 25 c.c. of strong ammonia solution and 20 c.c. of 10 per cent. solution of hydroxylamine hydrochloride. The liquid is heated for 15 minutes on asbestos millboard, shaking occasionally. Metallic mercury is thus precipitated, and, after cooling, and standing for some hours, is filtered and washed, then oxidised by fuming nitric acid, evaporated to dryness, the residue taken up by hydrochloric acid, and the mercury precipitated from the diluted solution at 70° or 80° C. as sulphide. The copper in the ammoniacal solution is determined by one of the usual methods. Separations of mercury from other metals of the copper, arsenic, and iron groups, are similarly executed, save that in certain cases, oxalic acid can be substituted for tartaric acid, and is preferable if the subsequent treatment of the filtrate from the mercury requires the destruction of the acid by heat.

Selenates and tellurates can be separated from sulphates or from phosphates by the same reagent; selenium is precipitated on boiling the acid solution of the hydroxylamine salt with the mixed salts, but the telluric solution must be made strongly ammoniacal after adding the reagent, and long heating is required to reduce to tellurium the whole of the tellurous salt formed. This difference affords a means of separating selenium from tellurium; but, to ensure accuracy, the mixed selenate and tellurate solution must be concentrated and strongly acid, solid hydroxylamine salt must be added (8—10 grms.), and the whole boiled for an hour with reflux condenser till the selenium collects and leaves a clear fluid above. The liquid is filtered, and the filtrate is then, after adding 1 or 2 grms. of the hydroxylamine salt, made strongly ammoniacal, 6 or 8 grms. more of hydroxylamine salt added, and the whole boiled, making up for loss of ammonia and hydroxylamine, till the tellurium has completely fallen.

Tellurium and palladium are separated by volatilising the tellurium at a red heat in a stream of bromine and carbon dioxide, the tellurium bromide in the receiver being oxidised and the tellurium precipitated as above, while the palladium remaining in the tube is determined by one of the usual methods.

The selenium from barium selenate was also completely precipitated by hydroxylamine in acid solution.

Hydrazine precipitates both seleniom and tellurium more easily than does hydroxylamine; but while selenium comes down even from nitric acid solution, tellorium does not. These facts form the basis of a satisfactory and accurate method of separation of selenium from tellurium.—J. T. D.

## *ORGANIC CHEMISTRY.—QUALITATIVE.*

*Sugar Products, Qualitative Test of Alkalinity or Acidity of.* J. Zamaron. Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 295.

Instead of testing the alkalinity or acidity of dark-coloured sugar products with litmus paper, it is better to add a small quantity of a 20 per cent. solution, with a miniature pipette, to drops of neutral litmus solution on a porcelain plate.

—L. J. de W.

*"Saccharin" in Beer, Detection of.* Wochenschr. für Brau. 1898, **15**, [45], 608—609.

In testing for saccharin by Herzfeld and Wolff's method (this Journal, 1898, 802), all the saccharin is not extracted, and when only a very little is present, it is liable to be entirely overlooked. Bruylant's method is more delicate, and the following modification of it showed the presence of saccharin in cases where the former method failed to detect it. At least 1 litre of beer is treated with an excess of sodium bicarbonate, concentrated on the water-bath to a thin syrup, and poured into 500 c.c. of boiling 95 per cent. alcohol. After cooling, the liquid is poured off from the precipitate, and the latter is again treated with boiling alcohol, and allowed to stand over-night. The united alcoholic extracts are then evaporated on a water-bath, and the residue is dissolved in 100 c.c. of boiling water. The solution is cooled, strongly acidified with phosphoric acid, and shaken with ether for several hours, as saccharin is only slowly extracted from its aqueous solution. After evaporation, the saccharin or dulcin can be detected in the the residue by taste and sugar estimations, or by Herzfeld's sublimation method; glycerin cannot appear in the ethereal residue.

A quick and easy method for the detection of saccharin, &c. in very sweet beers, is to determine the reducing and cane sugars present, and then to make up a solution of cane sugar of the strength indicated by the sum of these. If the beer has a sweeter taste than this sugar solution, the presence of foreign sweetening matters may be safely assumed.

—J. F. B.

*Iodoform, Some Reactions of, and Detection of, in Aqueous Liquids.* L. v. Stubenrauch. Zeits. Untersuch. f. Nahrungs- u. Genussmittel, 1898, **1**, [11], 737—741.

On warming iodoform powder with a dilute 1 per cent. solution of silver nitrate, there is an evolution of carbon monoxide and a precipitation of silver iodide—

$$CHI_3 + 3AgNO_3 + H_2O = CO + 3AgI + 3HNO_3.$$

The same reaction takes place without warming the mixture when a stronger (10 per cent.) solution of silver nitrate is used. On triturating solid silver nitrate and iodoform in a mortar, a reaction occurs with almost explosive violence, with the formation of silver iodide and probably carbon dioxide, and the liberation of dense clouds of oxides of nitrogen and iodine vapour.

Daccomo (Gazzetta, 1876, **6**, 247) showed that powdered iodoform decomposed in air or oxygen in the presence of sunlight with the liberation of carbon dioxide, but that when the air or oxygen in the flask was replaced by carbon dioxide or nitrogen, no decomposition took place. The author has made experiments to determine the influence of light on iodoform suspended in water, from which he concludes that its decomposition under such conditions is largely influenced by the intensity of the light, and that in the decomposition, free iodine and (probably) hydriodic acid are formed, both of which go into solution.

The principal tests for the detection of iodoform in aqueous liquids are: (1) The odour, which may be masked by other substances. (2) The microscopical appearance of the crystals obtained from an alcoholic or ethereal solution (hexagonal plates)—a test which Lustgarten (Monatshefte, 1882, **3**, 715) has shown to be liable to error. (3) The light test, in which the iodoform in chloroform solution is rapidly decomposed on exposure to the light with the liberation of free iodine. This test is only applicable when hydriodic acid and alkali iodides have been proved to be absent; and (4) Lustgarten's modification of the Reimer-Tiemann reaction. One or two drops of the alcoholic iodoform solution are added to a small quantity of an alkaline solution of phenol (phenol, 20 parts; NaOH, 40 parts; water, 70 parts), and the liquid gently warmed. A reaction occurs in which salicylic aldehyde is produced, and as the result of a side-reaction a red precipitate separates out and dissolves in the dilute alcohol, forming a carmine-red solution. This test can be used in the presence of potassium iodide, but in the author's opinion it has the drawback of being complicated, and not applicable under all circumstances.

The author bases a simple test for iodoform in suspension or dissolved in aqueous liquids on the fact that although it is decomposed by fuming nitric acid, it is stable in the presence of acid of sufficient strength to decompose hydriodic acid and alkali iodides. Thus, on adding *one drop* of fuming nitric acid to from 3 to 5 c.c. of an aqueous solution of potassium iodide, hydriodic acid, or an easily decomposable organic iodine compound, and then a little starch paste, there is an immediate blue coloration, whereas when iodoform is treated in exactly the same way the starch remains colourless. But if iodoform, in suspension or solution in water, is reduced by warming the liquid with one drop of glacial acetic acid and a little zinc dust, the filtrate, on the addition of *one drop* of fuming nitric acid and starch paste, gives a deep blue colour. If hydriodic acid or potassium iodide are present in only small quantities, so that the test before the reduction gives only a light blue colour, iodoform can be detected in the same liquid by the very much darker blue obtained in the filtrate after reduction. When, however, the first test gives a very dark blue, it is impossible to subsequently detect the iodoform by this reacton. The presence of albumin to any considerable extent also interferes with the test. In the practical application of the reaction, sodium nitrite and sulphuric acid may be used instead of fuming nitric acid. By means of this test the author has confirmed Lustgarten's statement that iodoform taken into the human system is given off in the urine in the form of alkali iodide or an organic iodine compound, but not as iodoform.—C. A. M.

*Indicators, Study of, on Sugar Juices and Syrups.* Guibal, Bull. de l'Assoc. des Chim. de Sucr. et de Dist. 1898, **16**, [3], 288—290.

A series of indicators were examined on lime water, second carbonation juice not sulphited, syrup not sulphited, a syrup sulphited. These were examined both in daylight and by gaslight, compared with the results given by litmus.

With lime water, litmus, cochineal, orcein, and methyl orange agree, while phenolphthalein and rosolic acid show lower results. With juices and syrups, phenolphthalein and methyl orange give very discordant results. Before the tests all the indicators were rendered sensitive, and neutralised.

With phenolphthalein the alkalinity is always lower than with litmus. Methyl orange gives excessively high figures. The alkalinity given by this indicator is about five times as much as it really is. It does not change sharply with juices and syrups, and does not give a distinct tint as with lime water, and the results are not to be compared with those of other indicators

Cochineal gives results slightly high. The change is pretty sharp, but less than with litmus both in daylight and by gas.

Orcein and litmus give results sensibly the same, but the change with orcein is less sharp than with litmus.

Results obtained with rosolic acid are inferior to those with litmus.

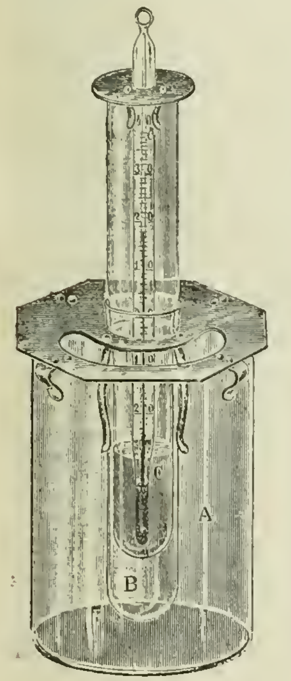
The differences between each indicator and litmus are a little greater in daylight than by gaslight, with the exception of orcein, where the reverse holds good. Litmus, when well prepared and rendered sensitive, is thus more reliable than the other indicators examined.—L. J. de W.

*Pyramidon (Dimethylamido-antipyrin), Detection of, in Urine.* A. Jolles. Zeits. anal. Chem. 1898, **37**, 441—442.

When a weak solution of iodine (a 10 per cent. solution in alcohol, diluted with 10 times its volume of water) is poured on the surface of, but not mixed with, urine containing pyramidon, a well-marked violet-red ring forms at the surface of demarcation, and gradually changes to red-brown. This reaction is characteristic.—H. T. P.

*Solidifying Point of Essential Oils, Method of Determining.* Schimmel's Semi-annual Report, Leipsic, Oct. 1898, 43.

For determining the solidifying point of essential oils the following slight modification of Beckman's apparatus may be used. A glass battery jar, A, is the bath for the freezing mixture or ice. The test tube, B, suspended in the metal cover, forms an air jacket round the freezing tube, C, preventing premature solidification of the oil to be examined. This tube, C, is wider at the top, narrowing down at the point where it rests on the rim of B, and to support it three glass bosses are attached about 5 cm. below the upper rim. The thermometer is graduated in half degrees, and is supported in the loose metal cover by three small springs, so that it may be easily raised or lowered. To make a determination, the freezing bath is filled with ice, or ice and salt, according to the temperature requisite; the oil to be tested is introduced into the freezing tube, C, to the height of about 5 cm., and the thermometer so inserted that it does not touch the sides of the tube. In the case of oils containing anethol, or similar bodies, all jarring or movement during cooling should at first be avoided, and the oil should be free from suspended particles, otherwise a premature solidification may take place. With such oils as those of anise and fennel, when the oil has reached about 10° below the normal point of solidification, crystallisation is induced by gently rubbing the side of the tube with the thermometer, or by introducing a minute portion of the solidified oil. Rapid crystallisation ensues, together with a considerable rise of temperature, the maximum point of which is taken as the solidifying point of the oil.—J. O. B.



## ORGANIC CHEMISTRY.—QUANTITATIVE.

*Oxygen in Illuminating Gas, Estimation of.* H. Lubberger. J. für Gasbeleucht. 1898, **41**, [43], 695—696.

An exact method of estimating small quantities of oxygen in illuminating gas is needed when air is added to the crude gas, in order to revivify continuously the purifying material. Pfeiffer's method of estimation (J. für Gasbeleucht. 1897, 356; this Journal, 1897, 635) has the disadvantage of needing an empirically prepared caramel solution, which will scarcely remain constant for a length of time. The method described below is a modification of Winkler's process for the determination of the oxygen dissolved in water. The Bunte burette is the apparatus used. The following solutions are required:—(1) "Solution of potassium iodide," of which 300 c.c. contain 10 grms. of caustic soda, 35 grms. of crystals of Rochelle salt, and 8·5 grms. of potassium iodide. (2) "Manganese solution," made by dissolving 14·5 grms. of manganese sulphate ($MnSO_4 + 5H_2O$) in 100 c.c. of water. (3) Centinormal solution of thiosulphate, of which 1,000 c.c. contain 2·480 grms. of sodium thiosulphate ($Na_2S_2O_3 + 5H_2O$). Water freed from air by Pfeiffer's method (this Journal, 1897, 635) is also required.

If the gas to be tested is under pressure, the burette is filled with water, and the gas is admitted at the upper three-way cock until the water is expelled. The lower cock is closed, and the upper is turned through half a revolution, so that gas escapes until the contents of the burette are at the atmospheric pressure. The flexible tube by which the gas was admitted is then removed and replaced by a short piece of flexible tube, plugged by a piece of glass rod. 5 c.c. of air-free water are then put into the funnel of the burette, and the water is allowed to flow in until it stands 1 c.c. above the lower cock. The upper cock is then closed by turning it to its original position. The lower cock is opened and water is allowed to flow out until only the capillary remains filled. If, on the contrary, the sample of gas is aspirated into the burette, the transverse way of the three-way cock should be first filled with water.

The burette is then placed in its clamp, and air-free water is forced in through the lower cock until it stands about three marks below the zero point, while the surplus gas escapes through the three-way cock and the filled funnel. The level is then read, the confining water is drawn out, and the funnel is emptied. If the gas contains sulphuretted hydrogen, 2 c.c. of solution of iodine (5 grms. of iodine and 10 grms. of potassium iodide dissolved in 100 c.c. of water) are admitted by the lower cock, the burette is agitated for some time, the solution is then drawn out, and traces of it are removed by running through air-free water until the outflowing water no longer reacts with solution of starch. If the gas is free from sulphuretted hydrogen the treatment with solution of iodine is omitted. 1 c.c. of manganese solution is admitted at the top, and 3 c.c. of solution of potassium iodide at the bottom of the burette. The burette is then grasped at its middle by the thumb, forefinger, and middle finger, and agitated for 10 minutes, so that at each movement of the hand the liquid is tossed from one end of the burette to the other. 1 c.c. of 25 per cent. hydrochloric acid is then admitted at the bottom, and the burette again agitated. If the gas contained oxygen, the liquid will now be yellow from the separated iodine. The funnel is filled with distilled water and the contents of the burette are washed into a beaker, and a little solution of starch added. The liquid is then titrated with centinormal thiosulphate until the blue colour disappears. After deducting 0·3 c.c. from the amount of thiosulphate taken, the remainder will give the volume of oxygen in the gas, according to the ratio 1 c.c. of thiosulphate solution = 0·06 c.c. of oxygen. The titration may, if preferred, be performed without washing the liquid from the burette, by admitting water until the liquid rises to the lowest mark, and then adding the centinormal thiosulphate until the yellow colour disappears. In this case, however, 0·6 c.c. should be deducted in place of 0·3 c.c. to allow for the capacity of the nozzle of the burette. The correction in both cases is the mean deduced from a number of blank tests. The method was checked by a number of analyses made on samples of gas prepared by adding air from a pipette graduated to one-hundredth of a c.c. to illuminating gas which had been deprived of oxygen by remaining for 24 hours over alkaline solution of pyrogallol. The following are the quantities of oxygen (in c.c.'s):—

| | | | | | |
|---|---|---|---|---|---|
| Taken .................. | 0·104 | 0·104 | 0·209 | 0·209 | 0·209 |
| Found.................. | 0·099 | 0·111 | 0·204 | 0·186 | 0·210 |

| | | | | |
|---|---|---|---|---|
| Taken .................. | 0·313 | 0·418 | 0·418 | 0·627 |
| Found.................. | 0·297 | 0·427 | 0·409 | 0·657 |

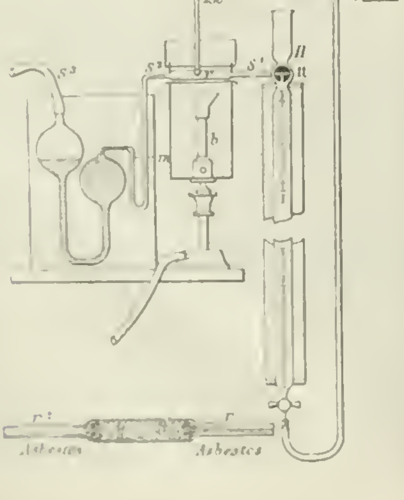
—J. A. B.

*Hydrogen, Methane, and Nitrogen in Mixtures of Gases; A Volumetric Method for the Estimation of, by Fractional Combustion with Cupric Oxide.* E. Jäger. J. für Gasbeleucht. 1898, **41**, [47], 764—766.

The apparatus used is shown in the annexed figure. The Bunte burette has a modified three-way stop-cock as shown. The combustion tube *r* terminates at the end which is connected with the burette in a capillary tube. The other end of the combustion tube is of greater internal diameter, and the tube is charged with powdered cupric oxide from this end. Plugs of asbestos fibre are inserted in the tube to prevent the oxide passing into its narrowed terminals. The tube is placed in an oven of sheet iron attached to the Bunsen burner *b*, and the bulb of a thermometer, the stem of which passes through the top of the oven, is adjacent to the tube. The wider end of the combustion tube is connected with a Hempel's absorption pipette containing solution of caustic potash. The joints at $s^1$ and $s^2$ are made by means of flexible tubing bound over with copper wire. Before commencing an analysis, the tightness of the joints is proved by blowing into the pipette at $s^3$, closing that inlet, and observing that the column of liquid in the capillary does not fall from the position it has taken, until the inlet is reopened, and that it then returns to its former position, which should be about the point *m*.

As soon as the tightness of the joints has been proved, the cock is turned to the position IV., the burner is lighted, and the combustion tube heated to 250°. The cock is then turned to position II., and the gas is passed from the burette slowly through the cupric oxide in the tube. The rate of passage of the gas should be about 100 c.c. in eight minutes, and the passage to and fro over the oxide should be continued until no further contraction is observed. At a temperature of 250° and thereabouts, only the hydrogen in a mixture of hydrogen, methane, and nitrogen is burnt, and the contraction is equal to the volume of hydrogen present. Correction should, however, be made (1) for the decrease in volume of the material in the tube owing to the reduction of a part of the cupric oxide to copper; in technical work, this correction may be neglected; (2) for the volume of oxygen in the air contained in the combustion tube and its capillaries. This oxygen disappears during the combustion. Its volume may, for practical purposes, be ascertained by a preliminary test, and the result used for the correction of the figures found in all subsequent analyses with the same apparatus.

The combustion tube is then heated to redness, and the residual gas is passed slowly through it several times until no further contraction of its volume takes place. The amount of the contraction gives the volume of methane in the gas, and the residue is the nitrogen. Trials showed that only the hydrogen was burnt at about 250°, but that its combustion was complete, while the methane could be subsequently burnt completely at a higher temperature. In analyses of gases, such as illuminating gas, the whole of the gas remaining after the carbonic acid, hydrocarbons, oxygen, and carbonic oxide have been taken up by absorbents, can be used for the estimation of its constituents by this method. In the methods of analysis hitherto generally used, only a portion of this residue is taken for the estimation of the methane, hydrogen, and nitrogen, and the liability to error is great, especially in the case of the nitrogen determination.—J. A. B.



*Waterproofed Cotton Fabrics ("Batist"), A Scheme for the Examination of.* R. Thal. Chem. Zeit. 1898, **22**, 867 and 880.

The following process was devised for the analysis of Mosetig's "batist," a material consisting of cotton impregnated with caoutchouc on one or both sides, which is largely used in the French army for compresses and antiseptic dressings.

The moisture was estimated by drying for one hour at 100° to 105° C. To free the fabric from caoutchouc and obtain it in a state fit for separate examination, four square verschok (1 verschok = $1\frac{3}{4}$ ins.) of the material were boiled with 150 c.c. of nitrobenzene, stirred well for three minutes, removed from the liquid, washed twice with 25 c.c. of 95 per cent. alcohol, and dried. The residue was treated with dilute hydrochloric acid, washed with water till free from chlorine, dried, weighed, and the ash determined. As the ash of cotton is very small in amount, the fabric was assumed not to contain any inorganic constituents, and the fixed matter recovered on incineration was considered to belong to the mineral substances of the caoutchouc mass. The nitrobenzene solution was boiled for half an hour, cooled, mixed with the spirit used for washing the cotton, allowed to rest for two days, and then filtered through a tared paper. The filter and its contents were washed with warm benzene, dried, and weighed. The total sulphur in the original "batist" was estimated, 7 sq. ins. being taken for analysis, as before. The ash was weighed, after combustion in porcelain. A sample was ignited with 2 parts of saltpetre and 1 of sodium carbonate, and the chlorine determined as usual. 7 sq. ins. were boiled for four hours with 8 per cent. alcoholic soda, according to Henriques' method, the insoluble portion being employed for a determination of the combined sulphur and ash. Previous experiments having shown that cotton diminishes in weight when treated in this manner, some of the "batist" fabric cleaned from caoutchouc with nitrobenzene was submitted to the same process, and the loss was found to be 13·59 per cent. of the dry material. Even the filter itself is attacked by the alcoholic alkali; and unless the residue can be manipulated without filtration, the necessary correction must be made. The proportion of caoutchouc proper in the substance could only be ascertained by difference.

The analytical data of the single and double materials were—

| —— | Single. | Double. |
|---|---|---|
| | Per Cent. | Per Cent. |
| Moisture | 3·04 | 2·97 |
| Cotton fabric | 63·58 | 56·54 |
| Sulphur (total) | 0·33 | 0·48 |
| Mineral matter (calculated as $C_6H_5NO_2$ residue). | 8·03 | 13·97 |
| Chlorine (combined) | 0·18 | 0·15 |
| Caoutchouc (by difference) | 24·84 | 25·89 |

The alcoholic soda treatment gave—

| | Per Cent. | Per Cent. |
|---|---|---|
| Insoluble residue | 88·95 | 88·80 |
| Correction for solubility of cotton | 8·70 | 7·86 |
| Sulphur (free) | 0·03 | 0·22 |
| Ash (dissolved) | 0·10 | 0·09 |
| Moisture | 3·04 | 2·97 |
| | 100·82 | 99·94 |

Henriques has recorded the solubility of Para-caoutchouc in 8 per cent. alcoholic soda as 2·5 per cent.; if the corresponding corrections are applied to the above percentages of caoutchouc as yielded by the nitrobenzene, the total matter insoluble in alkali becomes—

| —— | Single. | Double. |
|---|---|---|
| | Per Cent. | Per Cent. |
| —— | 100·82 | 99·94 |
| —— | 0·62 | 0·64 |
| —— | 101·44 | 100·58 |

These figures show that the alkali did not dissolve anything from the "batist," and the trifling excesses over the proper weight are probably due to the high quality of the original rubber. The dried caoutchouc masses gave the following results:—

| | Per Cent. | Per Cent. |
|---|---|---|
| Sulphur (combined) | 0·89 | 0·64 |
| Sulphur (free) | 0·09 | 0·54 |
| Talc | 24·06 | 34·52 |
| Chlorine (combined) | 0·35 | 0·37 |
| Caoutchouc | 74·43 | 63·93 |

The author has already observed that the fabric of a waterproofed article which has been vulcanised by the cold (chloride of sulphur) process loses the whole or part of its tensile strength, and generally turns yellow, when subsequently heated with nitrobenzene; while a similar material, after hot vulcanisation, is practically unaffected. As a mean of several tests, a strip of bleached calico, 18 cm. long by 1·5 cm. wide, was found to have a tensile strength, in Schopper's apparatus, of 13·3 kilos. After boiling with nitrobenzene alone for five minutes, the strength was 13·1 kilos. Boiled for three minutes in the presence of 0·5 per cent. of elemental sulphur, the strength was still 12·7 kilos. 0·005 per cent. of $S_2Cl_2$ reduced it to 6·1 kilos., 0·01 per cent. to 2·7 kilos. The diminution in resisting power therefore is neither due to the sulphur, the heat, nor the nitrobenzene, but presumably depends on the formation of hydrochloric acid from the chloride, which naturally rots the fibres. Examined in this manner, "batist," coated on one side, gave a tensile strength of 7·03 kilos., on both sides of 6·59 kilos., when in its original condition; after boiling with nitrobenzene, the strength fell to 3·25 and 3·26 kilos., respectively. It is accordingly seen to be manufactured by the cold vulcanisation process.—F. H. L.

### *Sumac, Examination of.* Spica. Leipziger Färber- und Zeugdr.-Zeit. 1898, 47, [12], 526.

A METHOD is given by which adulteration of sumac with other leaves may not only be detected, but also quantitatively determined. The method depends on the fact that a decoction of sumac, after treatment with caustic soda and lead acetate, gives a brownish-red solution, which becomes ruby on dilution with water.

5 grms. of the leaves are boiled for half an hour with 1 litre of water. The infusion is cooled, made up to the litre, filtered, and 25 c.c. of the filtrate treated with 5 c.c. of a solution of basic lead acetate of 1·184 sp. gr., and after shaking well, evaporated to 15 c.c. If the sumac be pure, the reddish-brown solution obtained in this way is almost perfectly clear; the presence of a precipitate is an indication that the sample was adulterated with foreign matter. The amount of adulteration is determined colorimetrically by diluting the solution with 250 c.c. of water, and comparing with standard solutions of safranine; pure sumac gives a tint similar to that obtained by dissolving 0·15 grm. of Safranine in 1 litre of water; adulterated samples give fainter tints.—A. S.

### *India-Rubber Substitutes, A Source of Error in Henriques' Method of Estimating.* R. Thal. Mag. Pharm. through the Chem. Zeit. 1898, 22, [92], 974.

IN the estimation of india-rubber substitutes by Henriques' method of extracting with 8 per cent. alcoholic caustic soda solution, an error is caused by the solubility of the filter paper in the liquid. The No. 589 filter papers of 12·5 cm. diameter (Schleicher and Schüll) were found, as a mean of twelve experiments, to lose 5·63 per cent. of their weight on treating with the caustic solvent. It is therefore necessary, when using these filter papers, to add about 5·5 per cent. of the weight of the dried filter to the weight of residue found, in order to get accurate numbers. If other makes of filter-paper are used, their solubility in alcoholic caustic soda must be determined.

Only the residue from india-rubber, containing or covered with little mineral matter, may be weighed without a filter, since the soda solution loosens and carries away small particles.—J. D. G.

### *Beer Worts and other Albuminous Liquids, Quantitative Estimation of the Various Types of Proteids of.* H. Schjerning. Zeits. Anal. Chem. 1898, 37, [7], 413—422.

IN a series of preliminary investigations (this Journal, 1894, 1103; 1895, 763; 1896, 621) concerning the proteids, the author has examined the value of various metallic salts, &c., as precipitating agents for quantitative purposes; and now describes methods for the estimation of proteids based on his experiences. The following reagents are required:—

1. *Stannous Chloride.*—50 grms. of tin are dissolved by heat in an excess of strong hydrochloric acid, to which a trace of platinic chloride has been added. The solution is concentrated until it weighs about 130 grms., and finally diluted with water to 1 litre.
2. *Lead Acetate.*—Solution containing about 10 per cent. of the normal salt, and 10—12 drops of acetic acid (45 per cent.) per litre.
3. *Mercuric Chloride.*—A 5 per cent. solution.
4. Pure dry *Ferric Acetate.*
5. Dilute *Acetic Acid*, containing 15 c.c. of the 45 per cent. acid per litre.
6. *Uranium Acetate.*—A 10 per cent. solution.
7. *Magnesium Sulphate*, in crystals.
8. *Sodium Phosphate* ($Na_2HPO_4.12H_2O$) solution containing 4 grms. per litre.
9. *Calcium Chloride.*—A 10 per cent. solution.

Six precipitations, in as many separate portions of the liquid under examination, are required. Before proceeding with these, however, the solution must be diluted until it contains about 0·07 per cent. of nitrogen; and if alkaline, each portion must be neutralised with the acid corresponding to the particular reagent about to be used. It must also be mentioned that some of the precipitations (with Fe, Pb, and Sn) proceed badly with proteid solutions deficient in mineral constituents. The ferric acetate precipitation furnishes a good criterion on this point. Under the conditions laid down, the whole of the iron should be precipitated on boiling, and none pass into the filtrate. Failing this, the sample must be treated as being poor in ash, and the deficiency made good artificially. The proteid precipitates are most conveniently collected and washed on "ashless" filters (11 cm. diameter), and the nitrogen subsequently determined by Kjeldahl's method in the precipitate *plus* filter, a correction being applied for any N present in the latter. The details of the various precipitations are as under:—

*Stannous Chloride.*—5 c.c. of stannous chloride solution are added to 25 c.c. of the proteïd liquid, and the mixture allowed to stand for 6—20 hours at the ordinary temperature. The precipitate is to be washed with cold water. If the proteïd solution has been found deficient in ash, 10 c.c. of calcium chloride solution (reagent No. 9) should be added, prior to proceeding as above, and the precipitate be washed with a cold 1 per cent. solution of calcium chloride.

*Lead Acetate.*—25 c.c. of the sample is treated with a sufficiency of lead acetate solution, heated to boiling, filtered, and the precipitate washed with cold water. The filtrate should contain lead; but care must be taken to avoid a large excess, in which the precipitate is somewhat soluble. The volume of lead solution required ranges from 0·2 c.c. for egg albumin to 6 c.c. for worts and beers. In the case of "ashless" samples, three volumes of sodium phosphate should be added for each volume of lead solution used, the mixture boiled, &c.; and in either case, a small correction (0·00021 grm. of N per 100 c.c. of filtrate and washings) must be added to the result, to allow for the solubility of the precipitate.

*Mercuric Chloride.*—5 c.c. of the mercuric chloride solution is added to 25 c.c. of the proteid liquid and the mixture allowed to stand 4—20 hours at the room temperature. The precipitate formed is washed with a cold 0·5 per cent. sublimate solution. In the final distillation, a little sodium sulphide must be added, to decompose mercury ammonium compounds.

*Ferric Acetate.*—0·8 grm. of the salt is dissolved in 40 c.c. of dilute acetic acid (reagent No. 5) and 50—100 c.c. of water, the mixture boiled, 20 c.c. of the proteïd solution added, the whole again boiled, immediately filtered, and the precipitate washed 3—4 times with boiling water. The filtrate should be free from iron; if this be not the case, the experiment must be repeated, 15—25 c.c. of sodium phosphate solution being added immediately after the proteid solution, and the mixture kept briskly boiling all the time.

*Uranium Acetate.*—20—25 c.c. of the reagent is added to 25 c.c. of proteïd solution, the mixture heated to boiling, then allowed to stand for some hours at the ordinary temperature, in a dark place. The precipitate is washed with a cold 1—2 per cent. solution of uranium acetate, and the final result corrected for solubility, by the addition of 0·00014 grm. of N per 100 c.c. of filtrate and washings.

*Magnesium Sulphate.*—20 c.c. of the proteid solution, acidified with 5—6 drops of acetic acid, is heated in a water bath to 33°—36° C., 18—20 grms. of powdered magnesium sulphate is then stirred in, and the mixture kept at the above temperature for ½—1 hour, being repeatedly stirred during that period. The resultant precipitate is collected, and washed with a cold saturated solution of magnesium sulphate, containing 4—5 grms. of acetic acid per litre.

The author remarks that the first five precipitations yield results that are too low in the case of proteïd solutions rich in alkaline salts, because these tend to form with the proteids comparatively soluble compounds.

The types of proteids, precipitated respectively by the above reagents, are indicated below:—

| Reagent. | Precipitate contains |
|---|---|
| Stannous chloride = *a*.......... | Albumin I. |
| Lead acetate<br>Mercuric chloride = *b*.......... | Albumin I.<br>" II.<br>Denuclein. |
| Ferric acetate = *c*.......... | Albumin I.<br>" II.<br>Denuclein.<br>Propeptone. |
| Uranium acetate = *d*.......... | Albumin I.<br>" II.<br>Denuclein.<br>Propeptone.<br>Peptone. |
| Magnesium sulphate = *e*.......... | Albumin I.<br>" II.<br>Propeptone. |

From which it follows that the

Albumins I. .............. = $a$
" II. .............. = $b - [a + (c - e)]$
Denucleins .............. = $c - e$
Propeptones .............. = $c - b$
Peptones.................. = $d - c$

Since the precipitations with lead acetate and mercuric chloride yield identical results, only one of them need be employed. In practice the latter is to be preferred, because it can nearly always be carried out successfully, and besides, is independent of the ash contents of sample. The paper is accompanied by a table of results, illustrating these methods. —H. T. P.

*Malt, The Analysis of; Proposals for Uniformity in.* L. Aubry. Third International Congress of Chemists at Vienna, through Wochensch. fur Brauerei, 1898, **15**, 497—499.

The author calls attention to the urgent need of some agreement between chemists as to the standard methods to be used in the analysis of malt, and puts forward a scheme which he thinks might be advantageously adopted.

The sample should be most carefully taken in order to represent a true average of the bulk. About 500 grms. should be sent for analysis, packed in closed bottles, paper parcels being strictly avoided. Full data of the nature and history of the malt should be furnished.

The mechanical analysis comprises the weight of a hectolitre, to be taken on not less than ¼ litre, the weight of 1,000 corns calculated on the dry substance, and particulars as to the colour, size, and uniformity of the corns. Percentage ratios should be determined of the number of mouldy and broken corns and of foreign seeds and the freedom from rootlets and dust. The corns should be graded by means of a sieve with holes of 2·5 mm. The condition of the interior of the corns and the length of the acrospires should be noted and expressed in percentage gradations. Lastly, the taste and smell should be observed.

For the estimation of moisture, 4 to 5 grms. of malt are weighed whole and ground without loss, and then dried for one hour at 80° C., and for three hours longer at 105° C.

The author describes very fully the precise conditions to be observed in the preparation and analysis of the extract. Fifty grms. of malt are ground to a fine meal and placed in a tared beaker with 200 c.c. of water at 45° C. The mixture is kept at 45° C. for half an hour, with constant stirring. The temperature is then regularly raised during half an hour to 70° C., and kept constant for one hour. The necessary stirring is best done by a slow motor. The time taken after arriving at 70° C. until complete saccharification is effected is recorded as the "time of saccharification." The progress of this reaction is observed by performing the iodine test at intervals of 5 or 10 minutes.

After one hour's digestion at 70° C., the mash is mixed with 200 c.c. of cold water, rapidly cooled, and the weight made up to 450 grms. with water. It is then filtered, the time of filtration being noted, and the physical appearance of the wort is observed.

The specific gravity of the filtered wort is accurately taken, and the solid matter calculated by Windisch's tables. The colour is determined by Brand's method of comparison with dilute iodine solution, and the nitrogen, ash, and phosphoric acid are estimated in the usual manner. The maltose is the only carbohydrate which need be determined, and this is done gravimetrically by Wein's method.

The diastatic power of malt is best determined by C. J. Lintner's modification of Kjeldahl's method.

*Note.*—The German abstractor, O. Reinke, remarks that, since the finely-ground meal gives a stronger extract than the coarse, this fact should be stated in the analytical report. He also thinks that the author should have described some standard process for the determination of the lactic acid, and that the estimation of maltose should be supplemented by a fermentation experiment with an active yeast.—J. F. B.

*Hops, Chemical Analysis in the Commercial Valuation of.* Th. Remy. Wochenschr. für Brau. 1898, **15**, [41], 530—536; [42], 556—557; and [43], 583—586.

The industrial value of hops cannot be judged solely from the percentages of the chemically determinable constituents present, as many important characters, perhaps the most important, cannot be quantitatively measured. The analytical factors supposed to influence the action of hops on beer-wort are the hop-resins, the tannin, and the soluble nitrogenous matter. The estimation of these is doubtless an aid to, but not a criterion for the valuation of the hops, and the other unmeasurable qualities, such as aroma and indefinite changes during storage, may completely upset a a valuation based on chemical analysis alone. Again, difficulties occur in the interpretation of the results, and whilst most of the desirable constituents may be fully up to the mark, one may be deficient, and it is difficult to say how far this deficiency may neutralise the good effects of the other constituents.

The author has fully investigated several samples of hops of the 1897 German crop from the last Berlin Exhibition. He also had the samples classified and valued by technical and trade experts, according to the grade of plucking and sorting, growth and lupulin content. He then endeavoured to trace connections between those valuations and the analytical results, but on the whole was unsuccessful. The expert classifications differed amongst themselves to a considerable degree, and except in very marked cases they also differed from the chemical results. Hops of very inferior quality were at the same time deficient in resin and *vice versâ*. From the very few data to hand, the author considers that the well-known superiority of hops from certain districts cannot be attributed to a high percentage of resin, as the variations in resin content from year to year in hops of the same districts are as great as those between hops rich in resin and those poor in the same. The only typical characteristic in this respect is shown by the hops of the Altmark district, which in two consecutive years, only contained 14·5 per cent. of resin, as against 22—18 per cent. in hops from other districts. The poverty in resin appeared to be connected with luxuriance in bearing.

Hayduck and Briant and Meacham have shown that the preservative and bitter principle of hops is the "soft resin," constituting about three-fourths of the total resin, and separable from the "hard resin" by petroleum spirit. Behrens asserts that the quantity of hops usually added to wort is more than sufficient to saturate the wort with "soft resin," even when the hops are only poor in the same, and hence the percentage is of little importance. Hayduck has shown that only about 50 per cent. of the resin is extracted by the boiling wort, but it does not follow that no more resin is extracted from rich hops than from poor ones. The author quotes experiments showing that the quantity of resin taken up by the wort is the same, whether it is added in the hops or in the isolated state, and though only about one-half is taken up, this is true within comparatively wide limits. The retarding effect on the subsequent fermentation is proportional to the resin taken up.

The author confirms Briant and Meacham's observation that the "soft resin" is converted into the "hard resin" by storage, especially at elevated temperatures, and thinks that results of determinations of the former should be accompanied by those of the total resin.

No connection could be traced between the expert classification and the percentage of "soft resin," but the author considers that this percentage is undoubtedly an important guide in judging quality of hops, and a more accurate method is urgently needed for its determination.

The tannin and its derivatives, as shown by Hayduck, precipitate some of the albuminoids of the wort which are not coagulated by boiling, and thus probably exert a preservative influence. Whether the tannin has other favourable effects on the beer, such as clearing, colour and flavour cannot be definitely stated, but the author finds that the kinds of hops most highly prized by brewers, contain the highest proportion of tannin. However, until the effects of hop tannin are properly determined, no definite inference can be drawn.

No connection could be traced between either the total or the soluble nitrogenous constituents and the quality of the hops, and their value is probably only that of yeast nutrients.

The best kinds of hops all contained a high proportion of potash in their ash, a circumstance which the author thinks is not without importance.

The mechanical analysis of the hops is of value in their valuation; a large proportion of axes and seeds indicates coarse hops of little value, not merely on account of their presence as worthless ballast but also on account of the general deficiency of valuable constituents which accompanies them.

As regards the absolute indispensability of Saaz hops for certain beers, the author states that it cannot be due to the preservative effect of the "soft resin" and to the tannin alone. Probably there is a special aroma, or a combination of a whole series of valuable constituents and low colouring power, which are only met with singly in other kinds of hops.

The author calls attention to the fact that more accurate and uniform methods are required for all the determinations connected with hop analysis and systematic investigation of the effects of the individual constituents.—J. F. B.

### *Malt, The Analysis of.* O. Reinke. Wochensch. für Brau. 1898, **15**, [42], 555.

The samples must be carefully taken from all parts of the bulk, and immediately placed in corked bottles to prevent loss of moisture and odour. About 1 lb. should be sent for analysis. Two methods are in vogue for carrying out the estimations: according to one, the malt is coarsely crushed, whilst in the other case it is ground to a very fine powder. The latter method was approved by the Vienna Congress of 1898. In this case, the solid matter in the extract is deduced by means of Windisch's tables from the specific gravity at 15° C. From the fine meal, from 1 to 4 per cent. more solid matter is extracted than from the coarse, and, whilst the maltose is from 1 to 3 per cent. less, the colour of the extract is greater to the extent of 10 to 20 c.c. of centinormal iodine solution per 100 c.c.

The yield of extract on the large scale is from 3 to 8 per cent. lower than that obtained on analysis of the fine meal, the differences found with good bright malt being less than with inferior samples.—J. F. B.

### *Wine, Determination of Total Acidity in.* E. Sellier and A. Vivien. Ann. Chim. anal. appl. **3**, 304—307. Chem. Centr. 1898, **2**, [16], 873.

For determining the total acidity in wine, the author recommends the method used by G. Brühns (Centralbl. f. Zucker Ind. d. Welt, 1897, 155) for the estimation of the acidity in press-, and diffusion juice.

100 c.c. of the wine are treated with 10 c.c. of lead acetate solution (300 grms. of neutral lead acetate per litre), the mixture shaken and filtered, any carbon dioxide present in the wine having previously been removed in a vacuum or by agitation. 50 c.c. of the clear filtrate are well mixed with 5 c.c. of a saturated solution of sodium sulphate, the whole filtered, and 25 c.c. of the faintly coloured filtrate, titrated, after adding 5 c.c. of phenolphthalein with $\frac{1}{10}$N NaOH (1) till the colouring matter of the wine changes to green, and (2) till the appearance of a permanent rose colour. A blank experiment should be carried out with the lead acetate solution, and correction made, if the latter be not perfectly neutral. The c.c. of alkali used, multiplied by 48·4 gives the c.c. required per litre of wine. The difference between the amounts of alkali used in the two stages of the titration should be an indication of the amount of colouring matter present in the wine. The results obtained by the above method only correspond approximately with those by the spot method with litmus paper, and the direct titration with phenolphthalein, differences of as much as six units occurring in the second place of decimals.—A. S.

### *Mustard Oil from Seeds of the Cruciferæ, Conditions of Development and Determination of Volatile.* G. Jörgensen. Nyt. Tidskrift for Fysik ok Kemi. **3**, 91—111; Chem. Centr. 1898, **2**, [18], 927.

The author used for his experiments partly commercial so called rape-seed cake, but consisting of *Brassica Napus* and *Br. Rapa*, with more or less of foreign kinds of Brassica, and also containing Sinapis-, Eruca-, and Erysimum seeds; and partly seeds of pure cultivated or unmixed cruciferæ, *i.e.*, *Brassica nigra*, *Br. Napus*, *Br. juncea*, *Br. dichotoma*, *Br. ramosa*, and *Br. glauca*.

Pure black mustard, on agitation with water for one hour at 15° C., develops the full quantity of mustard oil, but by longer digestion, especially at a higher temperature, certain losses occur. Ordinary commercial press-cakes require an addition of myrosin in order to develop the full quantity of mustard oil in one hour.

The mustard oil was determined by distilling the powdered substance with steam, and leading the distillate into a solution containing 30 grms. of potassium permanganate and 15 grms. of caustic potash per litre. The sulphuric acid formed was precipitated as barium sulphate, and the equivalent of allyl mustard oil calculated. As, however, the sulphuric acid formed may be partly due to substances other than the mustard oil, the author recommends collecting the distillate in a receiver containing 20 c.c. of strong ammonia water and 50 c.c. of spirits of wine, allow to stand overnight, evaporate the mixture gently till the vapours no longer react alkaline, cool, add 10 c.c. of sulphuric acid, and, after again evaporating, determine the nitrogen by Kjeldahl's method; 2N = 1 mol. of mustard oil.

Food-cakes that had exhibited a corrosive action on the digestive organs of cows showed no particular difference in the yield of mustard oil (0·8—1·0 per cent.) from the press-cakes made from Indian rape. Cakes in which Indian rape was not detected, yielded 0·61 per cent. after addition of white mustard. Pure rape yielded 0·22 per cent. of mustard oil; *Sinapis arvensis* and *Erysimum orientale* on the other hand, not a trace. The author examined the thiosinamine formed from the mustard oil in the different cases, and found that it often (with *Sinapis nigra, Brassica juncea*) agreed in composition with allyl thiourea, $CS.NH_2.NH.C_3H_5$. In other cases—from *Br. glauca, Br. dichotoma*, &c.,—the thiosinamine obtained was a crotonyl thiourea, $CS.NH_2.NH.C_4H_7$.—A. S.

*Ethyl Aldehyde, Volumetric Estimation of.* X. Rocques. J. Pharm. Chim. 1898, **8**, [9], 390—394.

Ethyl aldehyde in very dilute alcoholic solution is determined with sufficient accuracy by means of the rosaniline bisulphite reaction (this Journal, 1897, 265). This process is very delicate, and if employed for solutions of 0·1—2 per cent. strength, must be preceded by large dilution. The author has devised a method for solutions of such strength. Rieter's method consists in adding a solution of sulphurous acid, letting stand for four hours, dividing into two parts, estimating the free sulphurous acid in one part by $\frac{N}{100}$ iodine solution, decomposing the aldehyde-bisulphite compound in the other portion by aqueous potash in the cold, and then determining the total sulphurous acid after acidifying. Aqueous solutions of sulphurous acid, alkali sulphites and bisulphites are very much less stable than alcoholic solutions, an alcoholic bisulphite solution appears to be most suited to the titration of ethyl aldehyde. When the strength of the aldehyde solution reaches 1 per cent., the reaction with the bisulphite is not complete in four hours, but requires twelve. In the case of strong solutions, a considerable proportion of the aldehyde compound is not decomposed by the potash, and thus in Rieter's method a considerable error results.

The following is the proposed method. Dissolve 12·6 grms. of pure sodium sulphite in 400 c.c. of water, add 100 c.c. of N-sulphuric acid and make up to 1 litre with 96 per cent. alcohol. Let stand 24 hours and filter. Titrate the solution so obtained with $\frac{N}{10}$ iodine solution. Add 50 c.c. of the sulphite solution to 10 c.c. of the aldehyde solution, make up to 100 c.c. with 50 per cent. alcohol and shake. At the same time dilute 50 c.c. of sulphite solution with 50 c.c. of the same dilute alcohol. Let both flasks stand 24 hours at the ordinary temperature, shake again, withdraw 50 c.c., and titrate with $\frac{N}{10}$ iodine solution. From the difference in the amount of iodine required in the two cases, the aldehyde content is calculated. If the solution to be examined contain less than 1 per cent. of aldehyde, the sulphite and iodine solutions are to be diluted accordingly. Paraldehyde does not combine with sulphurous acid.

—A. C. W.

*Caramel [Sugar]; Chemical Nature, Occurrence in the Sugar Factory, and Determination of.* N. Fradiss. Bull. de l'Assoc. de Chim. de Sucr. et de Dist., 1898, **16**, [3], 280.

*See under* XVI., *page* 1162.

## XXIV.—SCIENTIFIC & TECHNICAL NOTES.

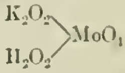
*Calcium Amalgam.* J. Ferée. Comptes Rend. 1898, **127**, [17], 618—620.

The author prepares the amalgam by the electrolysis of a solution composed of equal parts of crystallised calcium chloride and water. The amalgam formed must be protected from the action of the chlorine by a porous diaphragm, and the salt decomposed, and the mercury taken up should be constantly replenished. Rise of temperature must be prevented by means of cooling tubes. The author uses a current density of 2·5 ampères per sq. cm. of mercury electrode. The liquid amalgam so obtained is very quickly oxidised by the air, but it can be concentrated by distilling off the mercury in a sealed retort which has previously been exhausted. From 1,000 grms. of liquid amalgam, 12 grms. of solid were obtained, having the composition $Ca_3Hg_4$. The calcium is estimated by boiling under a reflux condenser with standard sulphuric acid.

Calcium amalgam is a whitish grey, porous solid, readily oxidised by air, and decomposing water wirh extreme rapidity. It can be preserved in sealed vessels. It loses mercury on heating in a current of hydrogen, but still retains some at a red heat; no calcium hydride is formed under these conditions. On heating in a current of nitrogen, calcium nitride $Ca_3N_2$, is produced, which can be analysed by treatment with dilute sulphuric acid and determination of the sulphates of calcium and ammonia which are formed. Calcium nitride has a grey colour, and is rapidly decomposed by moist air with evolution of ammonia.—J. F. B.

*Permolybdates.* Melikoff and Pissarjewski. Ber. **31**, [14], 2448—2451.

Muthmann and Nagel having failed to prepare the acid permolybdate of potassium peroxide—



$$\left.\begin{matrix} K_2O_2 \\ H_2O_2 \end{matrix}\right\rangle MoO_4$$

by the authors' method, have thrown doubt on the composition of the authors' preparation, and have suggested that it contained admixed hydrogen peroxide. The authors have carefully repeated their work (adding to a solution of potassium permolybdate the calculated amounts of potassium hydroxide and hydrogen peroxide, precipitating by alcohol, and carefully washing the precipitate with alcohol and ether, the whole of the operations being conducted with cooled liquids), and find that they again obtain the same bright red non-hygroscopic salt, which dissolves in water with evolution of oxygen, slowly changes colour in the dry state to yellow, through loss of oxygen, and explodes by friction, or in large quantities on simply rising in temperature to the ordinary temperature of the air. They find that the same salt is produced though the proportion of potassium hydroxide used in preparing it be lessened, and that of hydrogen peroxide increased; and that when a solution of the salt in ice-cold water is treated with excess of hydrogen peroxide, alcohol again reprecipitates the salt unchanged. The authors suggest that Muthmann and Nagel used too much alcohol, and precipitated some potassium peroxide with their salt. They consider that these salts are to be looked on as permolybdates of the metallic peroxides, rather than as salts of the metals.—J. T. D.

*Calcium Nitride, Preparation and Properties of.* H. Moissan. Comptes Rend. **127**, [15], 497—501.

Calcium, heated to bright redness in a stream of nitrogen gas, combines with it rapidly, and yields a crystalline chestnut-brown nitride, of density 2·63, and of composition corresponding with the formula $Ca_3N_2$. Heated in hydrogen it forms calcium hydride and ammonia. Heated in chlorine, bromine, or iodine vapour, it burns vividly, forming the halide. It burns also when heated in air or oxygen, in sulphur, or in vapour of phosphorus, forming lime, calcium sulphide, or phosphide. Boron and silicon have no action on the nitride, nor has carbon at a red heat; in the electric furnace, however, carbon converts it completely into carbide. Sodium, potassium, magnesium, are without action at a red heat. In nitrogen dioxide at a red heat the nitride burns, forming lime and nitrogen. Dilute mineral acids attack it, forming calcium salts and ammonia, but the presence of water is essential—without it no action occurs. Alcohol, heated in sealed tubes with the nitride, gives calcium ethylate and ammonia. Water attacks the nitride energetically in the cold, forming slaked lime and ammonia; we have here possibly a mode of manufacturing ammonia commercially from atmospheric nitrogen.—J. T. D.

### *Acetylene, Action of, on Fuming Sulphuric Acid.*

G. Schroeter. Ber. **31**, [13], 2189, 2190.

The production of methionic acid during this reaction can be reduced to a minimum, the main product then being acetaldehydedisulphonic acid or disulphoacetaldehyde and sulphates of the same, which are decomposed into the first-named by the action of hydrochloric acid.

On boiling with alkali, the aldehydedisulphonates are split up into methionic acid and formic acid, thus enabling the latter acid to be prepared direct from acetylene.

The author cannot confirm Barthelot's observation, according to which the reaction in question resulted in the production of acetylenesulphuric acid, which in turn yielded phenol when fused with alkali.—C. S.

### *Acetylene, Action of upon Mercuric Nitrate.*

P. Köthner. Ber. 1898, **31**, [14], 2475.

Referring to a recent publication by K. A. Hofmann (this Journal, 1898, 1078) on this subject, the author points out that he studied the action of acetylene on solutions of mercuric nitrate in 1895, and published the results of his investigation in pamphlet form (Halle, 1896), and subsequently in the Zeits. anorg. Chem. 1898, **18**, 48. The compound which Hofmann claims to have first prepared, by passing acetylene into a solution of mercuric nitrate, acidified with nitric acid, was there described, and its constitution discussed, and it was shown that the readiness with which it was decomposed into acetaldehyde furnished a means of preparing that aldehyde synthetically from acetylene. From the results of a series of analyses, the author found the white crystalline substance to have a composition corresponding to the formula, $C_2Hg_3NO_4H_2$, and came to the conclusion that it was a mercuro-carbide nitrate ($HgC \vdots CHg + HgNO_3 + H_2O$). As Hofmann found his preparation to have a different constitution, the author considers it not impossible that the precipitate may vary in composition according to the conditions under which it is obtained.—C. A. M.

### *Glyceraldehyde, Synthesis of.*

A. Wohl. Ber. 1898, **31**, [14], 2394—2395.

In a former communication (Ber. **31**, [11], 1796), the author described a method of preparing acroleïn acetate and converting it into the acetal of glyceraldehyde, which on treatment with decinormal sulphuric acid yields almost quantitatively alcohol, and a solution which, when evaporated *in vacuo* after the removal of the sulphuric acid, leaves a colourless syrup of glyceraldehyde. On dissolving this syrup in alcohol and adding three volumes of ether, small quantities of impurities separate out, and the clear solution, on evaporation again leaves the syrupy glyceraldehyde. This does not crystallise when left *in vacuo* over sulphuric acid, but shows signs of crystallisation when kept in an ice-chamber with constant stirring. By diluting with a few drops of water the syrup which had been concentrated in the vacuum, and then subjecting it to this treatment, the author readily obtained a crop of crystals. These were drained on a porous tile, washed with water, alcohol, and ether, and dried *in vacuo* over phosphoric anhydride at about 90° C. They contained carbon, 39·66 per cent., and hydrogen, 6·67 per cent., corresponding to the formula $C_3H_6O_3$. As thus prepared, glyceraldehyde is a white powder with a sweet taste, and resembling sugar in appearance. It is not hygroscopic, dissolves slowly in water, and is hardly soluble in alcohol or ether. Its melting point, which is not sharp, is about 132° C. On further heating it decomposes with an odour of burnt sugar. Its solution reduces Fehling's solution at the ordinary temperature. Its molecular weight, determined by the freezing point of its aqueous solution, was found to be 164 after the solution had been left for an hour, 102 after it had stood for a day, and the same after standing for three days, the calculated molecular weight being 90. From this the author concludes that it is probable that the substance in a crystalline condition is bi-molecular, but that in aqueous solution it is gradually converted into the simple glyceraldehyde. Whether the former is due to polymerisation or depends on the racemism of the substance is being further investigated, by attempting to esterify the glycerin acetal by means of an optically active acid chloride or anhydride, to separate the ester by crystallisation, and thus to isolate the dextro- or levo-rotatory glyceraldehyde.

—C. A. M.

### *Diphenylamine, Combination of, with Potassium.*

C. Häussermann. J. prakt. Chem. 1898, **58**, [19 and 20], 367—368.

The potassium compound of diphenylamine obtained by Merz and Weith (Ber. **6**, 1515) by the addition of potassium to fused diphenylamine was mixed with products of decomposition which gave it a dark colour. The author finds that the formation of the latter can be prevented by effecting the reaction at a comparatively low temperature (on a water bath) with simultaneous exclusion of the air. The resulting compound forms (in its freshly prepared condition) a yellow sandy powder, which rapidly absorbs oxygen from the air thereby assuming a black colour. It is insoluble in benzene, toluene, and similar solvents, nor does it dissolve in diphenylamine until a temperature of 200° has been reached. By passing a current of dry carbonic anhydride into the compound, which should always be immersed in ether, the potassium salt of diphenyl carbamic acid $(C_6H_5)_2NCOOK$ is obtained. It forms a white stable powder which is almost completely soluble in cold water.—D. B.

### *Acetylisation with the Assistance of Sulphuric Acid.*

H. Skraup. Monatsh. für Chem. 1898, **19**, [7—8], 458—460.

Thiele (Ber. 1898, 1249) has remarked how rapidly acetylisation by means of acetic anhydride proceeds after the addition of concentrated sulphuric acid. The priority of this observation belongs to Franchimont, who (Comptes Rend. **89**, 711) converted cellulose into a triglucose with eleven acetyl groups, by treatment with acetic anhydride and sulphuric acid alone, without the use of sodium acetate, as stated by Thiele. The process of acetylisation with strong sulphuric acid has been for many years in use in the author's laboratory, it proceeds more quickly and easily than other methods, and sometimes succeeds when others fail. For example, mucic acid readily yields the crystalline tetracetyl compound, whilst with acetyl chloride or acetic anhydride and fused sodium acetate, only amorphous products result. The reaction frequently proceeds with almost explosive violence, and it is often advisable to add the sulphuric acid mixed with acetic anhydride. Extremely small quantities of the acid are required; diethylmucic ester was mixed with acetic anhydride containing 0·01 per cent. of sulphuric acid, the temperature at once began to rise, and was 95° C. after 8 minutes. After cooling, filtering, and recrystallising from glacial acetic acid, the product was pure tetracetylester, with a yield of 79 per cent. of the theoretical.

—A. C. W.

### *Carbohydrates, Precipitation of, by Normal Salts.*

R. A. Young. J. of Physiology, **22**, 401; Zeits. f. d. ges. Brau. **21**, [39], 553—555.

The investigations detailed in the paper, extended to the crystalline carbohydrates, starch, dextrin, glycogen, and inulin, the test solutions being generally between 1 and 2 per cent. strength, and the precipitants employed including ammonium sulphate, magnesium chloride, sodium sulphate, sodium chloride, potassium sulphate, potassium iodide, and di-sodium phosphate.

The conclusions drawn by the author are that the "salting out" method of precipitation is as readily adaptable to colloid carbohydrates as to albuminoids, and that the separation of the individual carbohydrates is as easy to effect by this method as by alcohol or freezing.

The sulphates of magnesium and ammonium are the most useful precipitants. Sodium chloride acts best at 33° C., and is advisable in many cases. While the crystalline carbohydrates are not precipitable by neutral salts, starch and soluble starch are readily so, and hence can easily be separated from their hydrolytic products.—C. S.

*The Cinchona Alkaloids, Some Derivatives of.* W. Koenigs and M. Hoppner. Ber. 1898, **31**, [14], 2355—2364.

Methyl iodide unites with de-oxycinchonidine, $C_9NH_6.C_{10}H_{16}N$, in alcoholic solution at the ordinary temperature to form a compound, $C_{19}H_{22}N_2.CH_3I$, melting at 167°—168° C. This substance is transformed by caustic soda into methylde-oxycinchonidine, $C_{20}H_{24}N_2$, which melts at 64°—65° C., shows a slight dextro-rotatory effect in alcoholic solution and a blue fluorescence in a sulphuric acid solution. On heating with aqueous phosphoric acid, lepidine is formed.

The de-oxy bases obtained from quinine, conquinine, cinchonine, and cinchonidine are nearly 10 times as poisonous, in the case of frogs, mice, and guinea pigs, as the original alkaloids.

Cinchonine chloride hydriodide, on keeping, or on boiling with phosphoric acid, is decomposed with the evolution of formaldehyde. Skraup has observed a similar decomposition of quinine hydriodide. The hydriodide of cinchonidine chloride behaves in the same manner. When cinchonine hydriodide is boiled with phosphoric acid, the principal product is a base, $C_{19}H_{22}N_2O$, which exhibits great similarity with the allocinchonine of Lippmann and Fleissner, and with the apo-isocinchonine of Hesse. Cinchene, $C_{19}H_{20}N_2$, on warming with concentrated sulphuric acid, is mainly converted into sulphonic acids, but in part into sulphocinchene, $C_{10}H_{20}N_2SO_3$, insoluble in alkalis.—A. C. W.

*Cyanide Poisoning.* J. Chem. Metall. Soc. of S. Africa, 1898, **1**, 133.

It had previously been stated that subcutaneous injections of 2½—3 per cent. solutions of hydrogen peroxide, accompanied by the washing out of the stomach with a 2 per cent. solution of the same substance, acted as a powerful antidote to cyanide poisoning. Kobert claims priority, having applied the treatment successfully to dogs and cats which had been poisoned by cyanide. The hydrogen peroxide appears to form with the cyanide, oxamide, which, in the quantity likely to be produced, would be innocuous.

—W. G. M.

## *EDUCATIONAL.*

### Proposed School of Brewing.

*Pharm. J., Dec. 10th*, 1898, 624.

Mason University College, Birmingham, may probably before long have a school of malting and brewing established in connection with it. In an appeal for funds for that purpose, it is pointed out that, whilst England has a greater interest than any other country in brewing and malting, it is the only country interested in those trades in which there is no public systematic education given in the manufacture of malt and beer. In Germany (as the *Times* points out) there is the great school at Weihenstephan, near Munich, and, in addition, there are others in Berlin, Worms, Augsburg, and Munich. Austria has schools at Vienna and Prague; Belgium at Liege and Louvain; and at Copenhagen the Brewing School will for ever be connected with the classical researches of Hansen on the yeasts. Brewing is one of the businesses which manufacturers have allowed to be partly taken away from this country by foreign competitors, but it is now proposed that the training to be given at Birmingham shall be of the fullest nature both for maltsters and brewers, and that neither in the character nor extent of the instruction given shall the Birmingham school be inferior to any of those on the Continent. It is intended that the instruction shall be both scientific and technical. Examinations will be held, and diplomas awarded to successful students, and a proper standard by which a maltster's and brewer's training may be gauged will thus be established. It is estimated that 50,000*l*. will be necessary to carry out this project adequately, and of that 21,422*l*. has already been promised.

## New Books.

Les Terres rares: Minéralogie, Propriétés, Analyse. Par P. Truchot, Ingénieur Chimiste. Georges Carré et C. Naud, rue Racine, 3, Paris. 1898. Price 5 frs. H. Grevel and Co., 33, King Street, Covent Garden, W.C.

8vo volume, containing 315 pages of subject-matter, six illustrations, and table of contents at the end of the book. There is comprised in this work the details of the physical and chemical knowledge we possess of the metals of the rare earths. Regarding origin of these earths, the applications to illumination in the construction of mantles, and the great number of analytical schemes that have been devised and proposed, the author has thought well to divide his book into three parts for their treatment. I. The Mineralogical Part. Here is found a table of the minerals of the rare earths, with a detailed study of the principal of these, and in particular the Monazites, which are exclusively employed in the manufacture of incandescent mantles. All details are given as to the geographical position of these minerals. II. General Portion. Here is found a description of each of the rare metals, and of its salts with inorganic and organic acids. III. Analytical Portion. Comprising all that is actually known as to the different methods of fractionation and separation, the analytical characteristics of the different rare metals, and, finally, the different processes of analysis of commercial products, Nitrate of thorium, Thorium precipitate, Monazite sand, Incandescent mantles, &c.

It is stated in the preface that this work "will be useful to chemists and all those interested in the recent discoveries and remarkable researches of MM. Delafontaine, Étard, Lebeau, Langfeld, Moissan, Urbain, &c."

Chemisch-technisches Repertorium. Uebersichtlicher Bericht über die neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und industriellen Chemie, mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von Dr Emil Jacobsen. 37. Jahrgang. 1898. Erstes Halbjahr. Zweite Hälfte. Mit in den Text gedruckten Illustrationen. R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder, Schönebergerstr. 26, Berlin, S.W. H. Grevel and Co., 33a, King Street, Covent Garden, London, W.C.

This number for the first half year of 1898 and the second half thereof, treats of the following subjects of Applied Chemistry:—I. Foodstuffs. II. Paper. III. Photography. IV. Residuals; Manures; Disinfection and Sanitation. V. Soaps. VI. Explosives and Matches. VII. Preparation and Purification of Chemicals. VIII. Fine Chemicals, &c. IX. Chemical Analysis. X. Apparatus, Machinery, Electrotechnology, and Thermotechnology. XI. Appendix, &c. XII. New Books.

## Trade Report.

### *TARIFF CHANGES AND CUSTOMS REGULATIONS.*

### Export of Tinctures.

*Chem. and Druggist, Dec.* 3, 1898, 903.

Hitherto no drawback has been payable on medicinal preparations containing chloroform or ether. By an order printed below it will be seen that the Excise have made a considerable concession in regard to the most important of these preparations. Spirits of ether and chloroform can now be prepared in presence of an Excise officer, who will certify as to the quantity of spirit of wine used, on which the usual drawback will be paid on exportation. In addition, etherial tincture of lobelia, compound spirit of ether, and compound tincture of chloroform are added to the list of tinctures, &c., in which the spirit will be estimated at the Government laboratory, and drawback allowed on the quantity found, *plus* an allowance of 4 per cent. for waste. The general order (No. 17, 1898) contains the following paragraphs :—

Drawback may in future be allowed on the spirits used in compounding spirits of chloroform and spirits of ether exported or shipped for use as ships' stores, on condition that the spirits are first taken account of by the officer and the chloroform or ether added in his presence.

The quantity of spirits of wine to which the chloroform or ether is to be added must be specified, and the purpose for which it is used stated in the notice to pack, and the officer should take account of it as in the case of spirits of wine exported. No special allowance for waste is to be made in either case.

Drawback is also to be allowed on the spirit used in the manufacture of etherial tincture of lobelia, compound spirit of ether, and compound tincture of chloroform, as in the case of other medicinal spirits, on being exported or shipped for use as ships' stores. These preparations should be scheduled as entitled to the special allowance of 4 per cent. for waste.

### CUSTOMS TARIFF OF TASMANIA.

The following statement shows the specific and *ad valorem* rates of Customs duty now levied on articles imported into the colony of Tasmania, together with a list of articles which are admitted free :—

| Articles. | | Rate of Duty. | |
|---|---|---|---|
| (1) *Specific Duties, &c.* | | s. | d. |
| Acid, acetic, glacial acetic, and pyroligneous, imported in any quantity, of not less than one ton at any one time, by manufacturers, to be used in their own factories, for each gallon containing not more than 33 per cent. of absolute acid. | Per gallon | 1 | 4 |
| And for each additional one per cent. of acid. | ,, | 0 | 0¼ |
| Acid, acetic, glacial acetic, and pyroligneous, for each gallon of any strength not exceeding six per cent. of absolute acid. | ,, | 1 | 0 |
| And for each additional one per cent. of acid. | ,, | 0 | 0½ |
| Acid, sulphuric, muriatic, and nitric, not elsewhere exempt. | Per cwt. | 2 | 6 |
| Acid, tartaric and citric................ | Per lb. | 0 | 4 |
| Blue.................................... | ,, | 0 | 2 |
| Candles ................................ | ,, | 0 | 2 |
| Carbonate of soda...................... | ,, | 0 | 1 |
| Cement, mineral, including plaster of Paris. | Per cwt. | 0 | 9 |
| Glucose................................. | ,, | 6 | 0 |
| Glue.................................... | Per lb. | 0 | 1 |
| Gunpowder, blasting.................... | ,, | 0 | 1 |
| Gunpowder, sporting .................... | ,, | 0 | 6 |
| Gunpowder, F, FF, FFF ................ | ,, | 0 | 1 |
| Oil, crude, shale, or petroleum, imported solely for fuel purposes, or for the manufacture of gas, also Russoline. | Per gallon | 0 | 3 |
| Oil, kerosene and gasoline.............. | ,, | 0 | 6 |
| Oil, lubricating, in bulk, of which the chief component is mineral. | ,, | 0 | 6 |
| Oil, of all kinds, in bulk, not elsewhere specified, not being perfumed or medicinal oil. | ,, | 1 | 3 |
| Opium, or any extractive preparation thereof. | Per lb. | 20 | 0 |
| Paints (except artists' colours)......... | ,, | 0 | 0½ |
| Salt, in bulk ............................ | Per cwt. | 1 | 6 |
| Soap, fancy or perfumed ................ | ,, | 0 | 3 |
| Soap, common or laundry, and other saponaceous compounds, not perfumed or fancy, for household purposes. | Per lb. | 0 | 1 |
| Soda crystals .......................... | ,, | 0 | 0½ |
| Spirits, methylated, containing not less than 10 per cent. of commercial wood spirit, and permanently unfitted for drinking purposes, calculated and charged for duty on quantity not being less than one fifth part of a gallon. | Per gallon | 3 | 0 |
| Spirits, perfumed, perfumed waters, bay rum, and toilet vinegar. | ,, | 24 | 0 |
| Spirituous compounds, essences, fluid extracts, sarsaparilla, tinctures, medicines, infusions, and toilet preparations, not otherwise enumerated, containing :— | | | |
| Not more than 25 per cent. of proof spirit. | ,, | 3 | 9 |
| *Specific Duties, &c.*—continued. | | s. | d. |
| Spirituous compounds, &c.—*continued.* | | | |
| Not more than 50 per cent. of proof spirit. | Per gallon | 7 | 6 |
| Not more than 75 per cent. of proof spirit. | ,, | 11 | 3 |
| If containing more than 75 per cent. of proof spirit. | ,, | 15 | 0 |
| If containing spirit over proof, to be charged. | Per proof gallon | 15 | 0 |
| Provided that when calculated at 20 per cent. *ad valorem*, the amount exceeds the sum of duty payable at the rate of 15*s.* per proof gallon, duty shall thereupon become chargeable at the rate of 20 per cent. *ad valorem*. | | | |
| Starch .................................. | Per lb. | 0 | 1 |
| Sugar, crushed, ground, cube, loaf, tablet, icing, and castor. | ,, | 0 | 1 |
| Sugar, all other kinds .................. | Per cwt. | 6 | 0 |
| Turpentine .............................. | Per gallon | 1 | 0 |
| Varnishes (not elsewhere specified) .... | ,, | 1 | 6 |
| Vinegar, other than aromatic and raspberry, for each gallon of any strength, not exceeding 6 per cent. of absolute acid. | ,, | 1 | 0 |
| And for each additional, 1 per cent. of acid. | ,, | 0 | 0½ |
| All goods subject to duty at per 100 lb., or per cwt., or per ton, to pay duty on net weight, and on fractional parts of a quarter of a 100 lb., as if 25 lb., or a quarter of a cwt., as if 28 lb., and so in proportion. | | | |
| Unless otherwise provided, when duty is taken at per gallon, six reputed quart bottles, twelve reputed pint bottles, and twenty-four reputed half-pint bottles shall be held to contain one imperial gallon, unless found to contain liquid in actual excess of one imperial gallon. | | | |
| (2.) *Ad valorem Duties.* | | | |
| Paper, uncut, being "news," in original wrappers as it leaves the mill, under declaration that it will be used exclusively in the production of registered newspapers in Tasmania. | *ad valorem* | 5 per cent. | |
| Paper, uncut, not elsewhere specified, in sizes not being less than post, viz., 18½ ins. by 14½ ins., without printing or ruling thereon, in original wrappers as it leaves the mill. | ,, | 7½ | ,, |
| Detonators, blasting gunpowder, in cartridges and fuze. | ,, | 10 | ,, |
| Grease, axle, and other solid lubricants, not elsewhere specified. | ,, | 10 | ,, |
| Nitro-compounds, and other explosives, not elsewhere specified. | ,, | 10 | ,, |

*Articles admitted free of Duty.*

Acid, carbolic and boracic; acid, sulphuric, rendered unfit for other purposes than the manufacture of artificial manures; acid, sulphuric, for mining purposes only, or for the manufacture of aërated waters; alum, in bulk, unground; annatto, liquid or solid, and rennet; antimony ingots; antitoxin; arsenic, crude; articles in a crude state used for tanning, not elsewhere specified; asbestos, unmanufactured:

Bones, crude; borax in bulk; brewers' isinglass :

Carbon, bisulphide; clay, fire, lumps, and pipe, unmanufactured; coke; copper sulphate; copperas; cyanide of potassium.

Dextrine; dye woods and dye stuffs for manufacturing purposes only.

Ink, printing, ivory black, lamp black and vegetable black.

Kreosote, crude.

Leather, unmanufactured; lime, carbolate and chloride; logwood.

Manufactures from products of Tasmanian dysodile; molasses, raw, rendered unfit for human consumption.

Nickel in bars.

Oil, refuse shale, imported to be used in brick-making; oil cake and cocoa-nut cake and similar compounds im ported as food for cattle; oil, cocoa-nut and palm, unrefined; oil, cod, sod, whale, seal, and penguin oil, for tanning purposes; oil, direct from whale fisheries; oil, to be exclusively used in the preparation of leather, viz., curriers', sole leather, rough leather, finishing and kid oils; ores of metal of all kinds.

Paper for fruit-wrapping, not exceeding 10 ins. by 10 ins.; paper, vegetable parchment and imitation vegetable parchment, to be used exclusively for packing and wrapping butter; paraffin wax and mineral wax; Paris green, dry; phosphor bronze and bronze not otherwise manufactured; phosphorus; pitch; plumbago; potash and pearlash, crude and caustic potash, in bulk; pottery materials, viz., litharge, China clay, Cornish stone, felspar, manganese, oxide of cobalt; preservitas and similar preservatives, to be used in the preservation of fruit and dairy produce, in a fresh condition, not elsewhere specified, pyralicide.

Quicksilver.

Salt, rendered unfit for human consumption, and rock-salt; sheepdip; shellac, sandarac, and mastic; soda, ash, caustic, and silicate in bulk; solder and soldering fluid; sulphur; sumac.

Tannin and tannin extracts; tar; toxa and all other poisons for the destruction of rabbits; turmeric.

Ultramarine.

Valonia.

Wax, bottling.

Zinc, salts of and zinc shavings and discs; zinc in blocks and sheets, not perforated or otherwise manufactured.

### CLASSIFICATION OF COPPER SALTS IN RUSSIA.

*Bd. of Trade J., Dec.* 1898, 683.

The Russian Customs Department has notified, under circular of the 20th October (1st November), that only such salts of copper or of arsenic (including Parisian or Schweinfurth green), in powder or pressed cakes, shall be admitted duty free under observation to section 133 of the Customs Tariff, as may contain an admixture of not less than 5 per cent. of powdered charcoal, the object of the order being to prevent the salts in question being used as colouring matter.

### DRAWBACK ON EXPORTS OF SUGAR AND SYRUPS IN THE UNITED STATES.

*Bd. of Trade J., Dec.* 1898, 692.

(This circular is unsuitable for abstraction.)

## *EXTRACTS FROM DIPLOMATIC AND CONSULAR REPORTS.*

### BRITISH TRADE CONDITIONS, AND PROSPECTS OF, IN CERTAIN SOUTH AMERICAN COUNTRIES.

*T. Worthington. Government Report. Part* 1 *and* 2, *Chile,* 1—39. *Part* 3, *Argentine Republic,* 1—46.

*Part* 1 *and* 2.—The objects of this inquiry were to determine the causes, if any, which have occasioned a diminished demand for articles of British produce or manufacture, either absolutely or relatively to the demand for similar articles produced in other countries, also to ascertain in what directions, and under what conditions, a demand for British goods might be created or increased.

The following table shows the value of the merchandise cleared from bond from the principal manufacturing countries, for consumption at all Chilian ports:—

| Years. | Great Britain. | Germany. | United States. | France. |
|---|---|---|---|---|
| | Million Dols. | Million Dols. | Million Dols. | Million Dols. |
| 1887 | 20·1 | 11·6 | 3·2 | 5·5 |
| 1888 | 26·3 | 14·0 | 3·1 | 6·1 |
| 1889 | 27·9 | 14·8 | 3·8 | 6·5 |
| 1890 | 29·5 | 15·7 | 5·2 | 6·8 |
| 1891 | 27·8 | 12·1 | 4·1 | 4·9 |
| 1892 | 34·1 | 21·0 | 4·6 | 6·7 |
| 1893 | 30·8 | 17·0 | 4·5 | 4·1 |
| 1894 | 25·5 | 12·3 | 3·7 | 2·3 |
| 1895 | 32·0 | 17·3 | 4·5 | 1·6 |
| 1896 | 30·2 | 20·1 | 6·8 | 2·8 |

The several articles produced by foreign manufacturers, and competing with British made goods are dealt with and among them are many relating to the chemical trades.

*Alkali.*—The import of caustic soda (for native soap boilers) from England has increased largely, whilst that from Germany has fallen off. There is no native alkali made.

*Powder.*—Both mining and sporting is made in the country, the import trade being quite small and apparently declining.

*Beer and Ale.*—This trade is practically in the hands of native breweries. The imports are very small, those from Germany have been reduced by one-tenth of what they were five years ago. The duty has recently been raised to 3 dols. per dozen bottles.

*Candles.*—Common dips are made in the country, but there is a large importation which has increased 50 per cent., of candles, mostly from Belgium. There is a small and increasing trade from England, but the English article is too good and does not compete in price.

*Glass and Glassware.*—The trade in silvered glass for mirrors is a small one and entirely in the hands of Germany. The German and Belgian imports of glassware have increased, whilst the English have fallen off, the reason being that the English production is not cheap enough. Window-pane glass comes chiefly from Antwerp. In this article again the German and Belgian import has increased about as much as the English has diminished, the reason being assigned to cheaper freightage.

*Cement.*—The English import has increased, whilst the German (including Belgian) has declined. English cement sometimes comes in German vessels from Hamburg, the agents for which receive it in London, take it to Hamburg by steamer, and bring it out at a through freight from London to Valparaiso for 12*s.* per ton.

*Leather.*—The import of tanned hides from England has increased, whilst that from the Continent has fallen off. There are extensive tanneries in the country, which tan all the native hides. Native hides are exported largely, almost exclusively to Germany, a large portion of the hides being of a coarse quality, and destined chiefly for the Russian market.

*Oils.*—Linseed oil comes mostly from England. The German and United States imports of vegetable oil have increased as the English have fallen off.

*Painters' Colours and Materials.*—These come chiefly from England, except turpentine, which is received from the United States.

*Soap.*—This is made to a considerable extent in the country. The importation had increased, and in greater proportion from England than from the Continent, or the United States especially in scented kinds; but the heavy increase of duty, about 30 per cent. on common, and much more on scented soap, will tend to minimise imports henceforth.

*Sugar.*—There is a large importation of refined sugar from Germany, and of raw sugar from Peru and Australia. The native manufacture will, no doubt, flourish under the new tariff more than it has done; in fact, the increased duty on imported sugar is so heavy, as to lead many to expect imports to fall to a very small figure.

Proceeding to the causes of the depression in British trade in Chile, the commissioner voices the general complaint often experienced in inducing English manufacturers to (*a*) To take up a new pattern or design on a small order. (*b*) To make qualities very much lower than they have been accustomed to. (*c*) To make a low quality with a finish and get up, such as they might think only belonged to a higher class. (*d*) To send samples prepared in a sufficiently attractive form. (*e*) Generally to attend to minutes which might appear to them unimportant, but which may be very important as affecting the sale of goods here.

In some additional notes concerning Chile, the amount of imported caustic soda and sulphate of copper is given.

The figures are—

| —— | Caustic Soda. | Sulphate of Copper. |
|---|---|---|
| | Kilos. | Kilos. |
| Great Britain | 2,137,805 | 69,905 |
| Germany | 388,126 | 41,442 |
| France | 44,060 | Nil |

Soap, paper, glass, and mineral-water making should all increase under the new tariff. Wool is shipped unwashed. These industries are all in the central portion of Chile, although centres of special industries can hardly be said to exist.

In the Argentine Republic the native industries of beer, paper, matches, soap, shoes, common glassware, &c. appear to be developing. The wine product of the Mendoza district is estimated this year at 500,000 barrels of 200 litres each. There is much to be done with this business, the demand being far ahead of the supply. The following figures show ample margin for expenses and profits, *viz.* :—

Value of a hectare of bearing vines (French kind), 4,000 dols.

Yield of grapes per hectare, 200 quintals.

Value per quintal, 3½ dols.

About 7 quintals will yield 200 litres of wine.

Value per litre about 25 cents, or 50 dols. a barrel.

*Part 3.—The Argentine Republic.*—The Commissioner has made the following comparative table of the value (in millions of gold dollars) of goods received from the six principal countries for each of the last 10 years.

| Country. | 1888. | 1889. | 1890. | 1891. | 1892. | 1893. | 1894. | 1895. | 1896. | 1897. |
|---|---|---|---|---|---|---|---|---|---|---|
| United Kingdom | 44·0 | 56·8 | 57·8 | 28·3 | 35·8 | 32·5 | 35·6 | 39·5 | 44·7 | 36·4 |
| Germany | 13·3 | 15·5 | 12·3 | 6·2 | 10·7 | 11·0 | 10·7 | 11·2 | 13·9 | 11·1 |
| United States | 9·9 | 16·8 | 9·3 | 3·4 | 7·4 | 9·6 | 10·1 | 6·7 | 11·2 | 10·1 |
| France | 23·0 | 30·2 | 19·9 | 7·9 | 10·4 | 12·1 | 10·2 | 9·1 | 12·0 | 11·0 |
| Belgium | 11·1 | 14·0 | 11·0 | 6·4 | 6·6 | 9·6 | 9·0 | 7·4 | 8·5 | 8·0 |
| Italy | 7·8 | 10·2 | 8·7 | 4·2 | 8·4 | 9·3 | 8·9 | 10·4 | 11·4 | 10·9 |
| Other countries | 19·3 | 22·1 | 23·2 | 10·8 | 12·2 | 12·1 | 8·3 | 10·8 | 10·5 | 10·7 |
| Totals | 128·4 | 165·6 | 142·2 | 67·2 | 91·5 | 96·2 | 92·8 | 95·1 | 112·2 | 98·2 |

The following chemical trade items are dealt with :—

*Alkali.*—Soda ash and caustic soda is supplied by England. It is stated that caustic soda prepared by electrolysis is now offered more cheaply from the United States. Material of 70 to 72 degrees strength is required since it is only used by the more advanced soap makers. Bicarbonate of soda (and sesqui-carbonate) for making soda water, soda crystals for domestic uses, and ammoniacal carbonate of soda (solvae) used by glass makers, come almost entirely from England. The United States supplies as much copper sulphate as England, chloride of lime comes from several countries, but chiefly from England, whilst our chief competitor in chlorate of potash appears to be Belgium. Silicate of soda for soap makers is little used ; and the trade in ammonium salt is insignificant. Although mineral water manufacture is carried on extensively in the country the total imports are increasing.

*Gunpowder* is imported chiefly from England. The conservatism of English trading methods is illustrated by the circumstance that the sale of a well known English make is reported threatened on account of the makers declining to change their form of package.

*Beer and Ale.*—The importation of these articles is comparatively trifling.

*Candles of all Sorts.*—Both stearine and tallow candles are made in the country, the bulk of the small imports coming from the Continent. The local industry is protected.

*Caoutchouc.*—India-rubber tubes, plain and wired, and sheets, come from the Continent as well as England. The prices of English goods are 20 per cent. higher than those of other countries.

*Cement.*—The largest import is from Belgium, whence it appears to come cheapest; the figures of the four principal producing countries are as follows :—

*In Tons of* 1,000 *Kilos.*

| Country. | 1894. | 1895. | 1896. |
|---|---|---|---|
| United Kingdom | 10,419 | 11,218 | 13,073 |
| Germany | 857 | 973 | 1,529 |
| Belgium | 9,968 | 13,579 | 18,643 |
| France | 2,237 | 2,910 | 6,435 |

The materials for making cement are found in the country, and there appears to be a possibility of a successful local industry.

*Chemical Products and Preparations.*—Sulphuric acid, hitherto imported from Belgium and Germany, is now being made in the country. Boracic acid and Prussian blue are small local industries, tartaric acid comes from France and Italy.

*Leather.*—Tanning is a very large industry in the country, and the preparation of leather has reached a high degree of perfection. Nevertheless, a certain amount of waxed calf, kid calf, patent leather, and kid are still imported from France.

*Metals.*—Wrought and unwrought copper comes from England and the Continent. The supply of brass tube is mostly of English manufacture. Lead comes from the Continent and England. French lead is preferred for shot making, and the English for tubes. Unwrought tin is a small English import. Zinc, wrought and unwrought, comes from Belgium.

*Oil and Floor Cloths* come from England.

*Oil, Seed.*—Linseed is an export of the country, and oil is made from it to a considerable extent. There is, however, a large importation, whilst that of cotton, cocoanut, colza, and rape-seed oil is small by comparison. The importation of olive oil is large.

*Painters Colours and Materials.*—White lead comes in larger quantities from England than elsewhere. Powdered colours, acetate of lead, and gums are obtained cheaper from Hamburg than from England. Made paints still come from England to some extent, but ochres are French, and lamp-black German. Red lead comes from Germany and Italy besides England. Varnish of poor quality is made in the country. Putty comes from England and asbestos from Italy.

*Paper, other than Hangings.*—Paper making is an industry of the country. Writing paper and envelopes come chiefly from the Continent, as also a small portion of the paper for newspapers and wrapping paper.

*Salt.*—Fine and rock salt come almost entirely from England, and common salt from Spain.

*Soap.*—This is chiefly made in the country; Great Britain has a larger share of the imports than any one other country. A local candle factory prepares soap at a net profit of 1·20 dols. per 10 kilos.

*Sugar.*—Sugar is refined in the country, and is of a high grade. The production has been in excess of the requirements, and a certain amount is exported. A certain quantity of English sweetmeats are imported.

In his conclusions the Commissioner again insists on the lack of enterprise of British manufacturers (*loc. cit.*), and he concludes that they have lost the trade in greater or less measure by failing promptly to comply with the information as to changes in quality, make or get up, &c., which may have been conveyed to them.

### The Sugar Industry in Italy.

*French Consular Report.*

The French Consul at Genoa reports the formation in that town of a company, with a capital of 4,000,000 lire, for the manufacture of beetroot sugar. The refinery is to be erected in the environs of Bologna, but the management and working will be entrusted to Genoese engineers, where, moreover, the greater portion of the capital has been subscribed.

The number of existing beetroot sugar factories in Italy is four, *viz.*, at Savigliano, Sinigaglia, Legnano, and Rieti, the last being the most prosperous. Another factory, with a capital of 1,000,000 lire, was formed recently at Turin, and is now erecting buildings at Segni.

Italy is strenuously endeavouring to produce for herself the sugar she consumes. In 1897, 75,582 tons were imported, and, during the same year, the home production increased from 23,000 cwt. to 33,000 cwt.—J. L. B.

### The Manufacture of Artificial Manure in America.

*Ch. of Com. J., Nov.* 1898, 236.

The Austro-Hungarian Consul at Chicago writes: Precise statistics of the production of manures in the United States cannot be procured, but that it has greatly increased since 1890 may be inferred from the number of licenses issued in various States for the manufacture of this article. Licenses to manufacturers were issued in 1880 to the number of 364; in 1890, 390, and now their number is 700. The States of South Carolina and Florida, in which there are numerous phosphate works, are particularly interested in the manufacture. In the year 1896 the sales reached a total of 1,891,917 tons, having a value of 37·68 million dollars. The holder of a license is bound to see that each receptacle containing manure is accompanied by a certificate stating the chemical analysis of the manure, the net weight of the receptacle, and the name of the manufacturers; a duplicate of this certificate must at the same time be sent to the State official concerned, with a sample, which is analysed, and the result published.

### Paints used in Siam.

*Ch. of Com. J., Nov.* 1898, 236.

The French Minister at Bangkok says there is a somewhat considerable trade in colours for paints in Siam, where most of the buildings are of wood, but the Customs statistics do not permit of the extent of the imports being stated. Oil paints are the most important, and of these the consumption in recent years has greatly extended, owing to the building operations which have been carried out. The colours most generally employed are zinc white and white lead, the very heavy sales of these two products being explained by the fact that there is not an apartment in Bangkok into the painting of which they do not enter largely. Other colours which find a ready sale are greens, blues, browns, yellows, reds—varnishable red in particular, red lead, Sienna earths, and red and yellow Italian earths. Most of these are sent to Bangkok in the form of paint; but often the colours are mixed locally with linseed oil and spirits of turpentine, this being the most advantageous mode of procedure. In either case, most of the imports are from England. "That country has supplied Siam so long, and the use of English colours has become so habitual, that it is difficult to secure the acceptance of others without complying with the English taste, and the conditions as to price and consignment in use with the English."

## *GENERAL TRADE NOTES.*

### Production of Sisal in Mexico.

*Bd. of Trade J., Dec.* 1898, 718.

The production of sisal grass, or ixtle, in Mexico is increasing each year, according to a French consular report. This product is seldom sold directly from the producer to the consumer, the traffic being monopolised by a few commercial houses, chiefly located in San Luis Potosi and Cerritos, a small station on the central railroad. These establishments send their products to the markets at New York, Hamburg, Havre, and in small quantities to Antwerp, and through their agents they keep close track of the fluctuations of these markets.

The greater part of the fibre now comes for preparation to the establishment of M. Munoz, at Cerritos. This place has consequently become the point of departure for much of the ixtle traffic of the country and the freights for Europe are quoted: Cerritos-Hamburg, Cerritos-Havre, &c.

The harvesting month for this crop is September, and the production for the season of 1896–97 was estimated at 9,635,000 kilos., valued at 179,200*l.* silver. According to the official statistics, the exportation of this product for several years past has been as follows:—

| — | Kilos. | Value. |
|---|---|---|
| 1893–94 | 5,667,424 | 96,170 |
| 1894–95 | 4,342,621 | 72.820 |
| 1895–96 | 7,154,845 | 141.775 |
| 1896–97 | 9,165,467 | 169,370 |

It is seen from this table that during a very short time the exports of this product have increased enormously.

### Pine-Apple Fibre as a Textile.

*Bd. of Trade J., Dec.* 1898, 671.

The Calcutta Journal *Capital* again calls attention to the value of pine apple fibre, which has so far been unfavourably received both in England and India. There seems, however, to be some evidence that this fibre would make a good substitute for flax, recent tests in Singapore having shown that it is one-fourth stronger than flax, besides being impervious to moisture, thus rendering it particularly suitable as rope material. The mere process of bleaching suffices to destroy the adhesion between the bundles of fibres and so renders it fit for spinning. The isolated filaments are described as very fine, of regular diameter from end to end, but of different size and of remarkably flexibility. From the pine apple of the Philippines a famous cloth is manufactured, much esteemed for its fine hair-like fibres, though possibly this may belong to a different species from the pine apple of the Straits Settlements.

### The Cultivation of "Pindars" or Ground Nuts in Jamaica.

*Imp. Inst. J., Dec.* 1898, 341.

The ground nut or "pindar" (*Arachis hypogæa*) is a low-growing leguminous plant, which has not only acquired a high reputation as a cattle food in Europe and America, but has been also used as an article of human diet, and, if carefully cultivated, might contribute largely to the colonial food supply. An account of this interesting product is given in the October issue of the *Journal of the Jamaica Agricultural Society*, by Mr. F. Watts, the chemist of that society.

Although "pindars" are consumed freely in Jamaica, and are imported to a fairly large extent, the cultivation of the seeds in the island itself has not yet reached the proportions that would naturally be expected in a country where the soil, climate, and general conditions are so favourable. At present these nuts are consumed more as a luxury than as a regular article of food, though they form a most wholesome addition to the dietary, and their cultivation might well become an important industry. The oil which they contain is known to be quite suitable for many of the purposes for which olive and cotton-seed oils are at present imported. The residue left after the expression of the oil is of considerable value, and may used as a cheap article of food.

The sample nuts, as purchased, yielded about 67 per cent. of seeds, which, when pressed in a small hydraulic press, furnished—

| | Per Cent. |
|---|---|
| Oil of first quality | 31·5 |
| Oil of second quality | 5·0 |
| Cake or meal | 54·8 |
| Loss in handling, &c. | 8·7 |

An analysis of the "pindar" meal gave the following results :—

| | Per Cent. |
|---|---|
| Water | 9·72 |
| Oil | 5·68 |
| Starches and digestible carobo-hydrates | 36·08 |
| Protein | 40·05 |
| Nitrogenous matter other than hydraprotein | 1·07 |
| Crude fibre, indigestible carbotes | 3·86 |
| Mineral matter | 3·54 |

The mineral matter consisted chiefly of potash and phosphoric acid. From this analysis it is evident that "pindar" meal would be eminently suitable as a fertiliser, for which purpose it is valued at about 4*l.* 10*s.* per ton.

The vine itself, when properly dried, is also of considerable value as fodder for stock. "Pindar" hay is very little inferior to clover hay; moreover, it bears on its roots the nodular swellings characteristic of the *Leguminosæ*, which enable it to assimilate the nitrogen of the air, and so becomes capable of enriching soils which are poor in nitrogen.

There are several varieties of "pindars," and of these the Spanish possesses the advantages that the seeds may be planted more closely together, that its crop is comparatively easy to harvest, and that it matures in four or five months, while other varieties require about nine months.

In the United States it is usual to remove the seeds from the pods before planting; but in the West Indies it is advantageous to sow the seeds in the pods, as they are then protected from the ravages of the ants.

The average yield amounts to about 30 to 50 bushels per acre, the bushel varying from 22 lb. to 28 lb., according to the kind cultivated.

### THE SOAP INDUSTRY OF NEW ZEALAND.

*Bd. of Trade J., Dec.* 1898, 711.

The *New Zealand Trade Review* notices a new industrial enterprise started in the neighbourhood of Wellington. This is a soap factory, and it is stated that the appliances for the work are very complete and up-to-date, and that it is the proprietor's intention to devote himself chiefly to the manufacture of fancy soaps.

The production of candles and soap was among the earliest of the manufacturing industries established in the Colony, as was only natural when the increase in the flocks and herds resulted in a good supply of tallow. The days when New Zealand had to import her supplies of common soap have long since passed away, and the importation in the year 1897 was only 13 cwt.

There was once an export trade in common soap, chiefly to Fiji and the South Sea Islands. To judge from the returns, however, this trade seems to be steadily declining. In the year 1892, the exports were over 6,000 cwt., the value over 4,600*l.* But the figures have diminished year by year till in 1897 they stand as 2,845 cwt., value 2,038*l.*

The importations of fancy soaps are, however, increasing from year to year.

The figures for the last four years are as follows :—

| Years. | Quantity. | Value. | Soap Powder, Extract, &c. Value. |
|---|---|---|---|
| | Lbs. | £ | £ |
| 1894 | 415,642 | 8,980 | .. |
| 1895 | 452,181 | 10,473 | .. |
| 1896 | 591,530 | 14,452 | 3,902 |
| 1897 | 725,476 | 18,404 | 3,876 |

It may be stated that in the year 1896 a new classification was adopted in the statistics, and the imports of "powder, extract, &c." which are given separately in 1896 and 1897 were, before that, included under the general heading. This makes the increase the more striking.

Judging from the above figures, there is ample room for the new enterprise, and with an import duty of 25 per cent., the *Review* says that the proprietor ought to be able to make a success of it.

### PRODUCTION OF MANGANESE ORE.

*Bd. of Trade J., Dec.* 1898, 710.

According to an official report by the United States Government, the output of manganese from the United States for 1897 was 11,109 long tons, valued at 19,890*l.*, as compared with 10,088 tons, valued at 18,900*l.*, in 1896. The output of 1896 came from seven states: Arkansas, California, Georgia, North Carolina, Pennsylvania, Virginia, and West Virginia. In 1897 North Carolina and West Virginia reported no manganese production, while Michigan and Tennessee each reported a small production. The country's total output since 1880 has been 280,375 long tons, Virginia having contributed 163,980 tons; Georgia, 50,845 tons; and Arkansas, 46,068 tons.

The world's chief sources of manganese ore, according to the latest available reports which can be considered reliable, were as follows :—

| Year. | Country. | | Production.. |
|---|---|---|---|
| 1890 | Greece | (Metric tons) | 404,837 |
| 1897 | Caucasia | (Long tons) | 231,868 |
| 1897 | Spain (estimated) | " | 90,000 |
| 1896 | Germany | " | 44,350 |
| 1896 | Prussia | " | 42,935 |
| 1896 | France | " | 30,797 |
| 1896 | Belgium | " | 23,265 |
| 1895 | Japan | " | 16,052 |
| 1895 | India | " | 15,816 |
| 1897 | Brazil | " | 12,083 |
| 1895 | Chile | " | 10,748 |
| 1895 | Austria | (Metric tons) | 4,283 |
| 1896 | Italy | (Long tons) | 1,860 |
| 1896 | Portugal | " | 1,470 |
| 1897 | Great Britain (estimated) | " | 599 |
| 1896 | Queensland | " | 300 |
| 1895 | South Australia | " | 48 |

### SPONTANEOUS COMBUSTION OF LEATHER.

*Imp. Inst. J., Dec.* 1898, 341.

Within the last few years the leather trade has suffered very severely from fires, a few of which, through their magnitude, are still remembered. In many of these cases no reasons could be assigned for the outbreak. A short time ago Professor Eitner wrote that, "as doubts exist as to the danger of outbreaks of fire through spontaneous combustion, people take no precautions against its occurrence. The beginning of many fires in tanneries is in the currying shops, and particularly in the stuffing room, on account of the quantity of grease and fat present. At the end of last winter I had, at the Vienna Leather Industries Laboratory, an opportunity of observing how a fire can originate in a stuffing room. Some woollen cloths, which were saturated with cod oil, were placed in close proximity to a steam pipe. Suddenly an intense odour was given off from the cloths, which had turned brown. Owing to the heat generated and to the rapid oxidation of the oil, they burst into flame. In the stuffing room, goods impregnated with cod oil, coming into contact with any hot substance, the oxidation of the oils is forced, and flames are the result. Finished leather impregnated with cod oils can certainly, from the same cause, be ignited. Danger can also arise from the oil-wipers used for cleaning the stuffing tables. Other fats, such as tallow, mineral oil, möellen, resinous oils, which are used for stuffing, are comparatively not dangerous.—*Leather Industries.*

### PETROLEUM IN THE DUTCH EAST INDIES.

*Eng. and Mining J., Nov.* 26, 1898, 634.

United States Consul S. B. Everett writes from Batavia, Java, that for some time rumours have been afloat that the Sumatra oil companies were not as easy in their minds as they had been, and that the wells were running dry more quickly than new ones could be bored. No one seems to know exactly how much truth there is in all this, and many believe that it is merely a movement of insiders on the Amsterdam Stock Exchange to depress the stocks of these companies, with a view of buying in more and thereby tightening their control. If that be their object it has certainly succeeded; for, whereas the shares of the Royal Company (Koninklijke Maatschappij) were at one time as high as 1,010 per cent. premium, they are now very little over 200 per cent. premium, and there is every prospect of a further decline.

Where there is so much smoke, however, there is bound to be some fire, and there is no doubt that many of the wells are running dry at an alarming rate. This in itself would not be so very important, as there is much new territory, practically half Sumatra, which has not been worked, and which gives every indication of being good oil country; but the trouble is that the Dutch have neither capital nor men enough to go ahead, and they will not permit foreigners to help them out.

American oil experts are not permitted by the Dutch laws to work in the new Tamiang concession, although it is freely admitted that there are not enough Dutch experts competent to do the work. The alleged reason is that Tamiang being a so-called "dangerous district," the Dutch are afraid that if any foreigners should get killed by rebels there, their respective governments would make troublesome reclamations. This, however, does not explain the hostility to foreign capital, which is absolutely necessary if they are to do anything with Sumatra, Borneo, and New Guinea.

Last year, on a capital of 8,000,000 guilders (3,200,000 dols.), the directors of the Royal Company paid a dividend of 56 per cent., which was practically all the cash they had in the treasury at the time; and that, too, in spite of the fact that they knew their wells were running dry, and should have kept back every cent they had for the purpose of opening up new wells and more territory. Desparate as the situation will soon become, the Dutch Government will not permit either foreigners or foreign capital to take a hand. The same policy holds good with regard to coal and mining concessions in Borneo and the Celebes.

The situation, therefore, is full of encouragement for the Standard Oil Company. The Russian oil has been nearly driven out of the market, and if the wells now being worked in Sumatra run dry, and Dutch policy does not permit of new ones being opened, American oil will again have a monopoly and raise the price to a paying point. Many shrewd observers here, including even Dutchmen, believe that Americans will get control, in some way, of the better Sumatra oil fields. This may be true, for Sumatra is among the richest countries in the world, and has hardly been touched.

### THE COLD STORAGE OF FRUIT.

*Chem. Trade J., Dec.* 3, 1898, 356.

A report by Mr. W. P. Wright, county superintendent in horticulture, on the experiments in the cold storage of fruit being carried out under the auspices of the Kent County Council, has just been issued. The results of the experiments on strawberries may be epitomised as follows:—(1) The fruit cannot be kept long in a temperature of 36 or upwards; (2) it can be kept for three weeks at least in a temperature of 30 ; (3) it is necessary to surround the fruit with cotton wool, or, in the case of fruit in sieves, to place a pad of that material over the top. If this precaution is not taken, the fruit, though sound, becomes dull, and loses the fresh, inviting appearance which is so important when it is offered for sale. The general results were:—(1) The fruit must be placed in store in advance of dead ripeness, especially in the case of strawberries. Fruits that are fully ripe will keep for some time, but lose surface freshness, becoming seared and unsightly. (2) Cherries pecked by birds soon go off, and other fruits impaired in any way will not keep. To achieve success the fruit must be sound. (3) The fruit should be covered or surrounded with cotton wool. It was observable that that so treated retained its freshness much longer than that left exposed. (4) The chambers must be kept dry, and close-fitting doors are necessary. (5) All decaying or impure matter must be rigidly excluded.

### NICKEL IN TASMANIA.

*Eng. and Mining J., Nov.* 19, 1898, 611.

The discovery of nickel ore in the Rocky River Mine in Tasmania promises to be of some importance, according to the *Australian Mining Standard.* The ore has been found at the 110 and 155 ft. levels of the mine in connection with gold ores, and numerous analyses made have left no doubt of its nickel-bearing character. It is understood that development work is to be continued in order to ascertain the extent of the deposit. Nickel ore has been found in Tasmania before, but has not been worked on a commercial scale, though a few tons of ore were exported in 1894.

### MANURE TRIALS IN CAMBRIDGESHIRE.

*"Standard" Agric. Reports, Dec.* 1, 1898.

Some interesting experiments carried out in connection with the Cambridge and Counties' Agricultural Education Scheme were described by Mr. T. B. Wood, M.A., in an address to the Cambs and Isle of Ely Chamber of Agriculture a few days ago. In a rotation experiment, to last four years, and only half completed, the results, so far, indicate that it pays better to apply a small dressing of manures to the root crop, and another to each of the succeeding crops, than to use a heavy one for the root crop alone. In trials of different fertilisers upon mixed grasses and clovers grown in rotation, the satisfactory increase of nearly 28 cwt. per acre of hay was obtained from the application of 2 cwt. each of nitrate of soda and potash (presumably sulphate of potash), costing 1*l.* 13*s.* But neither potash nor superphosphate alone gave any considerable increase, though both added greatly to the proportion of clover in the hay. The nitrate of soda alone, costing 17*s.*, gave 13 cwt. per acre more hay than the yield of the unmanured plot; but, for the sake of increasing the clovers, greatly to the benefit of the succeeding corn crop, it pays well to add superphosphate and potash. In some experiments on potatoes, it was assumed, on the testimony of growers, that 4 cwt. of superphosphate would pay on all soils, and the problem was to ascertain what additional manuring would be most profitable. As compared with the produce of the unmanured plot, 4 cwt. of superphosphate gave about 1 ton of increase, and when the quantity of this manure was doubled, a further increase of half a ton was obtained at an additional cost of 9*s.*, which paid remarkably well. Sulphate of potash also proved highly remunerative. On a black soil at Benwick, full of organic matter, a mixture containing nitrate, and costing 22*s.* an acre, gave a crop 32 cwt. more than the produce of the unmanured plot. As disease was present, spraying with the Bordeaux mixture also greatly increased the yield of the potato crop.

### NEW ZEALAND GUM INDUSTRY.

*Chem. and Druggist, Dec.* 10, 1898, 937.

Following on the report of the Commission to inquire into the kauri-gum industry (this Journal, 1898, 621), the New Zealand Government have drafted a Bill to come into operation on 1st January next. The Bill contains

32 clauses, and some of its provisions are of a highly protectionist character. For example, all who dig for the gum must take out a license, which license will be given to none unless he can prove 12 months' residence in the colony, and is a British subject by birth or naturalisation. No mention of an export duty is made. There is a certain amount of anxiety apparent on the London and other markets as to the future of the gum, as the output will henceforth be restricted. The colony would probably be the sufferer, as the value of fine kauri is already so high that, should it be forced up, a substitute for the gum may be found, or research for synthetic gums (better than the ester gums) stimulated.

### Water Power for Electricity from Loch Ericht.

*Chem. Trade J., Dec. 3, 1898, 352.*

Among the more important proposals to come before Parliament next session in the form of private Bills, is one from Scotland, promoted by a syndicate, to authorise a scheme for conveying the waters of Loch Ericht, Inverness-shire, to the top of Loch Leven, on the West Coast of Scotland, at a point about seven miles above Ballachulish. The object is to secure at that point sufficient water power to generate about 40,000 electrical horse-power. This it is proposed to accomplish by means of a fall of 1,000 ft. The scheme is estimated to cost between 1,000,000*l.* and 1,500,000*l.*, and when it has been carried out, there is every reason to believe that a number of extensive factories in connection with chemical industries will be established at Loch Leven. Of late years it has been found that, owing to the abundance of water power available in America and Germany for the purpose of generating electricity at a very cheap rate, the British chemical manufacturers have felt the pinch of foreign competition. One great advantage of the Loch Leven site is that vessels drawing 14 ft. of water can get up to the top of the loch at high tide, so that the raw material required for the factories, as well as the latter's finished produce, could be conveyed by water instead of rail, and this, as is well known, means cheaper rates. The proposed scheme will, it is believed, cause certain large chemical manufacturers, who had been contemplating the transference of their works from this country to the United States, to revise their plans. As to the Parliamentary prospects of the Bill, it is considered probable that the riparian owners, especially those on the Tay, will offer some opposition, as the water proposed to be impounded flows into the Tay valley; but the promoters are confident they can overcome this difficulty by raising the level of the ground in one of the contributing valleys, so as to intercept the storm water, by which means the engineers say that they will be able to give a flow of water during the dry season twice as great as that which now enters the Tay, whilst they will also do much to mitigate the damage annually caused to the banks by the flooding in the wet season. It may be added that the particular area affected by the scheme is within the chief rainbelt in Scotland.

## *BOARD OF TRADE RETURNS.*

### Summary of Imports.

| Articles. | Month ending 30th November | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals | 1,650,649 | 1,768,774 |
| Chemicals and dyestuffs | 372,609 | 394,603 |
| Oils | 645,080 | 609,750 |
| Raw materials for non-textile industries. | 4,078,164 | 4,127,950 |
| Total value of all imports | 41,166,146 | 42,069,569 |

### Summary of Exports.

| Articles. | Month ending 30th November | |
|---|---|---|
| | 1897. | 1898. |
| | £ | £ |
| Metals (other than machinery) | 3,036,552 | 2,760,470 |
| Chemicals and medicines | 753,840 | 690,973 |
| Miscellaneous articles | 2,848,314 | 2,937,739 |
| Total value of all exports | 19,773,594 | 19,820,207 |

### Imports of Metals for Month ending 30th November.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| Copper :— | | | | £ | £ |
| Ore | Tons | 6,629 | 8,500 | 87,318 | 86,515 |
| Regulus | „ | 5,853 | 5,118 | 140,467 | 129,876 |
| Unwrought | „ | 4,526 | 5,991 | 222,691 | 327,913 |
| Iron :— | | | | | |
| Pig and puddled bar. | „ | 23,434 | 25,581 | 72,755 | 81,844 |
| Ore | „ | 470,277 | 357,678 | 339,147 | 258,828 |
| Bolt, bar, &c. | „ | 5,956 | 5,953 | 50,081 | 46,647 |
| Steel, unwrought | „ | 1,296 | 1,882 | 11,743 | 16,272 |
| Lead, pig and sheet | „ | 14,167 | 15,831 | 182,572 | 212,314 |
| Pyrites | „ | 51,453 | 52,548 | 82,278 | 91,369 |
| Quicksilver | Lb. | 80,250 | 8,450 | 7,330 | 816 |
| Silver ore | Value £ | .. | .. | 117,995 | 101,409 |
| Tin | Cwt. | 41,339 | 25,827 | 133,302 | 102,972 |
| Zinc | Tons | 5,923 | 5,561 | 100,796 | 112,453 |
| Other articles | Value £ | .. | .. | 152,174 | 199,546 |
| Total value of metals | | .. | .. | 1,650,649 | 1,768,774 |

### Imports of Chemicals and Dyestuffs for Month ending 30th November.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Alkali | Cwt. | 16,401 | 26,425 | 12,060 | 11,512 |
| Bark (tanners', &c.) | „ | 15,011 | 20,355 | 5,004 | 7,280 |
| Borax | „ | .. | 305 | .. | 260 |
| Brimstone | „ | 25,426 | 33,851 | 6,532 | 8,658 |
| Chemicals | Value £ | .. | .. | 118,678 | 132,060 |
| Cochineal | Cwt. | 654 | 550 | 3,768 | 3,104 |
| Cutch and gambier | Tons | 2,095 | 1,864 | 38,916 | 27,356 |
| Dyes :— | | | | | |
| Alizarin | Value £ | .. | .. | 26,688 | 21,485 |
| Anilin and other | „ | .. | .. | 48,820 | 46,679 |
| Indigo | Cwt. | 771 | 635 | 13,140 | 9,633 |
| Nitrate of potash | „ | 10,537 | 21,642 | 8,787 | 18,936 |
| Valonia | Tons | 1,528 | 1,507 | 15,439 | 16,508 |
| Other articles | Value £ | .. | .. | 74,777 | 91,102 |
| Total value of chemicals | | .. | .. | 372,609 | 394,603 |

### Imports of Oils for Month ending 30th November.

| Articles. | | Quantities. | | Value. | |
|---|---|---|---|---|---|
| | | 1897. | 1898. | 1897. | 1898. |
| | | | | £ | £ |
| Cocoa-nut | Cwt. | 8,887 | 25,368 | 12,757 | 28,972 |
| Olive | Tuns | 583 | 342 | 20,831 | 11,924 |
| Palm | Cwt. | 76,812 | 82,113 | 80,526 | 90,177 |
| Petroleum | Gall. | 16,680,094 | 15,143,472 | 283,434 | 276,343 |
| Seed | Tons | 3,190 | 3,342 | 66,764 | 62,869 |
| Train, &c. | Tuns | 1,220 | 2,269 | 19,434 | 35,644 |
| Turpentine | Cwt. | 56,816 | 12,012 | 63,457 | 14,438 |
| Other articles | Value £ | .. | .. | 97,877 | 89,383 |
| Total value of oils | | .. | .. | 645,080 | 609,750 |

### IMPORTS OF RAW MATERIAL FOR NON-TEXTILE INDUSTRIES FOR MONTH ENDING 30TH NOVEMBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Bark, Peruvian .. Cwt. | 1,115 | 1,184 | 2,652 | 2,848 |
| Bristles.......... Lb. | 322,309 | 349,260 | 38,166 | 38,274 |
| Caoutchouc...... Cwt. | 34,341 | 47,619 | 355,427 | 657,777 |
| Gum:— | | | | |
| Arabic......... " | 3,695 | 6,326 | 8,587 | 13,837 |
| Lac, &c. ....... " | 11,267 | 6,231 | 35,793 | 21,984 |
| Gutta-percha .... " | 1,780 | 4,577 | 15,288 | 52,201 |
| Hides, raw:— | | | | |
| Dry............ " | 44,495 | 33,409 | 116,875 | 82,448 |
| Wet........... " | 60,000 | 54,411 | 121,613 | 116,467 |
| Ivory............ " | 708 | 879 | 32,565 | 35,275 |
| Manure:— | | | | |
| Guano......... Tons | 3,038 | 2,795 | 18,395 | 16,832 |
| Bones......... " | 3,178 | 5,262 | 12,268 | 21,742 |
| Nitrate of soda... " | 4,460 | 8,369 | 32,852 | 65,162 |
| Phosphate of lime " | 20,740 | 18,301 | 32,368 | 25,873 |
| Paraffin.......... Cwt. | 83,340 | 74,674 | 72,122 | 64,121 |
| Linen rags....... Tons | 2,176 | 1,468 | 20,819 | 14,061 |
| Esparto.......... " | 12,729 | 10,484 | 54,775 | 42,478 |
| Pulp of wood:— | | | | |
| Chemical...... " | 39,403 | 19,695 | 193,260 | 131,234 |
| Mechanical .... " | | 20,430 | | 55,925 |
| Rosin............ Cwt. | 69,632 | 63,878 | 13,688 | 13,732 |
| Tallow and stearin .. | 153,330 | 139,585 | 152,737 | 152,260 |
| Tar ...............Barrels | 13,088 | 4,232 | 11,102 | 3,510 |
| Wood:— | | | | |
| Hewn ......... Loads | 204,762 | 170,723 | 433,657 | 335,447 |
| Sawn .......... " | 556,464 | 427,173 | 1,285,431 | 977,875 |
| Staves......... " | 10,222 | 8,012 | 39,960 | 44,789 |
| Mahogany ..... Tons | 7,050 | 7,298 | 63,163 | 63,133 |
| Other articles....Value £ | .. | .. | 914,511 | 1,078,635 |
| Total value ....... | .. | .. | 4,078,164 | 4,127,950 |

Besides the above, drugs to the value of 75,962*l*. were imported, as against 98,620*l*. in November 1897.

### EXPORTS OF METALS (OTHER THAN MACHINERY) FOR MONTH ENDING 30TH NOVEMBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Brass............ Cwt. | 10,454 | 9,827 | 46,964 | 42,877 |
| Copper:— | | | | |
| Unwrought.... " | 42,099 | 61,047 | 107,834 | 170,538 |
| Wrought...... " | 26,525 | 25,200 | 82,812 | 81,782 |
| Mixed metal... " | 22,277 | 19,058 | 52,425 | 47,159 |
| Cutlery .........Value £ | .. | .. | 181,190 | 184,813 |
| Hardware ....... " | | | | |
| Implements ..... " | .. | .. | 128,543 | 114,562 |
| Iron and steel ... Tons | 313,365 | 252,537 | 2,179,513 | 1,854,937 |
| Lead ............ " | 2,427 | 2,515 | 35,149 | 30,868 |
| Plated wares ... Value £ | .. | .. | 49,725 | 48,672 |
| Telegraph wires . " | .. | .. | 34,341 | 39,837 |
| Tin ............ Cwt. | 8,767 | 10,243 | 28,910 | 42,371 |
| Zinc............ " | 16,717 | 12,198 | 12,551 | 12,278 |
| Other articles .. Value £ | .. | .. | 96,295 | 83,776 |
| Total value ....... | .. | .. | 3,036,552 | 2,760,470 |

### EXPORTS OF DRUGS AND CHEMICALS FOR MONTH ENDING 30TH NOVEMBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Alkali............ Cwt. | 472,170 | 331,381 | 121,837 | 87,974 |
| Bleaching materials " | 131,948 | 111,116 | 41,116 | 30,167 |
| Chemical manures Tons | 25,306 | 30,720 | 150,898 | 176,059 |
| Copper sulphate .. " | 1,730 | 247 | 27,346 | 4,138 |
| Medicines....... Value £ | .. | .. | 102,924 | 104,062 |
| Other articles... " | .. | .. | 309,419 | 288,573 |
| Total value .......... | .. | .. | 753,840 | 690,973 |

### EXPORTS OF MISCELLANEOUS ARTICLES FOR MONTH ENDING 30TH NOVEMBER.

| Articles. | Quantities. | | Value. | |
|---|---|---|---|---|
| | 1897. | 1898. | 1897. | 1898. |
| | | | £ | £ |
| Gunpowder...... Lb. | 339,600 | 608,600 | 8,691 | 15,878 |
| Military stores.. Value £ | .. | .. | 134,897 | 142,742 |
| Candles.......... Lb. | 2,415,600 | 2,899,900 | 31,429 | 35,578 |
| Caoutchouc..... Value £ | .. | .. | 123,851 | 112,447 |
| Cement.......... Tons | 26,325 | 25,008 | 43,658 | 48,053 |
| Products of coal Value £ | .. | .. | 147,492 | 121,957 |
| Earthenware ... " | .. | .. | 151,861 | 151,327 |
| Stoneware...... " | .. | .. | 15,201 | 14,028 |
| Glass:— | | | | |
| Plate........... Sq. Ft. | 126,111 | 134,181 | 7,288 | 8,120 |
| Flint........... Cwt. | 8,075 | 7,871 | 20,483 | 18,425 |
| Bottles......... " | 74,619 | 75,957 | 33,865 | 35,252 |
| Other kinds.... " | 20,618 | 26,798 | 15,866 | 24,369 |
| Leather:— | | | | |
| Unwrought .... " | 13,325 | 13,279 | 109,887 | 116,976 |
| Wrought ..... Value £ | .. | .. | 36,765 | 40,348 |
| Seed oil.......... Tons | 4,866 | 3,679 | 83,823 | 65,935 |
| Floorcloth ..... Sq. Yds. | 1,916,700 | 1,733,100 | 81,946 | 76,188 |
| Painters' materials Val. £ | .. | .. | 142,141 | 154,128 |
| Paper ........... Cwt. | 75,341 | 75,902 | 117,897 | 121,138 |
| Rags............ Tons | 6,421 | 5,052 | 30,451 | 26,444 |
| Soap............. Cwt. | 61,351 | 62,164 | 64,200 | 68,268 |
| Total value .......... | .. | .. | 2,848,314 | 2,937,739 |

# Monthly Patent List.

* The dates given are the dates of the Official Journals in which acceptances of the Complete Specifications are advertised. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the said dates.

## I.—PLANT, APPARATUS, AND MACHINERY.

### APPLICATIONS.

24,088. R. Glaister. Improvements in air-compressing apparatus. Nov. 15.

24,257. C. E. Vernon and A. Ross. Improvements in or relating to apparatus for testing lubricants. Nov. 17.

24,312. L. H. Walter. Improvements in or relating to the utilisation of hertzian and similar radiations, and apparatus therefor. Nov. 17.

24,374. T. Callow and R. Orrock. The patent multi-glasine furnace. Nov. 18.

24,442. J. Craven, jun. Improvements in apparatus for distributing liquids. Complete Specification. Nov. 19.

24,498. J. Davie. Improved apparatus for evaporating water or other liquids by means of steam. Nov 21.

24,518. J. G. Davies. An improved apparatus for heating and for cooling liquids. Nov. 21.

24,542. Baron H. Tindal. An improved apparatus for sterilising liquids by means of ozone. Nov. 21.

24,543. Baron H. Tindal. An improved apparatus for generating ozone. Nov. 21.

24,906. C. Schneider. Nitro substitution under pressure. Nov. 25.

25,010. J. Müller. An improved exhauster for poisonous or corrosive gases and like chemical purposes. Complete Specification. Nov. 26.

25,168. S. Puplett. Improvements in condensers and evaporators. Nov. 28.

25,971. A. S. Barham. Improvements in apparatus for sterilising. Dec. 8.

26,199. W. P. Thompson. Improvements in surface condensers. Dec. 12.

26,214. E. B. Phillips and W. H. Bray. Improved electrical furnace. Dec. 12.

26,282. G. I. Cree. Charring furnace. Dec. 13.

26,361. O. P. Ostergren and M. Burger. Improvements in apparatus for the liquefaction of aeriform fluids. Complete Specification. Dec. 13.

26,416. A. B. Willway. An improved construction and arrangement of a valve for controlling the flow of gases. Dec. 14.

26,646. R. Baumann. Improvements in or relating to crucible furnaces. Complete Specification. Dec. 16.

26,688. J. N. Hochgesand. Improvements in pumps for liquid or gaseous bodies and for various uses. Dec. 17.

### Complete Specifications Accepted.

1897.

27,711. J. H. Carpenter. Apparatus for purifying air and gases; also for heating and cooling of the same. Nov. 30.

28,113. J. Bock. Process and apparatus for obtaining large, well-formed crystals from solutions. Dec. 7.

28,747. J. McNeil and J. Lewkowitsch. Evaporating or concentrating apparatus. Dec. 7.

29,286. T. H. Heriot. Apparatus for separating suspended solid from liquids, for separating soluble from insoluble solid matters, and for partially separating insoluble solid mtaters of different specific gravities. Nov. 30.

29,763. H. Kühne. Apparatus for measuring the velocity of air and other gas currents. Nov. 23.

30,273. G. R. Barclay. Pressure filter and percolater. Dec. 21.

1898.

964. D. Black and J. M. Wright. Filter presses and similar filtering apparatus. Nov. 23.

1033. A. J. Boult.—From E. L. Delsol, France. Filters. Nov. 23.

1844. W. Rushworth. Means for refrigerating. Nov. 30.

2452. T. Thorp. Apparatus for mixing gases. Dec. 7.

4229. A. G. Hoffmann and A. Schwarz. Process of and means for heating and evaporating liquids; also applicable to the heating of vapours and gases. Dec. 21.

4925. C. W. J. Thomsen. Drying apparatus. Dec. 21.

17,410. W. Eckardt. Improvements in kilns. Dec. 14.

18,879. T. H. Cobley. Production of colloidal tannates as disincrustants for use in land and marine boilers. Dec. 14.

18,910. J. Onions. Edge runner grinding mill for powdering marls, bones, cements, slag, &c. Dec. 14.

18,921. J. J. Fries and F. Gerhard. Furnaces. Nov. 23.

18,969. E. I. Braddock. Manufacture of composite pipes, and apparatus therefor. Nov. 23.

19,742. E. C. C. Stanford. Apparatus for separating soluble bodies having different degrees of solubility. Nov. 30.

20,875. A. J. Boult.—From O. D. Orvis, United States. Furnaces. Dec. 14.

21,787. R. Beese and A. Perlich. Contrivance for raising liquids. Nov. 23.

22,221. C. W. R. Goeresch and P. J. Eisenstuck. Gloryhole furnaces. Dec. 14.

23,688. C. Martini. Process of and apparatus for drying viscous and like substances. Dec. 21.

## II.—FUEL, GAS, and LIGHT.

### Applications.

24,002. L. Troubetzkoy. Improvements in acetylene gas apparatus. Complete Specification. Oct. 14.

24,007. L. Jacquinet. Improvements in or relating to incandescence burners. Nov. 14.

24,040. J. B. de Léry. Improvements in and relating to mantles or the like for incandescent gas and oil lamps. Complete Specification. Oct. 15.

24,065. P. Margulies. Improvements in acetylene gas generators. Nov. 15.

24,093. D. S. Williams. Acetylene gas generator. Complete Specification. Nov. 15.

24,103. G. Henze and B. Barg. A new or improved inverted incandescent burner. Complete Specification. Nov. 15.

24,157. S. T. Richardson and R. Price. Improved means for preserving carbide of calcium. Nov. 16.

24,185. E. Emptage. Improvements in the method of and apparatus for lighting or illuminating cycles and other vehicles by means of acetylene gas. Nov. 16.

24,239. W. J. Ginder. Improvements in connection with the generation of acetylene gas. Nov. 17.

24,242. J. Fischl. A process for producing a spirituous combustible in solid form. Nov. 17.

24,283. F. Juhasz. Improved automatic gas igniter for incandescent mantles. Complete Specification. Nov. 17.

24,364. R. Nordmann. Improvements in incandescent mantles. Nov. 18.

24,408. W. R. Ormandy. Improvements in or relating to gas-producers. Nov. 18.

24,409. W. W. Beech and H. Jones. Improvements in apparatus for generating and storing acetylene and other gases. Nov. 18.

24,493. J. H. Exley. Improvements in apparatus for the generation and storing of acetylene gas. Nov. 21.

24,501. W. Ellen. Improvements in or connected with acetylene generators. Nov. 21.

24,543. Baron H. Tindal. *See* Class I.

24,557. R. Pringle. Improved apparatus for increasing the heating efficiency of flames produced by the combustion of gaseous or other fuel. Nov. 21.

24,623. F. W. Newman and A. H. Critchley. Improvements in or connected with apparatus for the production of acetylene gas. Nov. 22.

24,627. S. Bandrowski. An improved artificial fuel, and method of manufacturing the same. Nov. 22.

24,653. H. G. Semadeni.—From U. C. Loewy, Austria-Hungary. Improvements in the manufacture of incandescent gas mantles. Nov. 22.

24,682. O. Falbe. Improvements in acetylene gas generators. Complete Specificatioo. Nov. 22.

24,771. F. W. Lanchester. Improvements in gas generators. Nov. 23.

24,785. C. J. Bailey and J. H. Nicklin. Improvements in apparatus for generating acetylene gas. Nov. 24.

24,846. W. Irwin. Improvements in apparatus for carburetting or enriching gas. Nov. 24.

24,862. A. Dickson and R. Wallace. Improvements in apparatus for the generation and the storage of acetylene gas. Nov. 25.

24,885. W. Riley and A. Kershaw. Improvements in or appertaining to oxygen gas generators. Nov. 25.

24,929. O. Meurer. A process for the combustion of liquid hydrocarbons. Complete Specification. Nov. 25.

24,975. E. L. Levetus. Improvements in or relating to acetylene generators. Nov. 26.

25,016. A. Schwass. Improvements in acetylene generators. Complete Specification. Nov. 26.

25,030. C. Bingham. Improvements in carburetting. Nov. 28.

25,160. C. Barnett. Apparatus for the production of acetylene gas. Nov. 29.

25,209. E. Pilous, G. Ottermann, and E. Nauheim. *See* Class B.

25,268. A. Rammoser, E. Röhrs, and H. Röhrson. An improved process for producing automatically igniting mantles for incandescent burners. Complete Specification. Nov. 30.

25,297. P. Bucher. Improvements in apparatus for generating acetylene gas. Nov. 30.

25,.00. M Kandler and C. Wehner. An improved process, and apparatus relating thereto, for the manufacture of carburets. Nov. 30.

25,317. G. Dillberg. Improvements in or connected with acetylene and similar gas generators. Nov. 30.

25,397. W. C. Grieg and J. H. Gardner. Improvements in and relating to the manufacture of candles. Complete Specification. Dec. 1.

25,449. W J. Rodwell. Improvements in the generation of acetylene and in apparatus connected therewith. Dec. 2.

25,452. H E. Smith and W. Doman. Improvements in acetylene gas generators. Dec. 2.

25,581. W. J. Dossetter. Improvements in or relating to acetylene gas generators and the like. Dec. 5.

25,653. W. L. Voelker. Improvements in the manufacture of incandescence mantles or bodies for illuminating purposes, and in material for use in such manufacture. Complete Specification. Dec. 5.

25,654. A. B. Griffin. Improvements in incandescent gas-burners. Complete Specification. Dec. 5.

25,771. H. S. Elworthy and H. Lane. Improved method and apparatus for producing lighting, heating, and power gas. Dec. 6.

25,853. W. P. Thompson.—From G. Thuillier and A. Aubry, France. Improved apparatus for generating, purifying, and storing acetylene gas. Dec. 7.

25,917. D. C. Beggs and W. Fielding. Improvements in apparatus for the manufacture of acetylene gas. Dec. 8.

25,968. G. M. A. Claude. Process of and apparatus for purifying, storing, and utilising acetylene. Dec. 8.

25,997. H. Jellett. A new form of acetylene generator Dec. 9.

26,018. A. G. Adamson. Improvements in portable gasholders for supplying high pressure gas to incandescent mantles used for photographic and other purposes. Dec. 9.

26,206. H. Austin. Improvements in, or in and connected with, the means employed for carburetting or vaporising oil for use in oil engines. Dec. 12.

26,215. W. J. Rodwell. Improvements in the enriching of gases by acetylene and in apparatus connected therewith. Dec. 12.

26,221. F. Missire and M. L. Missire, *née* M. L. Maguin. Improvements in mantles for incandescent gas lighting. Dec. 12.

26,237. D. J. Van Praag and F. W. Harker. A new or or improved generator for generating acetylene gas in small quantities. Dec. 12.

26,238. The Economical Gas Apparatus Construction Company, Limited, and A. I. Payne. Improvements in and relating to the valves of apparatus for the manufacture of water and producer gas. Dec. 12.

26,253. R. Brunck. Improvements relating to the process of coking with the recovery of by-products. Dec. 12.

26,254. W. H. A. Sievets. Improvements in mantles and the like for incandescence gas lighting. Dec. 12.

26,269. H. Poetter. A coke-oven. Complete Specification. Dec. 12.

26,327. E. S. D'Odiardi. *See* Class XI.

26,328. E. S. D'Odiardi. *See* Class XI.

26,347. J. H. Needles and H. W. Crozier. Improvements in acetylene gas generators. Dec. 13.

26,474. L. Denayrouze. Improvements in incandescence lamps for burning oils, alcohol, and other volatile fluids; also solid fats and resinous substances. Dec. 14.

26,488. J. Wilson. Improvements in, and in the manufacture of, incandescent gas burners. Dec. 15.

26,533. H. Rey. Improvements in the process of producing carburetted air and in apparatus therefor. Dec. 15.

26,616. M. Strackosch and F. Schmidt. Improvements in acetylene gas producers. Dec. 16.

26,628. J. S. Goodwin. Improvements in acetylene generating apparatus. Dec. 16.

26,665. J. Jackson. Improvements in and relating to incandescent gas burners. Dec. 17.

26,697. W. T. Sugg. Improvements relating to the compressing of gases. Dec. 17.

### COMPLETE SPECIFICATIONS ACCEPTED.

### 1897.

26,381. A. M. Plaissetty. New incandescent mantle by gas, mineral oils, alcohol, and process of fabrication related thereto. Nov. 23.

26,842. E. S. Bond. Means for selectively using acetylene or the like gas with ordinary lamp apparatus. Nov. 23.

27,428. H. S. Riant. Electric furnaces for the production of calcium carbide and other substances. Nov. 30.

28,418. G. Tripel. New or improved apparatus for use in burning liquid fuel. Dec. 7.

30,112. G. L. Albert, Count de Hamel de Manin. Manufacture of incandescence bodies for illuminating purposes. Nov. 30.

30,272. W. H. Baughan. Apparatus for generating, cooling, and storing acetylene gas. Dec. 14.

### 1898.

28. J. McConechy. Burners applicable to acetylene and rich oil-gas lighting. Nov. 23.

315. A. Z. Germains. Means for lighting, heating, and smoke consuming. Nov. 23.

1005. J. O. O'Brien.—From P. P. H. Macé and de Burgue, France. Apparatus for producing acetylene gas. Nov. 30.

1920. R. J. Moss. Apparatus for generating acetylene gas. Dec. 7.

2452. T. Thorp. *See* Class I.

2453. T. Thorp. Gas conduits. Dec. 7.

2599. R. Boecking. Cooling device for the discharge mains or pipes of ovens or gas-producing apparatus through which the gases are led off for the condensation of tar, ammonia, benzol, &c. therefrom, and for use as illuminant fuel and the like. Dec. 7.

2961. B. J. Atterbury, T. MacAlpine, and The Alcohol Syndicate, Ltd. New or improved production of acetylene compounds, and of ethylene. Dec. 21.

3172. G. B. Ellis.—From L. E. Lacroix, France. Production of ozone, and apparatus therefor. Dec. 21.

3185. C. Mathieu. Process and apparatus for feeding gas engines with carburetted air for producing an illuminating and heating gas by recarburetting the burned products. Dec. 21.

3914. L. Etaix. Process for the purification of acetylene gas. Dec. 21.

5578. G. F. Dinsmore. Process of and apparatus for manufacturing gas. Dec. 7.

8325. A. F. Bowers. Apparatus for generating acetylene gas. Nov. 30.

9269. W. P. Thompson.—From J. Grand, France. Means for producing acetylene gas. Nov. 30.

12,199. M. Graham. Apparatus for charging inclined gas retorts. Dec. 7.

15,396. D. M. Baumgarten. Acetylene gas generators. Nov. 23.

15,818. A. J. Boult.—From E. Salzenberg, Germany. Incandescent gas lighting. Nov. 23.

17,682. W. P. Thompson.—From N. J. Carey, United States. Heat generators. Nov. 23.

18,231. J. F. Bachmann, A. Vogt, C. C. Weiner, A. Konig, J. Kirchner, and A. Jöig. Electrical heating appliances formed of artificial stone. Dec. 7.

19,757. A. Landsberger. Process of purifying acetylene gas. Nov. 30.

20,430. G. Dillberg. Acetylene gas generator. Nov. 30.

20,541. F. B. Poerschmann and H. Schnieder. Acetylene generators. Nov. 30.

20,597. C. Weygang. Apparatus for use in burning liquid fuel. Nov. 30.

21,678. E. Festner and G. Hoffmann. Coke ovens. Nov. 23.

21,760. Count A. D. de Micheroux. Apparatus or oven for the production of coke and lighting gas with recovery of the by-products. Dec. 14.

22,110. J. Schneeweis. Acetylene generator. Dec. 7.

22,784. J. B. Lafond. Manufacture of water-gas. Dec. 7.

22,985. G. Coming, E. Coming, and The Briquette Coal Company. Artificial fuel. Dec. 14.

23,369. R. Beese and A. Perlich. Gas burners with self-igniting Auer filament. Dec. 21.

## III.—DESTRUCTIVE DISTILLATION, TAR, PRODUCTS, Etc.

### Applications.

24,891. W. Young and J. Fyfe. Improvements in and relating to retorts for the destructive distillation of shale. Nov. 25.

25,118. B. H. Thwaite. The treatment and utilisation of coke-oven gases, and apparatus therefor. Nov. 29.

25,681. W. Irwin and J. P. Leather. New method of extracting naphthalene from coal-gas. Dec. 6.

26,253. R. Brunck. *See* Class II.

### Complete Specifications Accepted.

1898.

408. C. D. Abel.—From E. Theisen, Germany. Method and apparatus for treating the distillation products from carbonaceous materials and the like. Dec. 7.

## IV.—COLOURING MATTERS and DYES.

### Applications.

23,985. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of basic safranine azo dyestuffs soluble in water. Nov. 14.

24,177. Vidal Fixed Aniline Dyes, Ltd., and L. Haas. Improvements in the manufacture of colouring matters. Filed Nov. 16. Date applied for May 5, 1898, being date of application in France.

24,183. Vidal Fixed Aniline Dyes, Ltd., and L. Haas. Improvements in the manufacture of colouring matters. Filed Nov. 16. Date applied for July 25, 1898, being date of application in France.

24,191. Vidal Fixed Aniline Dyes, Ltd. Improvements in the manufacture of colouring matters. Filed Nov. 16. Date applied for June 29, 1898, being date of application in France.

24,286. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of diamidodiphenylamine disulphonic acid, and azo dyestuffs therefrom. Nov. 17.

24,373. J. B. Koenig. Improved method of manufacturing dye-soap powder. Nov. 18.

24,383. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of dyestuffs for cotton. Nov. 18.

24,538. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of blue to black sulphurous dyestuffs. Nov. 21.

24,644. I. Lichtentag and A. Lichtentag. An improved writing ink. Complete Specification. Nov. 22.

24,752. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. The manufacture of new colouring matters of the naphthalene series. Nov. 23.

24,830. H. H. Lake.—From Farbwerke Mühlheim, formerly A. Leonhardt and Co., Germany. Improvements in the manufacture of colouring matters. Nov. 24.

25,312. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of a black dyestuff directly dyeing cotton. Nov. 30.

25,404. S. Pitt.—From L. Cassella and Co., Germany. Method of producing mono-azo dyestuffs dyeing fast shades on mordants. Dec. 1.

25,508. S. Pitt.—From L. Cassella and Co., Germany. The production of disazo dyestuffs which are fixed by the aid of metallic mordants. Dec. 2.

25,566. O. Imray.—From The Farbwerke vormals Meister, Lucius und Brüning, Germany. Manufacture of azo colouring matters. Nov. 3.

25,614. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the production of indigo colouring matters and of indigo on the fibre. Dec. 5.

25,634. G. B. Ellis.—From La Société Chimique des Usines du Rhône, anciennement G. P. Monnet et Cartier, France. Improvements in the manufacture of indigo colouring matters and intermediate products. Dec. 5.

25,643. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of a new black dyestuff for cotton, and raw material therefor. Dec. 5.

25,644. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Manufacture of a dyestuff directly dyeing cotton. Dec. 5.

25,650. J. Y. Johnson.—From The Badische Anilin and Soda Fabrik, Germany. Improvements in the manufacture and production of disazo colouring matters and of products for use therein. Dec. 5.

25,651. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of mordant-dyeing colouring matters. Dec. 5.

25,964. G. W. Johnson.—From Kalle and Co., Germany. The manufacture or preparation of paradiamidophenylamine carboxylic acid and of colouring matters therefrom. Dec. 8.

25,965. G. W. Johnson.—From Kalle and Co., Germany. Improvements in the production of colouring matters on the fibre. Dec. 8.

25,966. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture of indigo. Dec. 8.

25,066. O. Imray.—From The Farbewerke vormals Meister, Lucius und Brüning, Germany. Manufacture of a brown polyazo dyestuff. Dec. 9.

26,167. H. R. Vidal. Improvements in the manufacture of colouring matters. Dec. 10.

26,168. H. R. Vidal. Improvements relating to the manufacture of colouring matters. Dec. 10.

26,261. J. Y. Johnson.—From C. F. Boehringer and Soehne, Germany. Improvements in the manufacture or preparation of paraxanthine and intermediate products relating thereto. Complete Specification. Dec. 12.

26,262. J. Y. Johnson.—From The Badische Anilin und Soda Fabrik, Germany. Improvements in the manufacture and production of disazo colouring matters. Dec. 12.

26,625. G. B. Ellis.—From La Société Chimique des Usines du Rhône anciennement G. P. Monnet et Cartier, France. The manufacture of new sulphonic acids from indigo colouring matters. Dec. 16.

### Complete Specifications Accepted.

1898.

445. S. Pitt.—From L. Cassella and Co., Germany. Production of phloroglucine. Dec. 7.

2892. J. Gardner. Liquid compound for dyeing purposes. Dec. 21.

## V.—TEXTILES: COTTON, WOOL, SILK, Etc.

### Applications.

24,254. A. McMinn. Flax scutching. Nov. 17.

24,268. W. A. Phillips. Improvements in the manufacture of cotton and other threads. Nov. 17.

24,768. A. Bouret and F. Verbiese. A process for retting or steeping vegetable textile fibres. Nov. 23.

24,784. J. H. Wood. Improvements in apparatus for mercerising woven fabrics. Nov. 24.

25,018. F. Hasselmann. Improvements in treating vegetable fibres. Complete Specification. Nov. 26.

25,056. H. Edelhoff. Improvements in woven stuffs with silk imitations. Complete Specification. Nov. 28.

25,757. E. Edwards. — From F. Gatzsche, Germany. Improvements in floorcloth and in the method of manufacturing the same. Dec. 6.

25,881. F. Gros and P. Bourcart. A new method of treating cotton to give it the glossy appearance of silk. Dec. 7.

26,368. S. C. Davidson. Improvements in apparatus for drying yarns. Dec. 13.

26,377. C. L. Bachelerie. Improved apparatus and process for the treatment of ramie or other textile plants. Complete Specification. Dec. 13.

### Complete Specifications Accepted.

1897.

28,631. H. Pauly. Process for the manufacture of artificial silk. Dec. 14.

1898.

1839. G. W. Oldham. Method of and means for mercerising hanks of yarn. Nov. 30.

3685. J. Eddison. Manufacture of wadding. Dec. 14.

12,161. A. J. Boult.—From F. A. Jone, Germany. Improved fabric or material. Nov. 23.

## VI.—DYEING, CALICO PRINTING, PAPER STAINING, and BLEACHING.

### Applications.

24,446. S. J. Pegg and S. A. Pegg. Improvements in or relating to dyeing or washing machines. Nov. 19.

24,184. W. Mather and R. H. Haworth. A process and apparatus for continuous bleaching of fabrics or yarns in in open condition. Nov. 19.

24,767. Société Française de Couleur d'Aniline de Pantin, E. Ruch, and G. Nuth. A process for applying primary azo dyes to and fixing them on fibre in dyeing and printing. Filed Nov. 23. Date applied for April 23, 1898, being date of application in France.

25,017. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. Improvements in the dyeing of hair and furs. Nov. 26.

25,388. W. Elworthy. Improved apparatus for generating ozone. Dec. 1.

25,780. P. Villette. Improvements in machinery for dyeing textile fabrics in hanks or skeins. Complete Specification. Dec. 7.

25,823. J. Rhodes. Improvements in the preparing and dyeing of tops, slubbing, wool yarns, silk, mohair, alpaca, jute, or any other textile fabrics. Dec. 7.

26,602. W. Coventry. Improvements in or applicable to machines for dyeing yarn in the form of cops, bobbins, or the like. Dec. 16.

### Complete Specifications Accepted.

1897.

29,533. W. G. Heys. — From A. Sorthiois, France. Bleaching camel's hair, goat's hair, and the like. Dec. 21.

1898.

3604. J. T. Reid and H. Thorp.—Dyeing textile fibres, yarns, and fabrics. Dec. 14.

4613. T. Cadgène. Dyeing of textiles and the like, and apparatus therefor. Dec. 7.

6546. A. J. Boult. — From Elbers, Germany. Indigo printing and dyeing. Dec. 7.

## VII.—ACIDS, ALKALIS, and SALTS.

### Applications.

23,978. F. M. E. Basse and G. Faure. A process for the manufacture of bi-sulphite of soda. Nov. 14.

24,643. H. C. Bull, A. C. Oakes, and T. M. Thom. A new or improved process for producing lime and obtaining carbonic acid. Nov. 22.

24,909. H. Walker. An improved method of making calcium carbide. Nov. 25.

24,933. W. P. Thompson. — From G. Wischin, Russia. Improvements in or relating to the manufacture of sulphuric anhydride and sulphuric acid. Nov. 25.

24,978. J. Dekker. A new or improved process and apparatus for crystallising sodium carbonate and other similar salts in small crystals. Nov. 26.

25,020. A. Goldschmidt. Improvements in the production or treatment of bichromate of soda. Nov. 26.

25,027. H. H. Lake.—From La Société Bonnet, Ramel, Savigny, Giraud et Marnas, France. Improvements in the manufacture of baryta. Nov. 26.

25,158. T. R. Shillito. — From The Actiengesellschaft für Zinkindustrie vormals W. Grillo and M. Schroeder, Germany. Improved means for recovering sulphuric acid and sulphuric anhydride from gases. Nov. 29.

25,558. R. England. Improvements in the concentration of sulphuric acids and other liquids. Nov. 3.

25, 56. H. Maxim. Improvements in methods and apparatus for the manufacture of calcium carbide. Complete Specification. Dec. 5.

25,748. M. Muspratt, E. S. Smith, and The United Alkali Company, Ltd. Improvements in the manufacture of hypochlorite of soda. Dec. 6.

25,925. M. Muspratt, E. S. Smith, and The United Alkali Company, Ltd. Improvements in the manufacture of hypochlorite of soda. Dec. 8.

25,962. E. S. Smith and The United Alkali Company, Ltd. Improvements in the manufacture of solutions of hypochlorites of alkalis. Dec. 8.

25,963. C. L. Higgins, M. Muspratt, E. S. Smith, and The United Alkali Company, Ltd. Improvements in the manufacture of solutions of hypochlorites of alkalis. Dec. 8.

26,059. C. S. Bradley and C. B. Jacobs. Improvements in process of manufacturing oxides. Complete Specification. Dec. 9.

26,060. C. S. Bradley and C. B. Jacobs. Improvements in process for manufacturing soluble barium salts. Complete Specification. Dec. 9.

26,061. C. S. Bradley and C. B. Jacobs. Improvements in process of manufacturing benzene, anthracene, and other polymerised products of acetylene. Complete Specification. Dec. 9.

26,169. J. L. Kessler. An improved apparatus for concentrating sulphuric acid. Dec. 10.

26,259. J. Bueb. Improvements in and connected with the separation or production of hydrocyanic acid from gases containing such acid or cyanogen. Dec. 12.

26,450. G. W. Handscomb. Improvements in machinery or apparatus for the manufacture of salt from brine. Dec. 14.

26,477. J. d'Orlowsky. Improved process and apparatus for manufacturing calcium carbide. Dec. 14.

### Complete Specifications Accepted.

1897.

29,009. J. Bastick and W. A. Thornton. Electric furnaces for the production of calcium carbide and other substances. Nov. 30.

30,053. J. Hutton. *See* Class X.

1898.

233. Count H. Plater-Syberg. Process for the production of acetic acid. Dec. 21.

1524. J. Sharp. Separating, purifying, and analysing mixed waste acids, and apparatus therefor. Dec. 21.

1984. G. Bower.—From J. E. Hewes, United States. Process for the manufacture of carbide of calcium, and apparatus therefor. Dec. 7.

1985. G. Bower.—From J. E. Hewes, United States. Process for the manufacture of carbide of calcium, and apparatus therefor. Dec. 14.

2816. F. Hurter and the United Alkali Company, Ltd. Manufacture of acetone, and apparatus therefor. Dec. 21.

2997. E. Gearing. Preparation of calcic and other gas-producing carbides. Dec. 21.

8010. J. L. Roberts. Processes of preserving carbides. Nov. 23.

19,470. W. R. Lake.—From La Société Bonnet, Ramel, Savigny, Giraud et Marnas, France. Manufacture of dioxide of barium. Dec. 21.

20,050. A. Gutensohn. Method of and apparatus for producing basic cupric acetate. Dec. 21.

## VIII.—GLASS, POTTERY, AND ENAMELS.

### APPLICATIONS.

24,086. D. Laffan and C. J. Bury. Improvements in the production of marbled or painted surfaces on glass or the like. Nov. 15.

24,603. W. F. Mason and W. H. Wilson. Improvements in kilns for bricks, pottery, and the like. Complete Specification. Nov. 22.

25,359. C. Marson. Improvements in means for facing walls and other surfaces with glass. Dec. 1.

25,676. J. Kent. Improved method of and means for scouring ceramic-ware, and transferring designs, patterns, or pictures thereto. Dec. 6.

26,346. P. Jensen.—From C. Z. F. Rott and T. C. Steimer, United States. Improvements in and relating to the manufacture of hollow glass ware, and apparatus therefor. Complete Specification. Dec. 13.

26,441. H. Procter and H. Taylor. Improvements in inlaid ceramic art decoration. Dec. 14.

26,547. P. T. Sievert. Improvements in the manufacture of decorated glass sheets. Complete Specification. Dec. 15.

26,660. J. Hobson. Improved apparatus or appliance for use in decorating ceramic ware. Dec. 17.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

28,466. C. Keller and A. Keller. Manufacture of ceramic earthenware, porcelain, stoneware, glass, crystal, and such like ware, with metallic decorations, and furnaces and ovens therefor. Nov. 30.

29,837. A. Matthews and W. Matthews. Manufacture of chemical and other earthenware. Dec. 14.

1898.

16,666. F. Ruppel. Kilns for cement. Nov. 23.

17,140. T. Pfister. Manufacture of mosaics, tiles, bricks, and the like. Dec. 21.

21,346. C. Dörr. Plastic and hardenable substance, and a process for its manufacture. Nov. 30.

## IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

### APPLICATIONS.

24,285. The United Asbestos Company, Ltd., and J. A. Fisher. An improvement in fireproof ceilings and partitions. Complete Specification. Nov. 17.

24,491. A. J. Keeble and G. H. Innes. An improved method of making Cambridge Portland cement from chalk and marl. Nov. 21.

24,629. A. E. Alexander.—From D. W. Peck, United States. An improved process for producing artificial asphalt. Nov. 22.

24,630. A. E. Alexander.—From D. W. Peck, United States. An improved process for producing artificial asphalt. Nov. 22.

24,941. O Howl. Improvements in apparatus for drying bricks or other plastic articles. Nov. 25.

25,155. T. W. James. Improvements in the manufacture of artificial marble, granite, and the like. Nov. 29.

25,304. R. Haddan.—From R. Koepp and Co., Germany. Improvements in the process of manufacturing artificial stones. Nov. 30.

25,945. W. H. Coward. Composition for paving blocks and other purposes. Dec. 8.

26,216. W. Houben. An improved manufacture of hard and elastic material for use in covering floors, walls, mouldings, &c. Dec. 12.

26,554. E. Baubil. Improvements in the manufacture of slabs, flagstones, and the like. Dec. 15.

### COMPLETE SPECIFICATIONS ACCEPTED.

1897.

27,561. W. Johnson and H. Walker. Kilns for burning cements, limes, and other like substances. Nov. 30.

27,825. J. C. Sellars. Manufacture of cement. Nov. 30.

28,276. H. Spatz and H. Quistorp. Process for the production of asphalt powder for making street asphalt plaster. Nov. 20.

28,656. W. H. Northcutt. Combination paving blocks and system of paving.

28,842. H. W. Gruner. Composition for the manufacture of artificial stone, cement, and the like. Nov. 23.

1898.

2822. H. H. Leigh.—From F. Cohn and H. G. B. Brunckhorst, Germany. Fireproof ceilings. Dec. 21.

10,425. M. Bachert and D. W. O'Niel. Impregnating wood. Nov. 30.

18,953. S. Jablónski and S. Nieuiarowski. Manufacture of fireproof protecting composition and of fireproof articles therewith. Nov. 30.

23,159. A. H. van der Vijgh, H. van der Vijgh, and G. van der Vijgh. Manufacture of artificial stone. Dec. 14.

## X.—METALLURGY, MINING, ETC.

### APPLICATIONS.

24,087. W. R. Ormandy. Improvements in separating iron from other mineral substances and in apparatus therefor. Nov. 15.

24,288. H. Dachelet. Mechanical hot process of galvanising iron plates of any thickness. Nov. 17.

24,289. V. Beaurang. Improvements in the manufacture of zinc, and apparatus therefor. Nov. 17.

24,492. J. G. Inshaw. Improvements in the conversion of puddled iron, steel, or other metal into hollow blooms for the manufacture of seamless tubes and other purposes. Nov. 21.

24,509. H. Langen. Improvements in annealing pots. Complete Specification. Nov. 21.

24,749. W. Ivanoff. Improvements in the manufacture of iron and steel directly from ores and in apparatus therefor. Nov. 23.

24,878. A. G. Brookes.—From L. Mach, Germany. A new and improved alloy of aluminium and magnesium. Nov. 25.

25,015. L. Moris. New or improved process for treating the residue resulting from the extraction of platinum from platinum ore. Nov. 26.

25,097. W. L. Wise.—From J. Burns, United States. Improvements in the manufacture of alloys of copper and iron or steel. Nov. 28.

25,150. C. Roth and C. Kruka. Projectile consisting of malleable tungsten alloy. Complete Specification. Nov. 29.

25,243. W. Burgam and B. W. Winder. Improvements in and relating to annealing furnaces. Nov. 30.

25,431. T. Twynam and F. W. Harbord. Improvements in the treatment of zinc-lead sulphide ores. Dec. 2.

25,477. C. L. C. Berton. Process of treatment of lead, tin, copper, and other metals or metallic alloys with a view to prepare oxides in a pulverulent state. Dec. 2.

25,481. W. P. Thompson.—From P. Argall, United States. Improvements in apparatus for cooling ores. Dec. 2.

25,546. W. A. P. Werner. Improvements in and relating to the metallisation of surfaces. Dec. 3.

25,705. A. J. Boult.—From G. H. Olamer, United States. Lead coating for metal surfaces, and method of making the same. Complete Specification. Dec. 6.

25,784. J. Fraser. Process of extracting metals from their ores. Dec. 7.

25,875. G. Dillberg. Improvements in the extraction of metals. Dec. 7.

25,979. F. W. Martino and F. Stubbs. Improvements in or relating to the treatment of ores and the precipitation of precious metals from their cyanide solutions. Dec. 8.

25,983. A. Lavoix. Improved amalgamating apparatus for extraction of precious metals from minerals. Dec. 8.

26,134. F. J. Bergmann. An improved method of and apparatus for obtaining high temperatures for fusing refractory metals, for producing carbides, and for like purposes. Complete Specification. Dec. 10.

26,260. J. Patrick. Improvements in the manufacture of nickel alloys. Dec. 12.

26,391. W. S. Rock. Improvements in the production of oxide of iron. Dec. 13.

26,393. P. C. Van de Stadt. Soldering or brazing aluminium. Dec. 13.

26,502. A. S. Plews and J. W. Worsey. Improvements in metallurgical processes and in apparatus to be used in such or similar processes. Dec. 15.

26,526. E. J. Clark. A new or improved crucible for casting lead or like metals under pressure, especially applicable to the formation of secondary electric battery grids or plates. Dec. 15.

26,531. D. Martini. Improvements in and in means for reducing ores by ozone. Dec. 15.

26,717. F. Chaist. Improved process of soldering or brazing. Dec. 17.

### Complete Specifications Accepted.

#### 1897.

27,752. R. A. Hadfield. Manufacture of steel. Nov. 30.

27,753. R. A. Hadfield. Manufacture of steel. Nov. 30.

29,572. J. C. Richardson. Solvents for metals and the treatment of gold and other ores. Dec. 14.

29,611. W. F. Lay. Mining of placer deposits, or similar deposits of a sedimentary nature, and extraction of the gold or other valuable material therefrom. Dec. 21.

29,721. J. C. Montgomerie. Means applicable for use in the extraction of metals from metallic solutions. Dec. 14.

30,053. J. Hutton. The separation of tungstic acid from wolfram and other minerals containing tin and other metals. Dec 21.

#### 1898.

1106. L. Mond. Process for obtaining metallic nickel from nickel carbonyl, and apparatus therefor. Dec. 7.

2817. J. M. Gledhill. Apparatus employed for melting steel or other metal. Dec. 14.

5518. P. F. Secrétan. Aluminium nickel bronze. Dec. 21.

6606. C. E. Acker. Reduction of metals from their salts, and apparatus therefor. Nov. 30.

20,283. C. Brand. Mould for casting slag blocks. Nov. 23

20,328. G. Bennett. Steel ingot moulds. Nov. 30.

22,265. E. Raynaud. Means for facilitating extraction of alumina from aluminous ores. Dec. 7.

22,871. A. M. Nicholas. Rotating filtering apparatus, principally applicable for the separation of gold- and silver-bearing solutions from tailings, slimes, and the like. Dec. 21.

23,227. J. E. Sutphen. Amalgamator. Dec. 21.

## XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

### Applications.

24,005. A. J. Boult.—From Seduelf and Bratasched, Germany. Improvements in electrical storage batteries. Nov. 14.

24,104. E. Wilson and J. M. Cater. Improvements in apparatus connected with the charge and discharge of storage cells. Nov. 15.

24,171. H. Schloss. Improvements in voltaic cells having dry plates which may be regenerated. Complete Specification. Nov. 16.

24,210. H. H. Lake.—From v. d. Poppenberg's Elemente und Akkumulatoren, Wilde and Co., Germany. Improvements in the manufacture of electrodes for secondary batteries. Nov. 16.

24,307. J. C. Fell.—From W. Strzoda, Germany. A process for the electrolytic extraction of zinc and other metals from ores or waste containing them. Complete Specification. Nov. 17.

24,308. H. J. Haddan.—From Columbus Electrizitätsgesellschaft, Germany. Improvements in electric batteries. Nov. 17.

24,357. A. T. Ellis. Improvements in or connected with electric batteries. Nov. 18.

24,438. C. M. Stead. Improvements in or in connection with the construction of dry voltaic cells, accumulators, and secondary batteries. Complete Specification. Nov. 19.

24,530. C. T. J. Oppermann. Improvements in and relating to electrical accumulators. Nov. 21.

24,570. W. S. Rawson. Improvements connected with electric batteries. Nov. 22.

24,605. W. H. Hanson. Improvements in secondary battery plates, grids, and the like. Nov. 22.

24,611. N. H. Edgerton and A. H. Masters. Improvements in secondary batteries. Complete Specification. Nov. 22.

24,757. M. Wuillot. Improved manufacture of lead plates for secondary batteries. Nov. 23.

24,905. R. Bayer. Preparation of hydrocyanic acid and other cyanogen compounds from rhodanic acid and its salts by means of electrolysis. Nov. 25.

25,388. W. Elworthy. See Class VI.

25,410. C. A. Sahlström. Improvements in batteries or apparatus for producing ozone. Dec. 1.

25,734. W. Schlothauer and A. E. Preece. Improvements in secondary batteries. Dec. 6.

26,026. W. G. Heys.—From S. Silberberg, United States. Improvements in and relating to galvanic batteries. Complete Specification. Dec. 9.

26,116. W. G. Heys.—From S. Silberberg, United States. Improvements in and relating to galvanic batteries. Complete Specification. Dec. 10.

26,325. E. S. D'Odiardi. Improvements of electrodes of deep penetration. Dec. 13.

26,327. E. S. D'Odiardi. Improvements in electro-inhalers and nascent ozone producers. Dec. 13.

26,328. E. S. D'Odiardi. Improvements in electro vaporisers and ozonisers. Dec. 13.

26,343. C. Brault. Improvements in the manufacture and production of plates or elements for secondary batteries or electrical accumulators. Dec. 13.

26,351. J. Wetter.—From M. Maas, United States. Improvements in galvanic elements or batteries. Complete Specification. Dec. 13.

26,380. E. Giglio. Improvements in electric batteries. Dec. 13.

26,476. C. E. Burroughs. Improvements in batteries. Complete Specification. Dec. 14.

26,614. J. A. Mays. Process and apparatus for treating ores by electrolysis. Dec. 16.

26,695. W. Puttkamer. Improvements in electric accumulators or secondary batteries. Dec. 17.

26,703. H. T. Harrison. Improvements in primary batteries. Dec. 17.

### Complete Specifications Accepted.

1897.

27,508. O. March. Extraction of gold by electrolysis. Nov. 30.

29,014. J. Bastick and W. A. Thornton. Electric furnaces for the production of calcium carbide and other substances. Nov. 30.

1898.

1628. W. A. Crowdus. Secondary batteries. Dec. 7.

1735. A. G. Adamson, T. W. Allan, and Allan and Adamson, Ltd. Electrical accumulators or storage batteries. Nov. 23.

2684. F. Fisher, B. J. Round, B. J. Round, and A. Round. Apparatus for the electro-deposition of metals. Nov. 30.

3749. J. G. A. Rhodin. Method of oxidation and bleaching by means of electrolysis. Dec. 7.

4514. V. I. Feeny.—From H. Becker, France. Cathodes. Dec. 21.

20,956. L. Pirwitz and W. Behr. Non-conducting material or composition, and process for the production of the same. Dec. 14.

22,171. J. H. Graeber and H. Tobler. Insulating devices for the electrodes of accumulators. Nov. 30.

22,461. J. Y. Johnson.—From H. J. Dercum, United States. Primary batteries. Dec. 7.

23,243. P. Ribbe. Electric accumulators. Dec. 21.

23,716. R. Haddan.—From G. J. Miller, United States. Secondary or storage batteries. Dec. 21.

## XII.—FATS, OILS, AND SOAP.

### Applications.

24,200. H. Cheetham. Improvements in or relating to refining tallow and other fats, oils, or greases. Nov. 16.

24,375. C. Dreher. Improvements in the manufacture or production of resin soap. Nov. 18.

24,402. W. P. Thompson.—From A. Kartschmaroff, Austria-Hungary. Improved composition for cleansing purposes. Nov. 18.

24,456. D. H. McClelland and S. J. Butterfield. Improvements in oil purifiers. Complete Specification. Nov. 19.

24,740. G. Mitchell. Process and apparatus for extracting oil from seeds, nuts, beans, and the like. Nov. 28.

24,791. W. Evans. Improvements in the method of and means for purifying and filtering dirty and waste oils. Nov. 24.

25,034. S. V. Dardier. A new or improved soap. Nov. 28.

25,287. W. P. Thompson.—From J. Fitzgerald, jun., and J. C. Mahon, Canada. Improved cleansing compound for textiles and other delicate articles. Complete Specification. Nov. 30.

25,624. J. Foster. An improved cleansing composition. Dec. 5.

26,539. C. de la Roche. Improvements in the treatment of oils and essential oils, and particularly of drying oils. Dec. 15.

### Complete Specifications Accepted.

1897.

29,612. J. Hopkinson, T. Cowling, and F. Illingworth. Treatment of wool grease and of other like substances, and of derivatives therefrom. Nov. 23.

1898.

2836. H. Young. Saponaceous and bleaching compounds. Dec. 14.

17,510. H. L. Haas. Cleansing compositions containing ammonia. Dec. 21.

20,058. H. Hampel and V. Zampis. Method of preparation of a rosin soap for paper-sizing containing an admissible quantity of free rosin. Nov. 30.

21,369. R. Adam. Soap compound which can also be used as a combustible deodoriser, and process for the manufacture of the same. Dec. 7.

## XIII.—PAINTS, PIGMENTS, VARNISHES, RESINS, INDIA-RUBBER, Etc.

### Applications.

24,211. The East Ferry Road Engineering Works Company, Ltd., F. S. Tuckett, L. Lancaster, and W. W. and R. Johnson and Sons, Ltd., and E. M. Johnson. Improved method of and means for drying white lead. Nov. 16.

24,396. H Hobbs. An improved whitening, stiffening, and glazing composition for laundry use. Nov. 18.

24,848. M. Zingler. An improved method of treating decayed or other rubber. Complete Specification. Nov. 24.

24,932. A. Nevin and T. Bulfield. Improvements in compositions for distempering or painting. Nov. 25.

25,256. E. J. C. Christie and R. Anderson. Improvements in treating the milk, juice, or sap of trees, shrubs, or plants, yielding gutta, balata, rubber, or other rubber-like substances. Nov. 30.

25,275. A. J. Boult.—From P. Carié, France. Improved fireproof paint or composition. Nov. 30.

25,286. C. J. Grist. Improvements in the manufacture of composition having as a basis oxidised oil, or oxidised oil and a fibrous material. Nov. 30.

25,315. A. Roberts. Improvements in the manufacture of coloured marking-ink pencils. Complete Specification. Nov. 30.

25,772. E. O. Taflin. Improved drying and painting compound. Dec. 6.

26,123. R. Bayer. Improved process for producing pure lead oxide. Dec. 10.

26,157. J. de la Fresnaye. Improved process and apparatus for the extraction of substances contained in vegetable and other bodies, especially applicable to the extraction of rubber and the recovery of the solvents employed. Dec. 10.

26,434. H. C. Webster and T. French. Improvements in apparatus for use in drying white lead. Dec. 14.

26,617. The Marquis Henri Bertrand de Laqueuille. An improved process for the regeneration and purification of india-rubber, and the application of this process to ebonite, gutta-percha, gums, resins, balms, oils, essences, and the like. Filed Dec. 16. Date applied for June 16, 1898, being date of application in France.

### Complete Specifications Accepted.

1897.

26,867. J. S. Macarthur. Anti-fouling composition. Nov. 23.

1898.

661. E. Toussaint. Waterproof coating for walls and the like. Dec. 21.

3909. R. H. Biffen and E. W. Howard. Treatment of india-rubber, gutta-percha, and other gums. Dec. 21.

8106. H. H. Lake.—From C. Curtis, United States. Manufacture of a substitute for leather, rubber, and other flexible materials. Nov. 23.

15,140. H. J. Haddan.—From G. Zonca and Co., Germany. Proceeding for producing a varnish and colour mordant. Nov. 23.

19,869. C. D. Abel.—From H. C. Meyer, jun., Germany. Process for the treatment of resinous cane for the removal of the resin therefrom. Dec. 14.

22,888. D. F. Lucas. Composition for coating or painting roofs, vessels, and the like. Dec. 7.

## XIV.—TANNING, LEATHER, GLUE, and SIZE.

### Applications.

24,344. F. A. Cliff. An improved composition to be used for dressing leather. Complete Specification. Nov. 18.

24,505. J. Muir. Improvements in the treatment of pig skins or other skins or hides whereby they are rendered more economical, more durable, and more serviceable for standing tensile strain, attrition, wear, or for general use. Nov. 21.

24,844. J. V. Koch. Improvements in tanning. Nov. 24.

24,903. T. H. L. Hake. Improvements in tanning skins and hides and in apparatus therefor. Nov. 25.

25,017. C. D. Abel.—From The Actiengesellschaft für Anilinfabrikation, Germany. *See* Class VI.

25,188. A. E. Vidal. Improvements in tanning and in apparatus therefor. Nov. 29.

26,390. H. Holmes. Improvements in the art of preparing hides or skins for tanning and in liquid compositions of matter for treating such hides and skins. Complete Specification. Dec. 13.

26,470. J. Hutchings. Improvements in machinery or apparatus for treating hides, kips, or skins for the manufacture of leather. Dec. 14.

26,576. S. L. Johnson, E. Johnson, J. Brooksbank, and A. H. Gibbings. Improvements in the means or method of and apparatus for removing wool and other hair from skins. Dec. 16.

### Complete Specifications Accepted.

1898.

3678. A. MacFarlane and H. W. Stanbury. Process of treating gelatin, and the production therefrom of imitations of various substances. Dec. 14.

3959. J. Young. Apparatus for tanning, dressing, or otherwise treating hides or skins and leather. Dec. 21.

8106. H. H. Lake. *See* Class XIII.

21,663. J. L. Planckaert and F. Vuylsteke. Process for tanning rabbit and other skins. Dec. 7.

21,803. H. Wade.—From F. A. T. Moor, United States. Manufacture of leather. Nov. 23.

22,143. J. Sprachocinski and C. Stoldt. Impermeable leather. Nov. 30.

22,995. A. O. Eevenbye. A dressing composition for leather. Dec. 21.

## XV.—AGRICULTURE AND MANURES.

### Applications.

24,321. F. M Mooney and A. Dickson. Improved form of fertiliser. Nov. 18.

25,475. A. Frank and N. Caro. Improvements in the manufacture of nitrogen compounds. Dec. 2.

### Complete Specifications Accepted.

1898.

1435. G. O. J. Schüler. Production of double superphosphates. Dec. 7.

1541. O. Imray.—From Bauer and Co., Germany. Albumen preparations soluble in water made from glycerinophosphates and albuminoids. Dec. 7.

## XVI.—SUGARS, STARCHES, GUMS, Etc.

### Applications.

25,642. O. Imray.—From Ranson's Sugar Process, Ltd., France. Improvements in purifying and decolourising saccharine liquors. Dec. 5.

26,475. E. Luck and S. G. Luck. Improvements in purifying and decolorising saccharine liquors. Dec. 14.

### Complete Specifications Accepted.

1897.

27,534. W. P. Thompson.—From L. Bussy, H. Philippe, and P. Bussy, France. Impermeable water-resisting protegum or coating. Nov. 23.

28,589. J. J. Hignette. Process for the purification of saccharine juices, and apparatus therefor. Dec. 14.

1898.

1131. J. Y. Johnson.—From A. Verley, France. Manufacture of sugar. Dec. 7.

## XVII.—BREWING, WINES, SPIRITS, Etc.

### Applications.

24,253. W. Cutler. Improvements in the construction of union casks used in brewing operations. Nov. 17.

24,741. J. T. Bennett. Improved process and apparatus for removing impurities from alcoholic and other liquids. Complete Specification. Nov. 23.

24,751. J. Heron. Improvements in fermentation for the production of alcohol in the manufacture of beer, wines, and the like. Nov. 23.

25,101. R. Rückforth Improved process of treating yeast or yeast products. Nov. 28.

25,491. A. H. Deakin and T. F. Garrett. Improvements in and connected with the manufacture of turtle wine. Dec. 2.

25,505. H. Vuylsteke. Improvements in or connected with apparatus for carbonating beers. Dec. 2.

25,974. J. Imray.—From La Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis and D. A. Rosenstiehl, France. Improvements in the manufacture of cider for retarding the fermentation of the apple juice. Dec. 8.

### Complete Specifications Accepted.

1897.

29,089. G. F. Zimmer. Improvements in and connected with gelatinising and drying apparatus used in the manufacture of flaked maize, malt, and similar substances. Dec. 21.

1898.

162. C. C. Doig. Process and apparatus for evaporating the burnt ale, spent lees, and residual products of distilleries, and for utilising the steam thereby engendered for motive power. Dec. 7.

2782. M. J. F. Saladin. Apparatus for germinating barley and other cereals for use in breweries, distilleries, and the like. Dec. 21.

22,892. C. Schwager. Malting apparatus. Dec. 7.

## XVIII.—FOODS, SANITATION, Etc., and DISINFECTANTS.

### Applications.

*A.—Foods.*

24,291. R. Endler. A new or improved method of preserving eggs. Nov. 17.

24,403. M. Marx. Improvements in preserving eggs. Complete Specification. Nov. 18.

24,724. P. L. Dithmer. An improved process for the production of nutritive preserved cream preparations. Nov. 23.

25,101. A. M. Clark.—From F. Dippel, Germany. Method of preserving eggs, fruit, and eatables in general. Dec. 1.

25,462. J. E. H. Wilson. A new or improved method of treating fruit for preserving purposes. Dec. 2.

25,779. H. Ballantyne and The Hovis Bread Flour Company, Ltd. Improvements in food preparations. Dec. 6.

25,973. C. Jung, A. Brecher, and A. Kittel. An improved manufacture of animal food from skim milk. Complete Specification. Dec. 8.

26,342. J. Y. Johnson.—From W. S. Forbes, United States. Improvements in and apparatus for heating and distilling water, milk, and other liquids, for sterilising or other purposes. Complete Specification. Dec. 13.

26,568. H. Ballantyne. Improvements in food preparations. Dec. 15.

*B.—Sanitation.*

24,503. E. Finch. A new and improved system for purification of town sewage. Nov. 21.

24,628. E. Löwi. Improvements in process and apparatus for drying industrial vegetable wastes. Complete Specification. Nov. 22.

25,000. W. P. Thompson.—From C. G. Gsell, Germany. An improved process for purifying waste water or sewage by precipitation. Nov. 26.

25,209. E. Pilous, G. Ottermann, and E. Nauheim. Improved method of producing gas from town refuse and sweepings, and apparatus therefor. Nov. 29.

25,383. C. A. Atkinson. Improvements in or relating to the treatment or purification of sewage and apparatus therefor. Dec. 1.

25,409. C. A. Sahlström. Improvements in the process and apparatus for the purification and treatment of sewage. Dec. 1.

25,660. J. F. Johnstone. Improvements in apparatus for treating and drying fish and other offal. Dec. 5.

25,686. C. J. Lomax. An improved machine for the extraction of liquid from sewage sludge or other substances. Dec. 6.

25,697. A. Millar. Improvements in drying and purifying air. Dec. 6.

26,525. W. O. E. Meade-King. An improved filter for manufacturers' trade effluents, sewage, or water. Dec. 15.

26,645. W. Laurence. Improvements in treating, purifying, sterilising, softening, and clarifying water or impure liquids by heating and cooling, and apparatus therefor. Dec. 16.

*C.—Disinfectants.*

25,139. J. M. L. Messerli *née* Imbert. A new or improved process for producing gases and vapours for fumigating purposes, and apparatus therefor. Complete Specification. Nov. 29.

Complete Specifications Accepted.

*A.—Foods.*

1897.

26,754. W. G. Day. Methods of preserving perishable substances. Nov. 23.

1898.

2679. J. P. van Gelder and P. van Gelder. Apparatus for collecting and sorting waste products, and generally for purifying air or other gases. Dec. 7.

20,992. N. S. W. K. Buckhup. Method of preserving milk or cream. Dec. 14.

23,631. H. C. Plant. Process and apparatus for removing dirt from milk. Dec. 21.

*B.—Sanitation.*

1898.

149. F. Eichen. Apparatus for treating and purifying sewage waste and similar waters. Nov. 30.

19,486. W. P. Thompson.—From V. D. Anderson, United States. Driers for treating distillery slops, brewers' grains, slaughter house refuse, and other masses containing a large percentage of moisture, together with solids, which are of value for fertilising or other purposes. Dec. 14.

22,000. F. W. Wentworth-Shields. Construction of bacterial tanks and filters for the purification of sewage. Nov. 30.

*C.—Disinfectants.*

1897.

30,036. A. Guasco. Chemical product for antiseptic purposes, and burner or device for utilising the same. Dec. 14.

1898.

21,369. R. Adam. *See* Class XII.

## XIX.—PAPER, PASTEBOARD, Etc.

Applications.

24,013. N. Basset. Method of treating cellulose. Complete Specification. Nov. 15.

24,600. J. Almond and S. F. Andrews. Improved method of and apparatus for mixing, beating, and bleaching pulp and other paper makers' materials. Nov. 22

24,904. W. W. Beaumont. Improvements in apparatus for straining paper pulp. Complete Specification. Nov. 25.

25,620. P. Piette. Improved manufacture of iridescent paper or material. Complete Specification. Dec. 5.

26,010. J. A. Bodeu. Improvements in the manufacture of artificial ivory and analogous substances. Dec. 9.

26,070. J. F. Duke. An improved mode of producing copies of written or printed matter, and the manufacture of ink and paper for the purpose. Dec. 9.

26,234. W. C. Leechman. Improvements in paper. Dec. 12.

26,500. A. Masson. Improvements in or applicable to the manufacture of paper. Dec. 15.

26,501. A. Masson and R. Scott. Improvements in paper making machinery. Dec. 15.

26,587. J. Westaway. A new or improved process and composition of matter for making artificial ivory, bone, and the like. Dec. 16.

26,604. J. Rowley. Improvements in the manufacture of paper. Dec. 17.

Complete Specifications Accepted.

1897.

28,982. F. Langley, J. Whittingham, and J. Harris. Process of and apparatus for sizing paper. Dec. 14.

1898.

18,970. A. Wiedwald. Construction of wooden rollers to be used in the manufacture of paper. Nov. 23.

20,058. H. Hampel and V. Zambis. *See* Class XII.

## XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES, and EXTRACTS.

Applications.

25,378. H. E. Newton. — From The Farbenfabriken vormals F. Bayer and Co., Germany. The manufacture or production of pharmaceutical compounds. Dec. 1.

26,392. A. W. Shirley. Improvements in or connected with menthol cones. Complete Specification. Dec. 13.

26,639. E. Kańder. An improved process for producing methyl-morphine (codeine) and ethyl-morphine. Dec. 16.

Complete Specifications Accepted.

1897.

24,891. E. C. C. Stanford. Process for separating and obtaining the active constituents of the thyroid gland of the sheep and other animals. Dec. 7.

26,303. E. J. Boake. Treatment of essences, liquid drugs, and the like for facilitating their transport. Nov. 23.

29,451. E. H. Taylor. Improved system and apparatus in connection therewith, for the emulsification of essential and other oils and water, and for producing extracts or tinctures from other substances with water for use in the manufacture of flavoured mineral waters, and for other purposes. Dec. 7.

1898.

1897. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Process for the production of albumoses. Dec. 21.

3391. H. E. Newton.—From The Farbenfabriken vormals F. Bayer and Co., Germany. Process for the production of albumoses. Dec. 21.

20,117. G. W. Johnson. — From C. F. Boehringer and Sohne, Germany. Manufacture and production of formic aldehyde compounds of uric acid and alkyluric acids Dec. 21.

22,268. R. W. James. — From Société Française de Désinfection, France. Product for the sterilising of germs, micro-organisms, and the like. Nov. 30.

## XXI.—PHOTOGRAPHY.

### Applications.

23,910. C. A. Burghardt. Improvements in the method of and means for producing photographic pictures. Complete Specification. Oct. 14.

24,230. R. Greenland. The process of washing photographic prints, films, and the like. Nov. 17.

24,750. O. Moh and A. Heskiel. Improvements in and relating to the production of photographic negatives. Complete Specification. Nov. 23.

25,760. E. Mertens. Improved process for producing coloured photographs. Dec. 6.

25,135. C. Raliegh. An improved method of conducting certain phtographic operations, and apparatus applicable therefor. Dec. 14.

### Complete Specifications Accepted.

1897.

28,920A. J. W. Bennetto. Positives for coloured photographs. Nov. 30.

1898.

3257. C. Henry. Smokeless magnesium powder for photography, and apparatus for employing the same. Dec. 7.

## XXII.—EXPLOSIVES, MATCHES, Etc.

### Applications.

23,969. J. Okell.—From H. Pipitz, Austria-Hungary. Improvements in the treatment of wood for the manufacture of nitro-compounds, solutions, and other derivatives of cellulose or lignose. Nov. 11.

24,168. E. A. G. Street. Improvements in or relating to explosives. Filed Nov. 19. Date applied for May 21, 1898, being date of application in France.

24,555. A. G. Bell. Improvements in processes of making gunpowder. Nov. 21.

24,662. A. Luck. Improvements in explosives. Nov. 22

25,102. R. E. A. Gans. Improved manufacture of ignition material for matches and other purposes. Nov. 28.

25,215. G. C. Marks.—From F. W. Mead, United States. Improvements in machines for making matches. Complete Specification. Nov. 29.

25,175. A. Frank and N. Caro. See Class XV.

25,568. Gustav, Count von Geldern-Egmond. Improvements in the manufacture of explosives. Complete Specification. Dec. 3.

25,838. J. E. Evans-Jackson.—From U. Alvisi, Italy. Improvement in explosives. Complete Specification. Dec. 7.

26,081. L. Gathmann. Improvements in and relating to shells for high explosives. Dec. 9.

26,249. H. Maxim. Process and apparatus for the manufacture of multi-perforated rods or grains of explosive materials known as smokeless powders. Dec. 12.

26,332. W. P. Jones and H. M. Bates. A new or improved match. Dec. 13.

26,529. F. A. Halsey. An improved smokeless gunpowder. Complete Specification. Dec. 15.

26,798. D. M. Mefford. An improvement in explesive shells. Dec. 17.

### Complete Specifications Accepted.

1897.

18,642. F. M. Hale, J. Deas, and J. B. Reavil. Percussion fuses for explosive projectiles or shells. Nov. 23.

1898.

2261. A. de Stubenrauch. Process of manufacturing smokeless gunpowder. Dec. 21.

2412. E. Gurovits and M. Bals. Process for rendering more effective lead oxide-hyposulphite as constituent part for priming or firing compounds. Dec. 7.

2809. J. G. Dixon. Detonators for fog-signalling. Dec. 14

20,989. G. P. Bickford-Smith. Compositton for electrically firing detonators. Dec. 14.

20,990. G. P. Bickford-Smith. Detonators for firing explosives. Nov. 30.

21,111. C. A. Allison. — From P. Hathaway, United States. Matches and the method of and apparatus for producing the same.

22,955. S. L. Fog and A. G. Kirschner. Process for the manufacture of matches. Dec. 14.

## PATENTS UNCLASSIFIABLE.

### Applications.

23,981. J. H. W. Stringfellow. Improvements in waterproof and acid-proof compounds. Nov. 14.

24,194. G. Rosenwald. Improved means or method of treating horn to render it like mother-of-pearl in appearance without destroying its suppleness. Nov. 16.

24,226. L. Lenton. A new waterproof and air-proof material. Nov. 17.

24,378. E. W. Engels. Improvements in or relating to fire and acid-proof materials. Complete Specification. Nov. 18.

26,256. W. F. Browne. Improved method and means for treating peat. Dec. 12.

### Complete Specification Accepted.

1897.

29,387. L. C. Reese. Extraction from liquids of matters therein contained. Dec. 7.

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